

# **ANNUAL REPORT 2009**

INSTITUTE OF ION BEAM PHYSICS AND MATERIALS RESEARCH



Forschungszentrum Dresden Rossendorf Wissenschaftlich-Technische Berichte FZD – 528 2010

# **Annual Report 2009**

# Institute of Ion Beam Physics and Materials Research

Editors:

J. von Borany, V. Heera, J. Faßbender, M. Helm, W. Möller



#### **Cover Picture:**

The inset graph shows the temperature dependence of the resistance R of a germanium (Ge) substrate (red line) and a heavily gallium (Ga) doped germanium layer (grey-blue line) measured in a four point probe arrangement (schematic as inset, U: voltage, I: current). While the Ge substrate exhibits an exponential increase of the resistance with decreasing temperature as typical for semiconductors, the heavily Ga doped Ge layer behaves "metallic" and becomes superconducting below 0.3 K. The background shows a high-resolution cross-sectional electron microscopy image of the superconducting Ge:Ga layer.

For further information see:

T. Herrmannsdörfer et al., Phys. Rev. Lett. **102**, 217 003 (2009), reprinted at pp. 22 - 25 of this Annual Report.

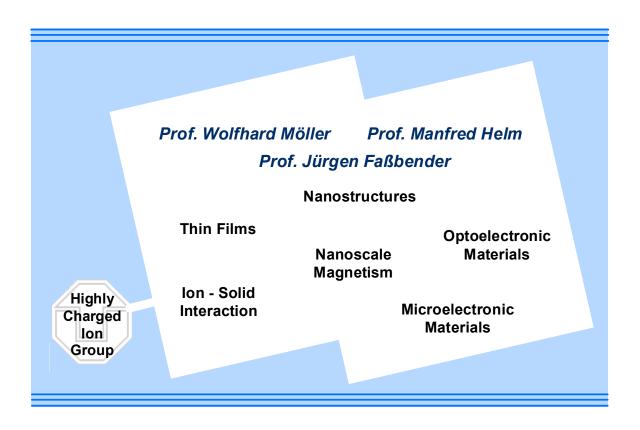
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# **P**reface by the **D**irectors

The Institute of Ion Beam Physics and Materials Research (IIM) is one of the six institutes of the Forschungszentrum Dresden-Rossendorf (FZD), and contributes the largest part to its Research Program "Advanced Materials", mainly in the fields of semiconductor physics and materials research using ion beams. The institute operates a national and international Ion Beam Center, which, in addition to its own scientific activities, makes available fast ion technologies to universities, other research institutes, and industry. Parts of its activities are also dedicated to exploit the infrared/THz free-electron laser at the 40 MeV superconducting electron accelerator ELBE for condensed matter research. For both facilities the institute holds EU grants for funding access of external users.



The diagram displays the presently six R&D topics of the institute, together with the associated Highly Charged Ion (HCI) Group of the TU Dresden. The research activities span a wide range of topics relevant for future information processing and energy technology, be it in the realm of nanoelectronics, optoelectronics, magnetoelectronics, spintronics, and future photovoltaics. Readers familiar with our institute will notice the appearance of a third director in the diagram, Prof. Jürgen Faßbender. He is the designated successor of Prof. Wolfhard Möller, who will retire in the course of the year 2010. In order to enable a smooth transition, Prof. Faßbender was instated, after a competitive selection process, as of Oct. 1, 2009, both as a director at the institute and as Professor for Applied Solid State Physics at the Technical University Dresden. He is a world-renowned expert for magnetic materials and structures and their modification by ion beams. Already since 2004 he was a group leader at the institute.

Highlights of last year's research are presented in this Annual Report through reprints of short papers that were published in leading international journals. In 2009, IIM staff published more than 190 papers in peer-reviewed journals, the highest number ever in its history. But since not only quantity counts, institute members were also co-authors of two publications in the prestigious journal "Nature Materials" and of five papers in Physical Review Letters, apart from about 60 more papers published at journals with impact factor >3.

2009 has again been an excellent year with respect to third party funding. Besides the continuation of the large project "Concepts of multifunctional memories" funded by the German Federal Government, the institute was successful in acquiring the large collaborative EU grant SPIRIT ("Support of Public and Industrial Research using Ion beam Technology") as a coordinator. This project constitutes a collaboration between the main ion beam laboratories in Europe and is granting transnational access to European users. Another essential part of funding arises from direct contracts with industry, including also local microelectronics and other high-tech industry. Thus also our funding spectrum reflects our span from successful basic science to relevant applied research.

An important milestone for the institute was the inauguration of a new 6 MV tandem accelerator on December 4, 2009, which was installed in a new, dedicated building. The machine will replace the outdated 5 MV Tandem for ion beam modification and analysis of materials, but additionally enable accelerator mass spectrometry (AMS). In relation to this, Dr. Silke Merchel, an AMS expert, joined the institute as a division head for ion beam analysis.

On the organizational side, FZD as a whole is preparing for its transition from being a member of the Leibniz Association of Research Institutes (WGL) to the Helmholtz Association of German Research Centers (HGF), which will formally take place in the beginning of 2011. This will require considerable changes in the way our research is funded, organized and administered. It will also open the way for new alliances and cooperations based on our scientific profile, and thus add to the stimulating atmosphere at the institute.

Finally we would like to cordially thank all partners, friends, and organizations who supported our progress in 2009. Special thanks are due to the Executive Board of the Forschungszentrum Dresden-Rossendorf, the Minister of Science and Arts of the Free State of Saxony, and the Minister of Education and Research of the Federal Government of Germany. Numerous partners from universities, industry and research institutes all around the world contributed essentially, and play a crucial role for the further development of the institute. Last but not least, the directors would like to thank all IIM staff for their efforts and excellent contributions in 2009.

Lo. flielle

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Prof. Wolfhard Möller

Prof. Manfred Helm

Prof. Jürgen Faßbender

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J. Seidel, L.W. Martin, Q. He, Q. Zhan, Y.H. Chu, A. Rother, M.E. Hawkridge, P. Maksymovych, P. Yu, M. Gajek, N. Balke, S.V. Kalinin, S. Gemming, F. Wang, G. Catalan, J.F. Scott, N.A. Spaldin, J. Orenstein, and R. Ramesh

Conduction at domain walls in oxide multiferroics

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Fano signatures in the intersubband terahertz response of optically excited semiconductor quantum wells

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A. Keller, A. Biermanns, G. Carbone, J. Grenzer, S. Facsko, O. Plantevin, R. Gago, and T. H. Metzger **Transition from smoothing to roughening of ion-eroded GaSb surfaces** Applied Physics Letters, Vol. **94**, Issue 19, Art.-No.: 193 103, 2009.

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S. Cornelius, M. Vinnichenko, N. Shevchenko, A. Rogozin, A. Kolitsch, and W. Möller Achieving high free electron mobility in ZnO:Al thin films grown by reactive pulsed magnetron sputtering

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A. Kanjilal, L. Rebohle, M. Voelskow, W. Skorupa, and M. Helm **Enhanced blue-violet emission by inverse energy transfer to the Ge-related oxygen deficiency centers via Er<sup>3+</sup> ions in metal-oxide semiconductor structures** Applied Physics Letters, Vol. **94**, Issue 5, Art.-No.: 051 903, 2009. Copyright 2009, American Institute of Physics

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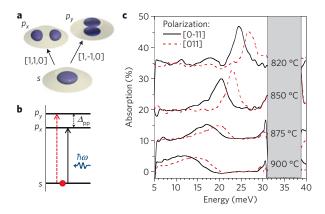
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# Long lifetimes of quantum-dot intersublevel transitions in the terahertz range

E. A. Zibik<sup>1\*†</sup>, T. Grange<sup>2\*†</sup>, B. A. Carpenter<sup>1</sup>, N. E. Porter<sup>1</sup>, R. Ferreira<sup>2‡</sup>, G. Bastard<sup>2</sup>, D. Stehr<sup>3</sup>, S. Winnerl<sup>3</sup>, M. Helm<sup>3</sup>, H. Y. Liu<sup>4\*</sup>, M. S. Skolnick<sup>1</sup> and L. R. Wilson<sup>1‡</sup>

Carrier relaxation is a key issue in determining the efficiency of semiconductor optoelectronic device operation. Devices incorporating semiconductor quantum dots have the potential to overcome many of the limitations of quantum-wellbased devices because of the predicted long quantum-dot excited-state lifetimes. For example, the population inversion required for terahertz laser operation in quantum-well-based devices (quantum-cascade lasers<sup>1,2</sup>) is fundamentally limited by efficient scattering between the laser levels, which form a continuum in the plane of the quantum well. In this context, semiconductor quantum dots are a highly attractive alternative for terahertz devices, because of their intrinsic discrete energy levels. Here, we present the first measurements, and theoretical description, of the intersublevel carrier relaxation in quantum dots for transition energies in the few terahertz range. Long intradot relaxation times (1.5 ns) are found for level separations of 14 meV (3.4 THz), decreasing very strongly to  $\sim$ 2 ps at 30 meV (7 THz), in very good agreement with our microscopic theory of the carrier relaxation process. Our studies pave the way for quantum-dot terahertz device development, providing the fundamental knowledge of carrier relaxation times required for optimum device design.

The predicted suppression of carrier relaxation rates in semiconductor quantum dots compared with quantum wells has motivated the study of quantum-dot carrier relaxation mechanisms and quantum-dot optoelectronic device development across a wide range of operating wavelengths<sup>3-5</sup>. The development of efficient devices based on intraband transitions operating at mid- and far-infrared wavelengths (terahertz range), such as quantum-cascade lasers, relies on a detailed knowledge of carrier relaxation processes. Direct measurements of intersubband relaxation times in quantum wells yield times of up to 500 ps in the terahertz range, showing a strong reduction to tens of picoseconds with increasing electron density in the excited subband<sup>6</sup>. This reduction is mainly due to electron-electron scattering in the upper sub-band<sup>7</sup> and heating of the two-dimensional electron gas, which permits efficient optical phonon scattering from the high-energy tail of the electron distribution<sup>6</sup>. In quantum dots these effects are suppressed because of the discrete nature of the energy levels. The potential for high-efficiency quantum-dot-based terahertz emitters has recently been highlighted by the record high-temperature operation of a quantumwell-based terahertz quantum-cascade laser in which extra lateral



**Figure 1** | **Intersublevel transitions in quantum dots. a**, Calculated wavefunctions for *s*-like ground and *p*-like first excited states in quantum dots. **b**, Schematic diagram of the energy levels of a quantum dot and allowed optical transitions. **c**, Normal-incidence absorption spectra (for two orthogonal linear light polarizations) of quantum-dot samples with different annealing temperatures measured at 10 K (the spectra are shifted for clarity).

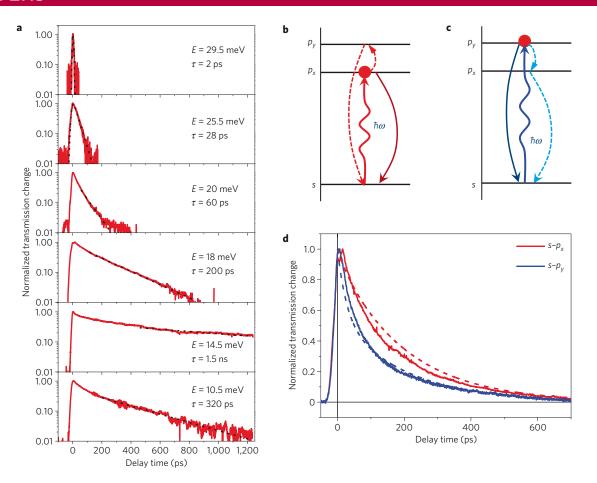
carrier confinement was provided by high magnetic fields in the growth direction<sup>8</sup>.

Self-assembled quantum dots (SAQDs) are an appealing solution for device incorporation. However, carrier relaxation has been studied over only a limited range of sublevel energy separations. Typically, the transition energy between the ground and first excited conduction band states lies in the range 40-60 meV for InGaAs SAQDs, above the Reststrahlen band  $(36 \text{ meV} \sim 9 \text{ THz})$ . Experimental studies<sup>9,10</sup> have demonstrated that the carrier relaxation in SAQDs occurs on a timescale of tens of picoseconds. This was explained in terms of anharmonicity of the polarons formed by the interaction between electrons and longitudinal optical phonons<sup>10–13</sup>. However, recent theoretical work based on a microscopic approach of polaron anharmonicity<sup>13</sup> has predicted a significantly longer lifetime for dots with sublevel spacing below the longitudinal optical phonon energy. Here, we report pump-probe investigations of the relaxation dynamics in InGaAs SAQDs in the few terahertz (sub-Reststrahlen) range. We show that the relaxation time shows a marked increase of three orders of magnitude over a narrow ( $\sim 15 \text{ meV}$ ) energy range up

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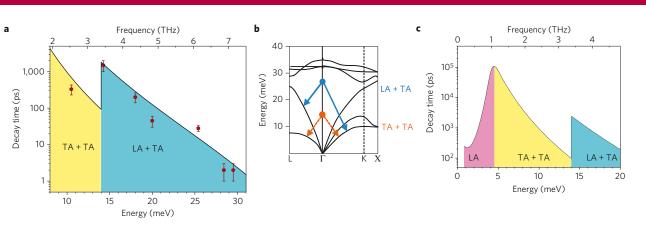
**Figure 2** | **Population dynamics in quantum dots. a**, Normalized time evolution of the  $s-p_x$  intersublevel transmission change at different excitation energies at 10 K: pump-probe measurements at excitation energy *E* (red solid lines) and exponential fit with time constant  $\tau$  (black dashed lines). **b**,**c**, Schematic of the possible relaxation paths after exciting either the  $p_x$  (**b**) or  $p_y$  (**c**) state. The solid arrows show the direct *p*-to-*s* intersublevel relaxation process, and the dashed arrows illustrate the relaxation path through acoustic-phonon-assisted transitions between  $p_x$  and  $p_y$  states. **d**, Pump-probe curves (solid line) for excitation into either the  $p_x$  or  $p_y$  state at the same energy of 18 meV and simulation (dashed line) taking into account the thermalization between the *p* states by acoustic phonon emission and absorption.

to a maximum of 1.5 ns, and we discuss model calculations that enable us to clearly identify the underlying mechanism responsible for the energy relaxation.

The investigated samples comprise 80 layers of SAQDs (areal density  $\sim 4 \times 10^{10}$  cm<sup>-2</sup>) separated by 50-nm-wide GaAs barriers, thus preventing both structural and electronic coupling between the layers. The dots of each layer are populated with electrons transferred from a Si-doped layer grown  $\sim$ 2 nm below them. The doping density was controlled in such a way as to charge each dot with at most one electron (see ref. 10 and references therein for more details). The fundamental intersublevel transitions occur in the mid-infrared region (around 50 meV) and involve one ground (s) state and a doublet  $(p_x \text{ and } p_y)$  of excited levels (Fig. 1a). The absorption spectrum shows two inhomogeneously broadened peaks shifted by a small anisotropic splitting  $\Delta_{pp} \sim 3.5 \text{ meV}$ , with each transition related to light polarized along either the [1, 1, 0] (s-to- $p_x$  transition) or [1, -1, 0] (s-to- $p_y$  transition) crystallographic direction (different effects have been invoked to explain the splitting of the  $p_x$  and  $p_y$  states: in-plane shape anisotropy<sup>14</sup>, piezoelectric field effects<sup>15</sup> and atomistic symmetry<sup>16</sup>). To decrease the transition energy below the longitudinal optical phonon energy  $\hbar\omega_{\rm LO}$ , we used the well-established method of post-growth thermal annealing<sup>17–19</sup> (see the Methods section). The absorption spectra of the annealed samples with temperatures ranging from 800 to 900 °C are shown in Fig. 1c (the absorption spectrum for the quantum-dot sample annealed at 800 °C is not shown because it overlaps strongly with the Reststrahlen band). The absorption spectra show several interesting features: (1) the absorption of annealed-sample peaks below the Reststrahlen band, with increasing redshift for increasing annealing temperature; (2) the material interdiffusion induced by the annealing does not eliminate the anisotropic splitting, which diminishes only slightly to about 2 meV; and (3) the lines become broader, indicating an enhancement of size and composition inhomogeneity of the dots during the annealing. These results clearly demonstrate that post-growth annealing provides a well-controlled means to obtain dots with average intersublevel spacing  $\Delta_{\rm sp}$  well below the Reststrahlen band (for example,  $\Delta_{\rm sp} \sim 10$  meV (2.4 THz) for the 900 °C sample).

The relaxation dynamics of excited carriers in quantum dots are studied using degenerate terahertz pump–probe spectroscopy. A free-electron laser 'FELBE' delivering linearly polarized picosecond pulses was used in the experiments<sup>20</sup>. An initial pulse excites the ground state to the  $p_x$  or  $p_y$  transition (the in-plane light polarization is chosen parallel to either of the crystallographic axes), and a second pulse probes the recovery of the absorption signal for this same transition as a function of the time delay between the pump and probe pulses. The results of low-temperature (10 K) pump–probe measurements for different excitation energies are shown in Fig. 2a. We observe a very strong variation in the recovery of the absorption bleaching when changing the transition energy. For transition energies close to the longitudinal optical

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**Figure 3** | **Intersublevel lifetimes in the terahertz region. a**, Variation of the decay time with the transition energy below the Reststrahlen band: experimental data (symbols) and theoretical calculations (solid line). The error bars are determined by the uncertainty in the mono-exponential decay fit. **b**, Schematic diagram of the different polaronic decay channels. **c**, Calculation of the relaxation time in an In<sub>0.1</sub>Ga<sub>0.9</sub>As/GaAs quantum dot of varying diameter showing the transition between electron relaxation by emission of one acoustic phonon (below ~1THz) and the polaron anharmonic decay (above ~1THz).

phonon energy, the decay curve is mono-exponential with a short time constant varying from  $2 \pm 1$  ps for  $\sim 30$  meV ( $\approx 7.2$  THz) up to  $60 \pm 5$  ps for 20 meV ( $\approx 4.8$  THz). With a further decrease of the transition energy, the time dynamic presents a bi-exponential decay. The mono-exponential decay time as well as the extracted long time component in the bi-exponential case is plotted as a function of the transition energy in Fig. 3a. The decay time increases strongly with decreasing transition energy up to  $1.5 \pm 0.5$  ns at  $\sim 15$  meV ( $\approx 3.6$  THz).

We assign the mono-exponential decay as well as the long time component in the bi-exponential case to the decay from the p to the s shells of the quantum-dot levels, whereas the short time component results from population interchanges among the p states  $(p_x \text{ and } p_y)$  by absorption or emission of acoustic phonons with energy corresponding to the anisotropic splitting  $\Delta_{\rm pp} \approx 2 \,{\rm meV}$  (see Fig. 2b). This interpretation is corroborated by further measurements in the bi-exponential regime, using light with opposite in-plane polarization to probe also the  $p_y$ transition (see Fig. 2c). The initial decay is faster when exciting an upper  $(p_y)$  state than a lower  $(p_x)$  one, as expected because at low temperature, phonon emission is much more probable than absorption. This interpretation is confirmed in Fig. 2d when comparing the experimental data to simulations (dashed lines) that include the calculated rates for the thermal exchange between the  $p_x$ and  $p_{y}$  levels. This model also provides a clear explanation for the absence of bi-exponential behaviour for energies above  $\sim 20 \text{ meV}$ , because the  $p_x$  to s relaxation time is shorter than or comparable to the  $p_x - p_y$  thermalization time.

Let us focus now on the interpretation of the strong energy dependence of the *p*-to-*s* intersublevel relaxation time, reported in Fig. 3a as a function of the intersublevel detuning. We expect that the scattering mechanisms responsible for the energy relaxation in bulk and quantum wells are either forbidden (elastic) or greatly reduced (inelastic) in quantum dots, owing to the discrete nature of the energy levels<sup>21,22</sup>. Instead, as we show below, the anharmonic decay of quantum-dot polarons (electrons strongly coupled to longitudinal optical phonons<sup>23–25</sup>) is the dominant relaxation mechanism below the longitudinal optical phonon energy down to the 1 THz regime. This is the same physical mechanism that has been shown previously to lead to efficient relaxation at energies above the longitudinal optical phonon energy<sup>9–13</sup> (between 20 and 65 ps in the 40–60 meV energy range<sup>10</sup>), but producing here a much higher sensitivity of the cooling rate with the amount of relaxed energy. This demonstrates the generality of the polaron formation and decay mechanism for energy relaxation in systems with three-dimensional quantization of the electronic states. We have calculated the intersublevel relaxation time using the microscopic model of anharmonic polaron decay developed in ref. 13 (see the Methods section). The Fröhlich polar coupling between electrons and longitudinal optical phonons is taken into account and leads to the formation of polaron states. The anharmonic terms of the lattice vibrational potential energy then trigger the polaron disintegration into a two-phonon state. We incorporate the three possible channels for two-phonon disintegration allowed in the studied energy range: LA + LA, LA + TA and TA + TA, where LA corresponds to longitudinal acoustic and TA to transverse acoustic phonons, as shown schematically in Fig. 3b. The calculated lifetime is plotted in Fig. 3a as a function of the transition energy. For each decay channel, the relaxation rate strongly decreases with decreasing intersublevel energy. Three factors mainly contribute to this behaviour: the reduction of the available density of two-acoustic-phonon states, the decreasing strength of the anharmonic couplings involved and the increasing detuning with respect to the longitudinal optical phonon energy. We find that the LA + TA channel dominates for intersublevel energies below  $\sim$ 33 meV (8 THz) (the longitudinal acoustic Debye energy plus the transverse acoustic Debye energy) but larger than twice the transverse acoustic Debye energy (taken as 7 meV here). Below  $\sim$ 14 meV, the TA + TA path becomes available, which explains the non-monotonic variation in the calculated and measured times around this energy.

In addition, we carried out pump-probe measurements at a temperature higher than 10 K at energies of 25.4 meV and 14.25 meV (see Supplementary Information). At transition energies around 25.4 meV, the temperature dependence of the intersublevel lifetime, which decreases from  $28 \pm 2$  ps at 10 K to  $8 \pm 3$  ps at 170 K, can be well fitted considering polaron decay into two acoustic phonons (TA + LA). For a pump-probe energy of 14.25 meV, the intersublevel lifetime was measured to decrease from  $1.5 \pm 0.5$  ns at 10 K to  $560 \pm 50$  ps at 30 K and  $260 \pm 30$  ps at 50 K. At this energy, a polaron decay time of 60 ps is expected at room temperature.

The overall close agreement between experiment and theory demonstrates that the energy relaxation in annealed SAQDs is governed by the anharmonic mechanism and becomes strongly inhibited when the level spacing decreases to the order of a few terahertz. However, it is known that in lithographically defined quantum dots, where the *s*–*p* transition energy is below 1 THz, an efficient cooling of carriers is ensured by the emission of one acoustic phonon<sup>26,27</sup>. To show the crossover between the two regimes, we have calculated the intersublevel lifetime for

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transition energies between 1 and 5 meV in quantum dots with fixed composition  $(In_{0.1}Ga_{0.9}As/GaAs)$  and fixed height (5 nm) and varying diameter. As shown in Fig. 3c, one-phonon emission does provide an efficient relaxation channel in the sub-terahertz domain, but quickly becomes inoperative for larger intersublevel energies. As a consequence of this acoustic-phonon bottleneck, the anharmonic polaron decay mechanism governs the *p*-to-*s* energy relaxation in SAQDs down to the 1 THz regime.

Our studies demonstrate that carrier cooling in quantum dots in the terahertz domain is due to the mechanism of anharmonic polaron decay, and that its rate increases by about three orders of magnitude when the energy spacing varies between 7 and 3.5 THz. This extreme sensitivity is a consequence of the polaronic nature of the relaxation processes in fully confined quantum dots. Our results provide important new information for the design of quantum-dotbased terahertz optoelectronic devices. The long carrier relaxation times, and the ability to controllably alter the times by varying the sublevel spacing, provide strong motivation for the production of, for example, terahertz lasers, which require the inhibition of the upper-to-lower relaxation and a fast emptying of the lower level.

#### Methods

Annealing. The annealing was carried out in a rapid thermal annealer with temperatures ranging from 800 to 900 °C. The annealing time was fixed to 4 min. During the annealing process, the samples were sandwiched between two GaAs wafers to prevent As out-diffusion from the sample surface and were kept in a nitrogen ambient environment. We prepared several samples annealed at 800, 820, 850, 875 and 900 °C to cover the investigated spectral range.

Experimental set-up. The degenerate terahertz pump-probe measurements were carried out using the Free Electron Laser Facilities 'FELBE' located at Forschungszentrum Dresden-Rossendorf (Germany)20. The free-electron laser delivered linearly polarized picosecond pulses with a repetition rate of 13 MHz tunable in the 18–250  $\mu m$  spectral range. The laser beam was split into pump and probe beams with pulse energies of  $\sim$ 1 and  $\sim$ 0.01 nJ correspondingly. The pump beam was modulated at a frequency of  $\sim$ 330 Hz using an optical chopper and a variable time delay between the pump and probe was introduced. An off-axis parabolic mirror of ~11 cm effective focal length was applied to focus both the pump and the probe beam onto the same spot on the sample, which was held in a He-flow cryostat with diamond windows. The pump and probe beams were hitting the sample at about a 10° angle to the normal. The change of the intensity of the probe beam after the sample was detected using a Ge:Ga detector and a lock-in amplifier, which was locked to the modulation frequency of the pump beam. Owing to the high repetition rate and stability of the laser, we were able to resolve an absorption change as small as ~0.05%. A mirror-based polarization rotator was also incorporated into the incident beam path to enable us to pump and probe either *s*– $p_x$  or *s*– $p_y$  quantum-dot transitions.

Modelling of the interdiffused quantum dots. We model both as-grown and annealed InxGa1-xAs/GaAs dots as truncated cones with in-plane (top) basis radius R(R/2), height h and uniform In fraction x in the dot region, residing on a thin wetting layer of the same In concentration, and surrounded by GaAs. The parameters of the as-grown quantum dots ( $R_0 = 122 \text{ A}, h_0 = 49 \text{ A}, x_0 = 0.55$ ) were chosen to fit both intersublevel and interband transition energies ( $\Delta_{sn} \approx 48 \text{ meV}$ and the photoluminescence peak is centred at 1,109 meV). For annealed dots, we assume that: (1) the In quantity inside the dot and the aspect ratio R/h are both preserved during the thermal process, which gives  $R = R_0 (x_0/x)^{1/3}$  for the annealed dot; (2) the confining potential depth  $\Delta V$  varies linearly with the In content:  $x_0 \Delta V = x \Delta V_0$ . Despite its simplicity, this model captures the main aspects of the interdiffusion, namely, an increase of the quantum-dot effective size and a decrease of its confinement potential, which both lead to a decrease of  $\Delta_{\rm sp}$ . We calculate that  $\Delta_{\rm sp}$  decreases from 48 to 10 meV on increasing the quantum-dot radius from  $R \sim 12$  to  $\sim 25$  nm with a corresponding decrease of the In fraction from  $x \sim 0.55$  to 0.07. The corresponding electron wavefunctions are used to calculate the matrix elements related to the coupling of the confined electron to both the acoustical and optical phonons. In particular, the Fröhlich coupling  $V_{\rm sp}$  (see ref. 13 for definition) varies from 3.2 meV for  $\Delta_{\rm sp} = 30$  to 2.5 meV when  $\Delta_{sp} = 10$  meV.

Anharmonic decay of polarons. The intersublevel relaxation time is calculated using the microscopic model in ref. 13. We use channel-dependent Gruneisen constants to fit the present data:  $\gamma = 1.3$  for the LA + LA channel (as in ref. 13), and  $\gamma = 4.0$  for the TA + TA and TA + LA channels. The transverse acoustic and longitudinal acoustic Debye energies are taken respectively as 7 and 26 meV. In the present work, we take into account the non-resonant Fröhlich couplings between

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the ground state and the longitudinal optical phonon replicas of the *p* states. As a consequence, we find that the polaron decay time  $\tau$  due to the anharmonic couplings is given by:

$$\frac{h}{h} = \frac{4\Delta_{\rm sp}^2}{(\hbar\omega_{\rm LO} + \Delta_{\rm sp})^2} \frac{V_{\rm sp}^2}{(\Delta_{\rm sp} - \hbar\omega_{\rm LO})^2 + V_{\rm sp}^2} \Gamma^{\rm ph}(\Delta_{\rm sp}) \tag{1}$$

where the function  $\Gamma^{\rm ph}(E)$  is defined in ref. 13. The intersublevel transition energy  $\Delta_{\rm sp}$  corresponds to the optically probed transition energy between *s*-like and *p*-like polaron states with a dominant zero-phonon component. This formula is used here instead of formula (6) used in ref. 13, which is valid only close to the longitudinal optical phonon resonance. The first term in the above equation arises from the interference between the resonant and the non-resonant couplings. This interference is found to be destructive for intersublevel transition energies that are small compared with the longitudinal optical phonon one ( $\Delta_{\rm sp} \ll \hbar \omega_{\rm LO}$ ). This effect contributes also to the inhibition of the polaron decay mechanism at low energies, in particular in the calculation shown in Fig. 3c.

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#### Author contributions

E.A.Z. and L.R.W. conceived the experiments; E.A.Z., L.R.W., B.A.C., N.E.P., D.S. and S.W. carried out the experiments; T.G., R.F. and G.B. made the theory and modelled the experiments; E.A.Z. and T.G. analysed the data; L.R.W., R.F., E.A.Z. and M.S.S. supervised the project; H.Y.L. grew the samples; T.G., E.A.Z., R.F. and L.R.W. wrote the paper with major input and edits from M.S.S. and M.H. All authors discussed the results and commented on the manuscript.

#### **Additional information**

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# Conduction at domain walls in oxide multiferroics

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Domain walls may play an important role in future electronic devices, given their small size as well as the fact that their location can be controlled. Here, we report the observation of room-temperature electronic conductivity at ferroelectric domain walls in the insulating multiferroic BiFeO<sub>3</sub>. The origin and nature of the observed conductivity are probed using a combination of conductive atomic force microscopy, high-resolution transmission electron microscopy and first-principles density functional computations. Our analyses indicate that the conductivity correlates with structurally driven changes in both the electrostatic potential and the local electronic structure, which shows a decrease in the bandgap at the domain wall. Additionally, we demonstrate the potential for device applications of such conducting nanoscale features.

orrelated oxide systems are an exciting and challenging area of condensed-matter research, with their interacting and competing charge, spin, orbital and lattice degrees of freedom forming new electronic and magnetic phases<sup>1,2</sup>. These phases can be controlled through stress, optical excitation and electric and magnetic fields and have great potential for applications in the fields of spintronics, information storage and communications. Among the correlated oxides, the multiferroics, which show more than one type of ferroic order in the same phase, are attracting particular interest<sup>3-6</sup>. The defining characteristic of a ferroic material is an order parameter (electric polarization in ferroelectrics, magnetization in ferromagnets or spontaneous strain in ferroelastics) that has different, energetically equivalent orientations, the orientation of which can be selected using an applied field. This often leads to the appearance of domains of differently oriented regions, separated by domain walls, coexisting in a sample<sup>7</sup>. Such domain walls will become more technologically important as the dimensions of individual elements in devices continue to shrink.

Although the morphology and properties of domains and their walls have been studied for more than 50 years, in recent times there has been increasing focus on novel functionality at domain walls<sup>8–12</sup>. For example, it has been predicted theoretically that the ferroelectric walls in magnetoelectric multiferroics can be ferromagnetic even if the domains themselves are antiferromagnetic<sup>9–11</sup>. Conversely, spin rotation across ferromagnetic domain walls in insulating ferromagnets can induce a local polarization in the walls of an otherwise non-polar material<sup>5,12</sup>. Experimentally, unusual functional properties of domain walls have also been observed: preferential doping along domain walls has been reported to induce two-dimensional superconductivity in WO<sub>3-x</sub> (ref. 13) and enhanced resistivity in phosphates<sup>14</sup>, whereas in paraelectric, non-polar SrTiO<sub>3</sub> the ferroelastic domain walls seem to be ferroelectrically polarized<sup>15</sup>. Local probes, particularly those based

on scanning probe microscopy<sup>14</sup>, are invaluable in observing such effects at the nanoscale.

Here we describe the observation of a new behaviour room-temperature electrical conductivity—at ferroelectric domain walls in the prototypical multiferroic BiFeO<sub>3</sub> (BFO). BFO is a room-temperature G-type antiferromagnet ( $T_{\rm N} \sim 650$  K) and a rhombohedral ferroelectric ( $T_{\rm C} \sim 1,103$  K), with a large spontaneous ferroelectric polarization (~90  $\mu$ C cm<sup>-2</sup>) along the pseudocubic 111 directions<sup>16,17</sup>. Such rhombohedral ferroelectrics possess 71°, 109° and 180° domain walls forming on {101}, {100} and planes that satisfy the requirement that  $\pm h \pm k + l = 0$ , respectively<sup>18</sup>. All three wall orientations have been observed in BFO (refs 19,20).

Epitaxial BFO films (~100 nm thick) were grown using laser molecular-beam epitaxy in (111), (110) and (100) orientations, using carefully controlled single-crystal SrTiO<sub>3</sub> substrates. A thin 50 nm layer of epitaxial SrRuO<sub>3</sub> was used as a bottom electrode for electrical-contact purposes. Ferroelectric domains were imaged using piezoresponse force microscopy (PFM) as described previously<sup>19</sup>. Controlled ferroelectric domain patterns were written using PFM by applying a d.c. voltage to the probe tip. Local electrical conductivity was measured using high-resolution conductive atomic force microscopy (c-AFM) (details of measurements are provided in the Methods section) by applying a bias voltage (below the polarization switching voltage) between the conductive AFM tip and the bottom electrode of the sample.

100-nm-thick epitaxial films grown on (110) surfaces show a two-variant ferroelectric domain structure in the as-grown states with average domain sizes between 5 and 10  $\mu$ m. On electrical switching at high fields, all three variations of domain walls can be created<sup>21</sup>; therefore, we begin our study on these (110)-oriented films. The typical morphology is shown in Fig. 1a. We measured the RMS roughness of the films to be ~0.5 nm and there are no observable surface features, either before or after switching,

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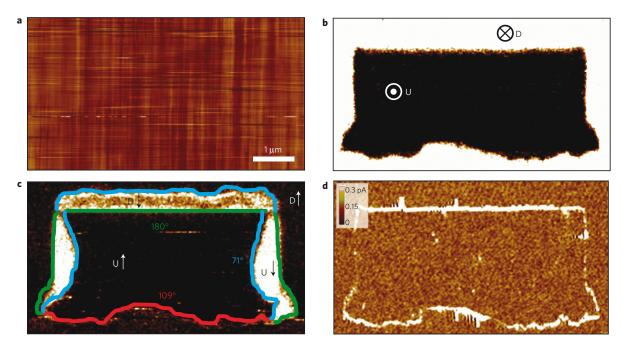


Figure 1 | Conductivity at different wall types. a, Topography of a BFO(110) thin film, r.m.s. roughness of 0.5 nm. b, Out-of-plane PFM image of a written domain pattern in a monodomain BFO(110) film showing the out-of-plane polarization component of the domains to be either down, labelled as 'D' (white), or up, labelled as 'U' (black). c, In-plane PFM image of a written domain pattern in a monodomain BFO(110) film showing all three types of domain wall, that is, 71° (blue), 109° (red) and 180° (green), as inferred from the combination of both out-of-plane and in-plane PFM images. In these images, both the out-of-plane (U or D) component and the in-plane projection of the polarization direction (shown as an arrow) are also labelled. d, Corresponding c-AFM image showing conduction at both 109° and 180° domain walls; note the absence of conduction at the 71° domain walls.

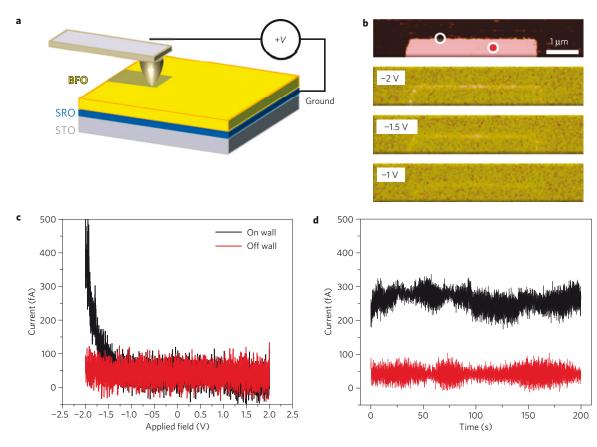
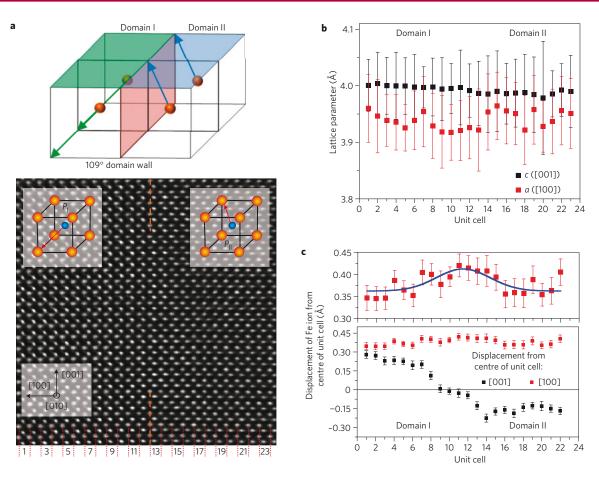


Figure 2 | Conduction at domain walls. a, Schematic illustration of the experimental c-AFM set-up. b, (Top) Out-of plane PFM image of a written 180° domain in a monodomain BFO(110) sample and (lower) corresponding c-AFM current maps for -1, -1.5 and -2 V sample bias made with a Pt-coated tip. c, I-V curves taken both on the domain wall (black) and off the domain wall (red) reveal Schottky-like behaviour. d, Time dependence of the current both on the wall (black) and off the wall (red) at an applied sample bias of -2 V. Results are qualitatively similar for N-doped diamond tips.

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**Figure 3** | **Structural analysis of domain walls. a**, Schematic diagram of 109° domain wall and exit-wave-reconstructed HRTEM image of a 109° domain wall imaged along the [010] zone axis. **b**, Extracted *a* and *c* lattice parameters for each unit cell across the domain wall. **c**, Extracted Fe-ion displacement relative to the Bi lattice for each unit cell across the domain wall. A close-up (upper panel) reveals an increase in the component of polarization perpendicular to the domain wall.

corresponding to the conducting features. In Fig. 1c, we show the in-plane PFM image of a written domain pattern controlled to have all three domain-wall types. The complicated domain shapes occur only when the high voltages required to stabilize all three domain-wall variants are applied<sup>21</sup>. The various domain-wall types are determined using both in-plane (Fig. 1c) and out-of-plane (Fig. 1b) PFM images and are labelled accordingly. Our main result is shown in the corresponding c-AFM trace (Fig. 1d): the occurrence of electrical conduction at 109° and 180° domain walls, and the absence of conduction at 71° domain walls. BFO films grown on (001)- and (111)-oriented substrates also consistently show conduction at 109° and 180° domain walls (Supplementary Information, Fig. S1); in no cases do 71° domain walls show conduction within the resolution of our measurements.

Next we discuss in detail our c-AFM measurements on the (110)-oriented BFO films. A schematic diagram of the experimental set-up is shown in Fig. 2a. The spatial resolution of the technique is limited by the tip radius of ~20 nm. Figure 2b (top panel) shows a PFM image of two domains separated by a 180° domain wall. The corresponding c-AFM images (lower panels) show enhanced conduction at the domain wall for applied bias voltages of -1 to -2 V. Current–voltage (I-V) curves (Fig. 2c) show resistive behaviour within the domain (red data) and Schottky-like behaviour suggesting activated conduction at the domain wall (black data). We repeated our I-V measurements with a number of different c-AFM tip materials—including Pt and N-doped diamond—and found similar Schottky-like behaviour with slightly shifted conduction onsets. Furthermore, the current is persistent

#### Table 1 | Electronic structure at ferroelectric domain walls.

Domain wall type (°)	Electrostatic potential step (eV)	Change in bandgap (eV)
71	0.02	0.05
109	0.15	0.10
180	0.18	0.20

Calculated values of the potential step and reduction in bandgap at all three domain-wall types.

over a timescale of at least 3 min, which is limited by the drift in our scanning system (Fig. 2d). These time-dependent data indicate that the origin of this current is not displacement of domain walls. Additional details of conduction at domain walls are provided in the Supplementary Information (Supplementary Information, Fig. S2). Ultrahigh-vacuum-based c-AFM measurements were used to further probe the nature of conduction and I-V characteristics of the conducting domain walls—including the observation of enhanced current values.

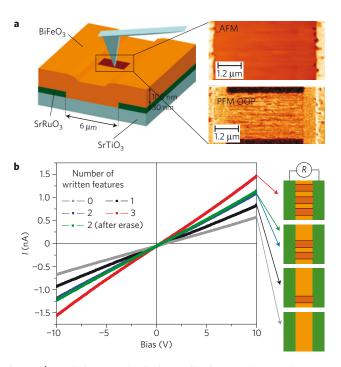
To understand the observed electrical conductivity, we performed a combined transmission electron microscopy (TEM) and density functional theory (DFT) study of the domain-wall structure and properties. The structure of domain walls in ferroelectric perovskites and related materials has been the focus of several recent DFT (refs 18,22) and TEM (refs 23–25) studies, and the structural distortions associated with ferroelectric domain

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walls are now reasonably well characterized. However, studies of the associated electronic properties are in their infancy<sup>26</sup>. We chose the 109° domain wall (shown schematically in Fig. 3a) for our study because we do not obtain conduction at 71° domain walls and because imaging of 180° domain walls with high-resolution TEM (HRTEM) presents practical problems in terms of locating the wall. (For completeness, HRTEM images of 71° domain walls are shown in Supplementary Information, Fig. S3.) We use (001)-oriented samples for the TEM analysis, because we can easily control the density of 109° domain walls during growth for this orientation. TEM images were acquired using the exit-wave reconstruction approach to eliminate the effects of objective-lens spherical aberrations; such images can be directly interpreted in terms of the projection of the atomic columns<sup>27</sup>. A representative image for such a 109° domain wall taken along the [010] zone axis of a cross-sectioned BFO/SRO/STO(001) film, where SRO is SrRuO<sub>3</sub> and STO is SrTiO<sub>3</sub>, is shown in Fig. 3a. Analysis of this image enables us to determine the lattice parameter in the plane of the film (a) ([100]) and the lattice parameter out of the plane of the film (c) ([001]) (Fig. 3b). As expected, the in-plane lattice parameter is slightly smaller and the out-of-plane lattice parameter larger than the values in bulk BFO (3.96 Å; ref. 16) owing to the strain inherent in the epitaxial films. In addition, we find that both the in-plane and out-of-plane film lattice parameters are unchanged in the vicinity of the domain wall. We also extract the relative displacement of the Fe ion with respect to the Bi sublattice, and resolve it into components parallel ([001]) and perpendicular ([100]) to the domain wall (which lies in a (100) plane running vertically across the image and labelled with a dashed orange line) by quantitative analysis of the HRTEM data (Fig. 3c); this distance is representative of the local polarization. The component of the displacement parallel to the domain wall (along [001]) decreases in magnitude to zero at the centre of the domain wall before changing to the same magnitude (but opposite sign) on the other side of the wall, reflecting the change in polarization orientation of the domain. Interestingly, the perpendicular displacement component (along [100]) shows a small increase at the domain wall; we return to the implications of this later. Similar studies were completed on a 71° domain wall as well (Supplementary Information, Fig. S2). Again, only minor variation in lattice parameters was observed across the domain wall. In this case a similar step in Fe-ion displacement is observed parallel to the domain wall, but we are unable to resolve a step in the perpendicular component across the wall.

To investigate the influence of these structural changes on the electronic properties, we next performed a density functional study of the structure and electronic properties for all three ferroelectric domain-wall variants (details in the Methods section). First we performed full structural optimizations of the ionic positions with the lattice parameters fixed to their experimental bulk values; in particular, the oxygen polyhedral rotations around the polar axis, which are known to have a profound effect on both the magnetic<sup>28</sup> and electronic<sup>29</sup> properties and cannot be easily extracted from the HRTEM data, were accurately calculated. As the sense of the oxygen rotations around the polar axis is independent of the direction of polarization along the axis<sup>28</sup> we studied two scenarios: first we initialized the sense of rotation to be continuous across the domain boundary and second we changed the rotation sense when the polarization direction changed. As expected, we found that domain walls with continuous oxygen rotations are considerably lower in energy, because this avoids formation of an antiphase boundary associated with the octahedral rotations. In addition, we investigated domain-wall configurations centred at both the Bi-O and Fe-O planes and found the Bi-O walls to be slightly lower in energy, confirming the findings of the HRTEM analysis. Our lowest-energy calculated configuration for the 109° domain wall has a domain-wall energy of 206 mJ m<sup>-2</sup>.

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**Figure 4 | Proof of concept for device application. a**, Schematic illustration (left) of in-plane electrode structure (green) and how scanning probe tips can be used to controllably create conductive domain-wall features between electrodes. The images on the right show AFM (top) and out-of-plane PFM (bottom) contrast for this written domain area on a BFO(110) sample. **b**, *I*-*V* characteristics of the devices measured between the two in-plane electrodes show that the current can be incrementally controlled through creating or erasing the conducting domain walls.

To confirm that our calculated structure is consistent with our TEM data, we analysed the layer-by-layer polarization, defined as the sum over the bulk Born effective charges<sup>29</sup> multiplied by the displacements of the ions from their centrosymmetric reference positions in each layer. As expected, the local polarization in the middle of the domain is close to the value calculated for bulk BFO using the same computationally and experimentally determined lattice parameters ( $\sim$ 93 µC cm<sup>-2</sup>; refs 29–32), confirming that our supercell is large enough to capture the essential physics. Consistent with our TEM analysis, we find an abrupt change in the parallel polarization component across the domain wall and a small change in the normal component at the domain wall.

Our calculations indicate that this small change in the normal component of the polarization across the 109° domain wall leads to a step in the electrostatic potential (planar and macroscopically averaged) of 0.15 eV across the domain wall (Table 1); a similar step was computed and explained previously across 90° domain walls in PbTiO<sub>3</sub> (ref. 22). Such a potential step should enhance the electrical conductivity by causing carriers in the material to accumulate at the domain wall to screen the polarization discontinuity. Our calculations for the 180° domain wall also yield a variation in the normal component of the polarization, and a corresponding potential step of 0.18 eV (Table 1). The normal component results from the polarization rotating towards successive adjacent corners of the perovskite unit cell, through a 71° and than a 109° change in the polarization direction before reaching the reversed polarization<sup>16</sup>. This behaviour is in striking contrast to the 180° polarization reversal in tetragonal ferroelectrics<sup>22</sup>, where the polarization changes in only one direction within the wall plane and no normal component occurs. The 71° wall, however, exhibits no anomaly in the perpendicular component of the ion displacement at the centre of the wall, again consistent with our

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TEM data, and therefore exhibits a negligible electrostatic potential step at the domain wall (Table 1).

Finally, we calculate the electronic properties of our structurally optimized domain walls, in particular by comparing the layer-by-layer densities of states in the domain wall and mid-domain regions. Within the central region of the domain, we find, as expected, that the local density of states resembles that of bulk BFO, and the local Kohn-Sham bandgap is equal to the value of 1.3 eV obtained for bulk BFO with the same choice of Hubbard parameter U and exchange interaction J, 3 and 1 eV, respectively29. (We emphasize that, although the DFT Kohn-Sham bandgaps do not correspond to experimental bandgaps, changes in DFT gaps caused by changes in bandwidth as a consequence of small changes in structure for the same DFT implementation are qualitatively meaningful.) As the domain wall is approached, we find that the changes in the structure do indeed cause changes in the band width and the positions of the band edges. This leads in the  $109^{\circ}$  ( $180^{\circ}$ ) case to a 0.1 eV (0.2 eV) reduction in the bandgap in the domain-wall layer from the mid-domain calculated value of 1.3 eV (Table 1). For activated conduction at room temperature, such a change in bandgap, or in band-edge offset relative to the Fermi energy of the tip, should lead to considerable changes in conductivity. Consistent with its absence of conduction, the reduction in bandgap in the  $71^{\circ}$  case is smaller (0.05 eV) (Table 1). Interestingly, the magnitude of the bandgap reduction is sensitive to the details of the lattice parameters used in the calculation; if we constrain the lattice parameters to the bulk values, the changes in the bandgap are larger than those for the relaxed structures given in Table 1. This suggests that band-structure changes at domain walls might be tunable by epitaxial strain.

Our conductivity measurements, TEM analysis and DFT calculations therefore suggest two mechanisms, which probably combine to yield the observed conductivity at the 109° and 180° domain walls: (1) an increased carrier density as a consequence of the electrostatic potential step at the wall and (2) a decrease in the bandgap within the wall and corresponding reduction in band offset with the c-AFM tip. Both factors are the result of structural changes at the wall.

The potential of these conducting domain walls for possible device applications is illustrated in Fig. 4. By creating a simple device structure consisting of in-plane electrodes of SRO separated by a  $6\,\mu\text{m}$  spacing (Fig. 4a), we can measure the I-V characteristics of BFO films and domain walls macroscopically. Additionally, the SRO provides nearly Ohmic contacts with the BFO films and thus we can gain further insight into the conduction of the walls in the gap, without any interference from the AFM tip during the measurement process. Monodomain (110)-oriented BFO films were grown on top of the SRO in-plane electrode device structures on STO(110) substrates. Conducting domain-wall features (here are shown 180° domain walls, Fig. 4a, right) that connect the two in-plane electrodes were written using PFM. Again, no morphological surface features were observed that correspond to the written domain pattern. I-V measurements (Fig. 4b) reveal a step-like increase in the measured current between the two in-plane electrodes on addition of a controlled number of conducting domain walls. The steps in conduction are essentially equidistant, increase proportionally to the total number of domain walls written and show completely reversible behaviour on erasing a given feature. I-V curves for zero, one, two and three domain features are shown in Fig. 4b. Such material functionality has potential application in both logic and memory applications, as the wall location (and hence electronic conduction) can be precisely controlled on the nanoscale. This demonstrates a possible rewritable, multiconfiguration device set-up that uses nanoscale conductive channels (that is, conducting domain walls). Finally, on the basis of a simple sheet-resistance model, we can estimate the resistivity of a

single domain wall in BFO to be of the order of  $1-10 \Omega$  m, which is between five and six orders of magnitude lower than for bulk BFO.

In summary, our results show that ferroelectric domain walls in multiferroic BFO show unusual local electronic transport behaviour that is quite different from that in the bulk of the material or in conventional ferroelectrics. The conductivity is consistent with our observed changes in structure at the domain wall and can be activated and controlled on the scale of the domain wall width—about 2 nm in BFO. The present work represents a 'proof of concept' that domain walls are discrete functional entities, which may be addressed and sensed, suggesting potential utility in novel nanoelectronic applications.

#### Methods

Nanoscale conductivity measurements were performed on a Digital Instruments Nanoscope-IV Multimode atomic force microscope equipped with a c-AFM application module (TUNA). The investigations were performed with commercially available N-doped diamond-coated Si tips (NT-MDT) and Ti/Pt-coated Si tips (MikroMasch). Current-amplification settings of the c-AFM equipment of 1 and 10 V pA<sup>-1</sup> at an applicable voltage range of  $\pm 12$  V were used. For a typical scan rate of 0.5–1.0 µm s<sup>-1</sup>, the noise level was of the order of 50 fA at a bandwidth of 250 Hz. All data were acquired under ambient conditions and at room temperature and all such c-AFM measurements were made within a few minutes after the domain wall was created by electrical switching.

Structural investigations have been carried out using a Philips CM300 field emission gun microscope with a point-to-point resolution of 1.7 Å and information resolution of 0.8 Å. The domain structure was investigated in cross-section [010] orientation. The representative phase of the complex electron exit wave, as shown in Fig. 3a, was reconstructed from a series of 20 high-resolution images of the BFO 109° domain wall using the TrueImage software package<sup>33,34</sup>. According to the weak phase-object approximation for a thin object, the heavy Bi columns projected along the [010] direction correspond to the brightest dots and the less bright ones represent Fe in the phase.

DFT calculations were performed using the projector augmented wave implementation of the VASP code<sup>35</sup> with standard projector augmented wave potentials (Bi,  $6s^25d^{10}6p^3$ ; Fe,  $3p^64s^23d^6$ ; O,  $2s^22p^4$ ) and the local spin density approximation (LSDA) +U method to describe the exchange-correlation potential<sup>36</sup>. We used a cut-off energy of 550 eV and U/J values of 3/1 eV; these values were shown in previous work<sup>29</sup> to give a bandgap of 1.3 eV for bulk rhombohedral BFO and to yield a small bandgap even in the high-symmetry cubic perovskite phase. We constructed supercells containing two oppositely polarized domains and two domain walls; each domain contained six Bi2Fe2O6 units initially set to the bulk BiFeO3 structure. Note that the systems in our calculations are in all cases electrically neutral with uncharged domain walls. We used the experimental bulk lattice parameters, with the rhombohedral angle set to 60° so that the supercell could accommodate the broken symmetry of the domain wall. The Brillouin zone integration was performed on a 5  $\times$  5  $\times$  1  $\Gamma$ -centred Monkhorst–Pack grid incorporating a Gaussian averaging, with an energy width of 0.05 eV. Ionic positions were relaxed within the local minimum of maintaining two domains and domain walls per supercell until the total energy of the supercell converged to within 0.01 eV and the forces acting on individual ions were below 0.03 eV  ${\rm \AA^{-1}}$ The magnetism was set to the G-type antiferromagnetic ordering observed in bulk BFO and remained unchanged throughout the relaxation of the domain boundary. All parameters chosen for the supercell calculations were tested in calculations for single unit cells and shown to yield well-converged total energies, with atom positions and band structures in good agreement with previous work.

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#### Additional information

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#### Superconducting State in a Gallium-Doped Germanium Layer at Low Temperatures

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We demonstrate that the third elemental group-IV semiconductor, germanium, exhibits superconductivity at ambient pressure. Using advanced doping and annealing techniques of state-of-the-art semiconductor processing, we have fabricated a highly Ga-doped Ge (Ge:Ga) layer in near-intrinsic Ge. Depending on the detailed annealing conditions, we demonstrate that superconductivity can be generated and tailored in the doped semiconducting Ge host at temperatures as high as 0.5 K. Critical-field measurements reveal the quasi-two-dimensional character of superconductivity in the  $\sim$ 60 nm thick Ge:Ga layer. The Cooper-pair density in Ge:Ga appears to be exceptionally low.

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Since the first observation of superconductivity in 1911, the search for new superconducting materials has offered quite a few surprises, such as the discovery of several classes of high- $T_c$  compounds [1], but recently also the observation of superconductivity in the doped elemental semiconductors diamond [2] and silicon [3]. Although superconductivity has been observed in several doped binary semiconductors already starting in the 1960s, e.g., in doped tellurides such as GeTe [4], in SrTiO<sub>3</sub> [5], flanked and motivated by a considerable amount of theoretical work [6], as well as in SiC [7] recently, it has not been seen in the simple elemental representatives at ambientpressure conditions before. Superconductivity of Si and Ge in their metallic high-pressure phases has been reported by Wittig already in 1966 [8]. However, it needed more than four decades to drive Si and diamond superconducting at ambient-pressure conditions, owed to the sophisticated preparation techniques required and maybe due to the prejudice that superconductivity might not be possible in the archetypical semiconductors. Compared to Si and diamond, Ge seems to be even less promising for the search of superconductivity as theoretical studies predict only a weak tendency towards superconductivity in heavily *n*-type [9] and *p*-type doped Ge [10].

In order to obtain superconductivity in group-IV semiconductors, heavy *p*-type doping well above the metalinsulator transition is required. Otherwise the chargecarrier density of these materials is too low to create a superconducting state at low temperatures. Ekimov *et al.* have observed superconductivity in diamonds containing high boron (B) concentrations prepared by a high-pressure and high-temperature technique at temperatures up to  $T_c =$ 2.3 K [2]. Bustarret *et al.* [3] have investigated boronsupersaturated Si layers processed by ultra-short-time laser melting and found superconductivity at  $T_c = 0.34$  K. For this work, Ga has been chosen as acceptor atom due to its high solid solubility in Ge (up to ~1 at. % at 700 °C). Ion implantation as an effective method for introducing high concentrations of dopants has been used. The ion-beam technique is highly selective since it allows for a precise control of the amount of the implanted species and its depth distribution. Here, we have succeeded in producing samples with a peak concentration of 8 at. % Ga in a thin layer of Ge with a full width at half maximum of approximately 60 nm (see Fig. 1). However, high-dose ion implantation causes severe lattice damage which may be difficult to anneal as observed for B-doped diamond where the formation of graphite may further complicate a reconstruction of the lattice [11,12] and where the superconducting phase might be located in amorphous boron-rich intergranular layers and pockets [13]. In Ge (and Si), annealing of implantation damage is easier to achieve than in diamond. Here, the challenge is to reconstruct the crystalline structure without causing long-range diffusion, precipitation, and subsequent clustering of acceptor atoms in the supersaturated semiconductor. Flashlamp annealing in the millisecond range is known to be a versatile tool to remove the implantation damage and to achieve dopant activation in thin semiconductor layers [14-16]. Because of the short thermal-processing time, this method is much less affected by diffusion and precipitation problems. Depending on light-pulse energy and duration, flashlamp annealing leads to solid-phase epitaxial or liquid-phase epitaxial regrowth of the amorphous layers formed during the high-dose implantation. Here, we have treated samples using various flashlamp fluences  $\Phi$  after implanting. This resulted in samples with a slightly decreased peak concentration of 6 at. % Ga after annealing (see Fig. 1), corresponding to  $2.6 \times 10^{21}$  Ga atoms/cm<sup>3</sup>. No evidence for the formation of Ga clusters or filaments has been found during careful sample analysis. The charge-carrier concentration of the Ge:Ga samples has been determined at low temperatures,  $T \le 2.5$  K, by means of Hall-effect measurements (see inset of Fig. 3). A summary of the most important parameters of five samples is given in Table I. The hole concentration extracted from Hall measurements

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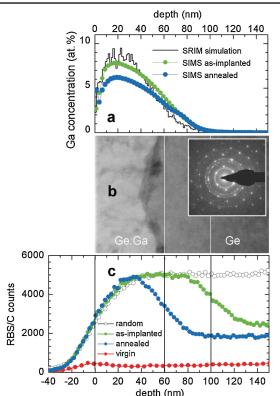


FIG. 1 (color). (a) Ga depth distribution obtained by secondary ion mass spectrometry (SIMS) of a Ge:Ga sample before and after annealing ( $\Phi = 50.8 \text{ J/cm}^2$ ) compared to a Ga profile simulated for an as-implanted Ge:Ga sample using the SRIM code [22]. (b) The cross-sectional transmission electron micrograph and the diffraction pattern (inset) from the same region demonstrate that the implanted layer is completely (poly)recrystallized after flashlamp annealing down to about 50-60 nm, followed by  $\approx 10$  nm thin wavy interface (indicated by the left white line) between the polycrystalline layer and the single-crystalline region. The initial amorphous-crystalline interface is indicated by the right white line. (c) Results of a channeled-ion Rutherford backscattering (RBS/C) analysis confirm the random orientation of the Ge grains. The backscattering signal for the annealed sample reaches the same value as in random orientation. For comparison the RBS/C results of unimplanted (virgin) and as-implanted Ge are shown.

varies considerably with the flashlamp fluence. At a small fluence,  $\Phi = 45.5 \text{ J/cm}^2$ , the hole concentration is up to 1 order of magnitude smaller than the Ga concentration determined from secondary ion mass spectrometry (SIMS), indicating the only partial electrical activation of the dopants. With increasing fluence, the hole concentration also increases to  $1.4 \times 10^{21} \text{ cm}^{-3}$  reflecting that half of the implanted Ga atoms have been activated.

Measurements of the electrical-transport properties were carried out from 300 K to 20 mK and are presented in Fig. 2. Data were taken using low ac-excitation currents (down to 10 nA) in order to keep the electrical current density in the thin Ge:Ga layer moderate. The unimplanted sample exhibits the typical behavior of semiconducting

TABLE I. Fluence  $\Phi$  used during 3 ms of flashlamp annealing, critical superconducting transition temperature  $T_c$  (taking the 50% drop of *R*), critical magnetic field aligned in plane,  $B_{c\parallel}$ , and perpendicular to the Ge:Ga layer,  $B_{c\perp}$ , as well as charge-carrier concentration obtained from Hall-effect measurement,  $n_{\text{Hall}}$ , of five Ge:Ga samples.

Ge:Ga sample	а	b	с	d	e
$\Phi$ (J/cm <sup>2</sup> )	45.5	48.1	50.8	53.6	62.4
$T_c$ (K)	• • •	0.14 <sup>a</sup>	0.45	0.19	• • •
$B_{c\parallel}$ (T)	• • •	•••	1.1	0.18	•••
$B_{c\perp}$ (T)	• • •	•••	0.30	0.025	•••
$n_{\rm Hall} \ (10^{21} \ {\rm cm}^{-3})$	0.28	0.32	0.43	1.4	1.0

<sup>a</sup>For sample b, a nonzero residual resistance was measured at  $T < T_c^*$ .

bulk *n*-type Ge with a resistance drop due to the increasing mobility upon lowering the temperature, and a subsequent resistance increase due to carrier freeze-out. The asimplanted Ge:Ga sample also shows an increasing resistance below about 40 K despite the high doping level. This is due to the large damage induced by implantation and implies that the dopants are not electrically activated. In contrast, the sample which has been flashlamp annealed (fluence  $\Phi = 53.6 \text{ J/cm}^2$ ) for t = 3 ms exhibits metallic behavior, indicating that a significant part of the dopants has been electrically activated. Below T = 0.5 K, the resistivity drops to zero (Fig. 2) indicating the onset of superconductivity. In more detail, in the inset of Fig. 2 it is shown how the annealing conditions influence the normal and superconducting properties of the Ge:Ga samples. The residual normal-state resistivity is clearly affected by the annealing conditions. Upon increasing flashlamp fluence, the residual resistivity drops by a factor of 3. Compared to that, we observed a more elaborate dependence of the superconducting properties on the annealing conditions (see Table I). By keeping the flashlamp expo-

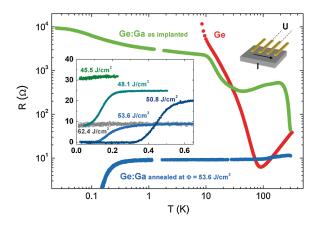


FIG. 2 (color). Temperature dependence of the electrical resistance of unimplanted Ge and as-implanted as well as annealed Ge:Ga ( $\Phi = 53.6 \text{ J/cm}^2$ , t = 3 ms) taken in B = 0. Only annealed Ge:Ga exhibits superconductivity below 0.5 K, depending on the flashlamp fluence  $\Phi$  (see inset).

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sure time constant (3 ms) but varying the fluence stepwise in the range  $45 \le \Phi \le 62.4 \text{ J/cm}^2$ , and consequently varying the maximum annealing temperature, the occurrence of superconductivity can be triggered and, when further increasing  $\Phi$ , even suppressed. The fluence of 45.5 J/cm<sup>2</sup> is not yet sufficient to induce a sample modification which allows for the creation of a superconducting state. At  $\Phi = 48.1 \text{ J/cm}^2$ , the onset of superconductivity is visible (inset of Fig. 2) and the highest transition temperature,  $T_c = 0.45$  K, is achieved at  $\Phi = 50.8$  J/cm<sup>2</sup>. Tuning the annealing conditions to higher fluences, superconductivity starts to disappear. At  $\Phi = 53.6 \text{ J/cm}^2$ ,  $T_c$  is reduced to about 0.2 K and at  $\Phi = 62.4 \text{ J/cm}^2$  a superconducting transition has not been observed in the temperature range accessible to us, i.e., at  $T \ge 20$  mK. The reproducibility of the annealing process with respect to the creation of superconductivity has been verified for several samples prepared separately under identical conditions. Thus superconductivity only occurs in a narrow region of optimized parameters; in particular, the activation of charge carriers appears to be rather delicate. It should be mentioned that the maximum  $T_c$  is not observed in the sample with the largest charge-carrier concentration 1.4  $\times$  $10^{21}$  cm<sup>-3</sup>, but in the sample that has only one third,  $0.43 \times 10^{21}$  cm<sup>-3</sup>, of the highest level of activated Ga atoms instead. The width of the superconducting transition is rather broad, reflecting the inhomogeneous chargecarrier profile of the Ge:Ga layers.

The superconducting transition temperature of Ge:Ga,  $T_c \leq 0.45$  K, compares well to the one of B-doped diamond [2], C:B, taking into account the qualitative expectation of phonon-mediated superconductors,  $T_c \sim \Theta_D$ . Whereas the ratios of the superconducting transition temperatures,  $T_c(C:B)/T_c(Ge:Ga) = 5.1$ , and of the Debye temperatures,  $\theta_D(C:B)/\theta_D(Ge:Ga) = 1860 \text{ K}/374 \text{ K} =$ 5.0, match well, the comparison of these properties with the data of B-doped Si [3] ( $T_c = 0.34$ ,  $\Theta_D = 645$  K) is less striking and might emphasize the influence of doping levels and individual electron-phonon coupling on the superconducting state of group-IV semiconductors. However, a reasonable scaling of  $T_c$  and  $\Theta_D$  might support the notion of phonon-mediated superconductivity in this material class. Although the critical temperature of Ge:Ga is about 0.5 K, the critical magnetic field,  $B_c$ , reaches a value slightly above 1 T (see Table I). Both samples that undergo a complete superconducting transition exhibit a distinct dependence of the superconducting critical field on its orientation relative to the Ge:Ga layer. Superconductivity remains stable to clearly higher critical magnetic fields,  $B_{c\parallel}$ , when aligned in plane compared to when aligned perpendicular to the Ge:Ga plane,  $B_{c\perp}$ . The anisotropy reaches a maximum in the sample which has been annealed at  $\Phi = 53.6 \text{ J/cm}^2$  where  $B_{c\parallel}/B_{c\perp} = 7$  (see Fig. 3). From a qualitative point of view, this strong anisotropy of the critical magnetic field is typical for a thinlayered superconductor and may be considered as proof

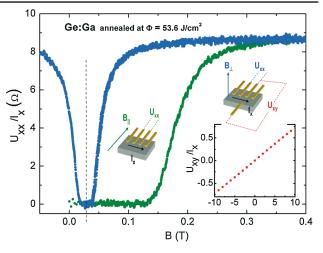


FIG. 3 (color). Electrical-transport data of a Ge:Ga sample (flashlamp annealed at  $\Phi = 53.6 \text{ J/cm}^2$ ) as function of the magnetic field aligned in plane  $B_{\parallel}$  (green), and perpendicular  $B_{\perp}$  (blue). The offset of *B* (dashed line at ~27 mT) results from frozen flux in the superconducting 20 T magnet after Hall-effect measurements (red data in inset).

that Ga is incorporated in the Ge matrix homogeneously in a thin layer in agreement with our structural analysis. The anisotropy of the superconducting critical field also allows for excluding superconductivity mediated by Ga clusters in the samples. The maximum possible in-plane fields,  $B_{c\parallel}$ , are attributed to the so-called Pauli limit where the high polarization of the electron spins is responsible for Cooperpair breaking, whereas  $B_{c\perp}$  is considered to be related to the orbital-limiting fields. It is remarkable that the theoretical Pauli limit in simple approximation [17],  $B_P/T_c =$ 1.84 (T/K), is exceeded in the sample with the highest  $T_c$ (0.45 K) and  $B_{c\parallel}$  (~1.1 T) annealed at 50.8 J/cm<sup>2</sup>, where  $B_{\parallel}/T_c = 2.4$  (T/K) (see Fig. 4). However, the large relative width of the superconducting transition hinders further conclusions. The temperature dependences of  $B_{c\parallel}(T)$  and  $B_{c\perp}(T)$  clearly deviate from the one of a simple bulk BCS superconductor,  $B_c(T) = B_{c0}[1 - (T/T_{c0})^2]$ . Even the Werthamer-Helfand-Hohenberg (WHH) theory [18] as a quantitative approach for type-II superconductors with a linear upper critical field,  $B_{c2}(T) \sim T$  at  $T > 0.5T_{c0}$ , does not reasonably describe the critical field of Ge:Ga as  $B_{c\parallel}(T)$  is linear down to lower temperatures,  $0.1T_{c0}$ , and  $B_{c\perp}(T)$  is even superlinear at  $T < T_{c0}$ . A more detailed study of the curvature of  $B_{c\parallel,\perp}(T)$  will be the subject of a more extended work on Ge:Ga where various scenarios for deviations from WHH theory (see, e.g., Boebinger et al. [19]) will be taken into account. From the orbital-limiting critical field, we deduce the coherence length as in the case of bulk superconductors according to  $\xi = (\phi_0/2\pi B_{c\perp})^{1/2}$ , where  $\phi_0 = h/2e = 2.068 \times 10^{-15}$  Wb is the flux quantum. From  $B_{c\perp}$ , one obtains  $\xi = 33$  (115) nm for  $\Phi =$ 50.8 (53.6)  $J/cm^2$ . These values may be taken to estimate the in-plane critical field,  $B_{c\parallel}(\xi) = \sqrt{6}\phi_0/(\pi\xi d)$ , where d



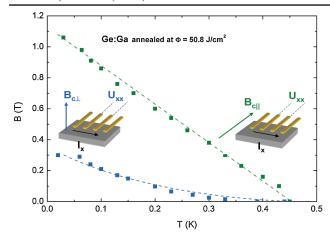


FIG. 4 (color). Field-temperature phase diagram of the Ge:Ga sample (flash lamp annealed at  $\Phi = 50.8 \text{ J/cm}^2$ ) with the highest  $T_c$  at 0.45 K for two magnetic-field orientations relative to the Ge:Ga plane (see illustrations).

is the thickness of the Ga-doped layer and  $\sqrt{6}$  is a correction factor for a thin-plate geometry [20]. Using d =60 nm leads to  $B_{c\parallel}(\xi) = 0.81$  (0.23) T for  $\Phi = 50.8$ (53.6) J/cm<sup>2</sup>, in reasonable agreement with the experimental values (Table I). By use of the nonlinear Ginzburg-Landau theory, the critical-current density  $J_c$  of a thin-film superconductor may be estimated [21] using  $J_c = ehn_s/(3\pi\sqrt{3m\xi})$ . We have measured a critical superconducting current of about 10  $\mu$ A. Using the superconducting critical-current density,  $J_c = 10 \ \mu \text{A}/(1 \text{ cm} \times$ 60 nm) =  $1.7 \times 10^4 \text{ A/m}^2$  (spanned by the 60 nm thick Ga-doped Ge layer and the sample width of 1 cm) and the above calculated coherence length  $\xi$ , allows for an estimate of  $n_s$ . We obtain a superconducting charge-carrier density,  $n_s = 2.7 \times 10^{14} \text{ cm}^{-3}$  ( $\Phi = 53.6 \text{ J/cm}^2$ ), which is by orders of magnitude smaller than the normalconducting charge-carrier density, deduced from Hall measurements,  $n \approx 1 \times 10^{21}$  cm<sup>-3</sup>. In this context, an effective mass of the charge carriers equal to the bare mass of free electrons,  $m = m_e$ , has been used for simplicity. In the case of light holes,  $m \ll m_e$ ,  $n_s$  would be even smaller. Using  $\lambda_L = \sqrt{m/(\mu_0 n_s e^2)}$  for the calculation of the London penetration depth leads to  $\lambda_L$  of the order of  $10^2 \mu m$ . Such a large London penetration depth makes the detection of superconductivity by inductive measurements virtually impossible in our thin Ge:Ga layers. Also the observation of a superconducting transition of Ge:Ga by means of heat capacity and NMR will be challenging as only a tiny jump of specific heat and an exiguous decrease of Pauli susceptibility and NMR Knight shift can be expected due to the very low Cooper-pair density. Therefore, the transport measurements reported here appear to be the only reliable means for the proof of superconductivity. According to  $\lambda_L/\xi$  of about 10<sup>3</sup>-10<sup>4</sup>, Ge:Ga may be considered as a superconductor in the extreme type-II limit.

In summary, the finding of superconductivity in Ga-doped Ge layers sheds new light on doped elemental semiconductors which might even serve as superconducting model systems as they allow for a tuning of their superconducting parameters via a modification of their hole concentration. The combination of ion implantation and subsequent flashlamp annealing can be employed to fabricate tailored thin-layer superconductors. Although the critical superconducting temperatures of Ge:Ga are far below those of high-temperature superconductors, there is a qualitative relationship between these systems concerning their  $T_c(x)$ -phase diagrams. Both in the hightemperature superconductors and in Ge:Ga, there is a limited range of the charge-carrier (hole) concentration x which allows for the occurrence of superconductivity, and there is an optimum concentration where  $T_c(x)$  is maximum. For further conclusions, of course, it would be of interest to explore the detailed relation between annealing parameters, carrier density, as well as critical temperatures and to also try other dopants in order to generate superconductivity in Ge.

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#### Interstitial-Mediated Diffusion in Germanium under Proton Irradiation

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We report experiments on the impact of 2.5 MeV proton irradiation on self-diffusion and dopant diffusion in germanium (Ge). Self-diffusion under irradiation reveals an unusual depth independent broadening of the Ge isotope multilayer structure. This behavior and the observed enhanced diffusion of B and retarded diffusion of P demonstrates that an interstitial-mediated diffusion process dominates in Ge under irradiation. This fundamental finding opens up unique ways to suppress vacancy-mediated diffusion in Ge and to solve the donor deactivation problem that hinders the fabrication of Ge-based nanoelectronic devices.

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Over the past few years the elemental semiconductor Ge has been the subject of many experimental [1-13] and theoretical investigations [14-25] to elucidate the electronic and diffusion properties of point defects as well as their interaction. Understanding these properties helps to develop strategies for efficient defect engineering that are crucial for the fabrication of the next generation of nanoelectronic devices. Utilizing Ge instead of silicon (Si) for complementary metal oxide semiconductors (CMOS) technology one can take advantage of the higher electron and hole mobilities in Ge compared to Si [26]. Whereas the p-channel Ge-MOSFET (metal oxide semiconductor fieldeffect transistor) made of heavily B doped source and drain regions was already demonstrated [27], the n-channel MOSFET remains a challenge due to the enhanced diffusion of *n*-type dopants such as *P*, As, and Sb under extrinsic doping conditions and the deactivation of the donors for concentrations exceeding  $10^{19}$  cm<sup>-3</sup> [7,9,27]. The enhanced diffusion is a consequence of the singly negatively charged donor-vacancy  $(AV)^{-}$  pair that mediates donor diffusion in Ge according to the reaction [7,9]

$$(AV)^- \leftrightarrow A_s^+ + V^{2-} \tag{1}$$

where  $A_s^+$  and  $V^{2-}$  are the singly positively charged substitutional donor with  $A \in \{P, As, Sb\}$  and the doubly negacharged vacancy  $(V^{2-})$ , respectively. The tively deactivation is related to the formation of inactive donorvacancy clusters whose formation is favored due to Coulomb attraction between  $A_s^+$  and  $(AV)^-$  via the reaction [9]

$$(AV)^{-} + A_{s}^{+} \leftrightarrow (A_{2}V)^{0}.$$
<sup>(2)</sup>

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The formation of  $A_2V$  and even bigger clusters  $A_nV_m$  is consistent with the predictions of density functional theory calculations [23]. Reactions (1) and (2) indicate that the donor-vacancy pair mediates both the diffusion and deactivation of *n*-type dopants in Ge. Effective defect engineering that aims to suppress the enhanced diffusion and deactivation of donors in Ge should reduce the concentration of the AV pairs. In this letter we demonstrate that defect engineering with Ge interstitials makes it possible to effectively suppress the enhanced diffusion of donor atoms.

The formation enthalpy of interstitials is predicted to be much higher than that of vacancies in Ge [19,20]. Accordingly, atomic transport in Ge under thermal equilibrium is mainly mediated by vacancies [7]. However, interstitials in Ge can be formed under irradiation as demonstrated in previous studies [10,13]. Stimulated by these results and the understanding on the evolution of interstitial clusters in Si [28], the impact of implantation damage on the diffusion in Ge was investigated by several research groups [11,12,29]. However, post anneals of dopant implanted Ge did not reveal any significant TED or transient retarded diffusion (TRD) [29,30]. Instead, it is generally observed, that the implantation damage in Ge recovers fast even at low temperatures [29,30].

In order to investigate the impact of interstitials on diffusion in Ge we performed diffusion experiments under proton irradiation. For these experiments we utilized a single crystalline Ge isotope multilayer structure consisting of 20 alternating enriched <sup>70</sup>Ge (96% enrichment) and natural Ge layers. With a thickness of 15 nm for each layer a total thickness of 300 nm is obtained. The isotope struc-

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ture was grown by molecular beam epitaxy (MBE) on a (100)-oriented Ge wafer at 250 °C. The distribution of <sup>74</sup>Ge within the multilayer measured with secondary ion mass spectrometry (SIMS) is illustrated in Fig. 1. In addition, we used a MBE grown structure with six B-doped Ge layers. The B-doped layers are each about 25 nm thick and separated by 100 nm undoped Ge. With a 100 nm thick undoped Ge cap and a buffer layer of 100 nm the total thickness of the single crystalline B-doped Ge structure is about 800 nm. A SIMS analysis of the asgrown B-doped structure is illustrated in Fig. 2. Finally, a Ge sample implanted with P at 30 keV to a dose of  $3 \times 10^{15}$  cm<sup>-2</sup> was prepared. In order to prevent outdiffusion of P during annealing a 10 nm thick SiO<sub>2</sub> layer was sputter deposited on the Ge sample before implantation. A SIMS analysis of the as-implanted P profile is illustrated in Fig. 3.

Samples with lateral dimensions of  $4 \times 4 \text{ mm}^2$  were cut from the as-grown or as-implanted Ge wafers, thinned to a thickness of  $30(\pm 5) \mu \text{m}$ , and polished with Nalco 2360 (Bucher AG, Switzerland) to obtain a scratch free and specular surface on the back. The samples were mounted on a graphite holder and fixed via a graphite plate with a circular aperture of 3 mm in diameter. In this way the outer

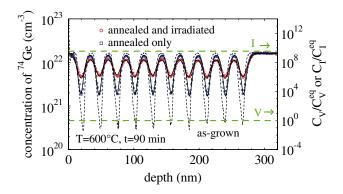


FIG. 1 (color online). Concentration profiles of <sup>74</sup>Ge measured with TOF-SIMS before (as-grown: thin dashed line) and after annealing (symbols) at the temperature and time indicated. Only every 6th data point is shown for clarity. The <sup>74</sup>Ge profile from the covered part of the sample (blue squares) represents Ge diffusion under equilibrium condition. The corresponding black solid line shows the expected Ge diffusion profile for selfdiffusion under equilibrium conditions at 600 °C taking into account published self-diffusion data [34]. The <sup>74</sup>Ge profile obtained from the proton irradiated area of the sample (red circles) indicates an enhanced Ge diffusion under irradiation with respect to equilibrium conditions. The corresponding black solid line shows the numerical simulation of self-diffusion under irradiation assuming different boundary conditions for vacancies and self-interstitials (see text for details). The corresponding concentration profiles of vacancies and self-interstitials normalized to the respective equilibrium concentration (see right y axis) are displayed by the lower and upper green dashed lines, respectively.

part of the Ge sample was covered with graphite and not exposed to the proton beam. The graphite holder was placed on a boron nitride heating plate that enables heating of the Ge sample during irradiation. The temperature was controlled with a thermocouple mounted 1 mm below the sample in the graphite holder. The whole sample holder was attached to a high vacuum chamber. Protons of 2.5 MeV were supplied via a beam line from a dynamitron accelerator. The beam was defocused and swept to achieve a homogeneously irradiated circular area with a diameter of 1 cm. An electron suppression, which consists of a negatively biased (600 V) screen, ensures that the measurement of the proton current is not hampered by secondary electrons. Proton irradiations were performed at 570 and 600 °C with a proton flux of 1.5  $\mu$ A. The high energy of the protons assures that the protons penetrate through the Ge sample as this is ascertained by simulations of the "stopping and range of ions in matter" (SRIM [31]). After annealing under proton irradiation the respective concentration profiles of <sup>74</sup>Ge, B, and P were measured with SIMS. The depth of the SIMS craters was determined using

Figure 1 shows the concentration profile of <sup>74</sup>Ge after proton irradiation at 600 °C for 90 min (see red circles). The broadening of the isotope structure under thermal equilibrium measured with SIMS at the outer part of the samples is illustrated by the blue squares. The Ge profile from the proton exposed inner part of Ge isotope sample

an optical profilometer.

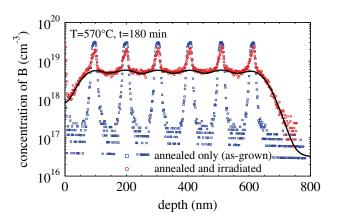


FIG. 2 (color online). Concentration profiles of *B* in Ge measured with TOF-SIMS after diffusion annealing at the temperature and time indicated. For clarity only every 6th data point is shown. The *B* profile from the covered part of the sample (blue squares) represents *B* diffusion under equilibrium conditions. This profile equals the as-grown profile because *B* diffusion in thermal equilibrium is very slow [4]. The *B* profile obtained from the proton irradiated area of the sample (red circles) indicates an enhanced *B* diffusion under irradiation with respect to equilibrium conditions. The black solid line shows a simulation of *B* diffusion based on Fick's second law with a concentration independent effective diffusion coefficient of  $7.5 \times 10^{-16}$  cm<sup>2</sup> s<sup>-1</sup> which exceeds the equilibrium diffusion of *B* by several orders of magnitude.

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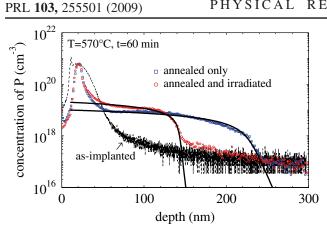


FIG. 3 (color online). Concentration profiles of P-implanted Ge measured with TOF-SIMS before (thin black dashed line) and after diffusion annealing (symbols) at the temperature and time indicated. Only every 6th data point is shown for clarity. The *P* profile from the covered part of the sample (blue squares) represents P diffusion under equilibrium conditions. The corresponding black solid line shows the expected P profile for diffusion under equilibrium conditions at 600 °C taking into account published data [7]. The P profile obtained from the proton irradiated area of the sample (red circles) indicates a retarded P diffusion under irradiation with respect to equilibrium conditions. The corresponding black solid line is a numerical simulation of the concentration dependent P diffusion that reveals a factor of 20 lower effective diffusion coefficient of P under irradiation compared to equilibrium conditions. Note, the 10 nm thick SiO<sub>2</sub> cap layer was not removed for the SIMS analysis and considered in the simulations of P diffusion by an offset in the profile. The high *P* concentrations in the peak region of the implanted P profiles likely reflect the formation of Pclusters.

shows a more pronounced intermixing. This unambiguously demonstrates an enhanced self-diffusion under irradiation. The homogenous broadening of the isotope structure under irradiation is, however, very unusual. This reveals that no gradient in the concentration of the native point defects versus depth exists. Usually the free surface acts as sink for vacancies and self-interstitials that are formed by proton irradiation in equal numbers and concentrations (see, e.g., [32]). The homogenous broadening of the Ge isotope structures reflects that thermal equilibrium is disturbed at the surface. The Ge profile alone can not tell us whether this holds for both vacancies and self-interstitials or just for one of these defects. In order to identify the surface condition, we performed additional experiments with B-doped and P-implanted Ge samples. Under thermal equilibrium the diffusion of B in Ge is very low; its diffusion coefficient is several orders of magnitude smaller than the self-diffusion coefficient [4]. This behavior is in accord with theoretical calculations [18] that predict a repulsive interaction between substitutional B and the vacancy. Annealing of the B-doped multilayer structure at 570 °C under proton irradiation leads to a very strong intermixing as illustrated by Fig. 2. For comparison, the B profile beneath the outer part of the Ge sample, covered by a graphite plate, did not show any broadening at all, i.e., the as-grown B profile is reproduced. The strong enhancement of B diffusion under irradiation is likely caused by interstitials whose concentration under irradiation exceeds their thermal equilibrium concentration by several orders of magnitude. The B spikes with a concentration of  $3 \times 10^{19}$  cm<sup>-3</sup> that interfere with the diffusion profile reveal an immobile fraction of B in Ge. This is similar to the behavior of B in Si where B-interstitial clusters have been identified as the origin of the immobile B fraction [28]. The concept of an interstitialmediated diffusion in Ge under irradiation is confirmed by the diffusion behavior of P. P diffusion is mediated by donor-vacancy pairs via the vacancy mechanism [see Eq. (1)] [7,9]. In the case interstitials dominate under proton irradiation the diffusion of P should be retarded. Indeed, Fig. 3 demonstrates a reduced penetration depth of P compared to the profile measured beneath the covered part of the same Ge sample.

The results of the impact of proton irradiation on selfand dopant diffusion in Ge show that the Ge surface is not an efficient sink for interstitials. The interstitials created during irradiation are assumed to be reflected at the Ge surface. The vacancies that are produced in equal number are annihilated at the surface and in the bulk via recombination with interstitials. During irradiation the concentration of interstitials increases and, finally, dominates over the vacancy concentration, and promotes an interstitialmediated diffusion. This diffusion behavior can be modeled on the basis of a continuum theoretical approach that considers the formation of point defects by irradiation and their annihilation via the Frenkel pair reaction [10]. Numerical simulations provide an accurate description of the experimental Ge profile when reflecting boundary conditions for the Ge interstitials are assumed. The calculated profile is given by the black solid line in Fig. 1. Analysis of the *B* profile reveals an enhancement factor of  $2.8 \times 10^7$ for B diffusion under irradiation compared to equilibrium diffusion [4] (see Fig. 2). The analysis of the P profiles shown in Fig. 3 yields a retardation factor of about 20 for P diffusion under irradiation.

The inability of the Ge surface to annihilate interstitials leads to the interstitial-mediated diffusion under irradiation. Recently, interstitials in Ge were formed via electron irradiation and directly observed by means of HRTEM [33]. Their direct observation would be hardly possible in the case the Ge surface is a perfect sink for interstitials. The property of the Ge surface seems to hold for bare surfaces like those prepared in Ref. [33] and surfaces covered with impurities. Our samples were annealed and proton irradiated in a vacuum of about  $10^{-6}$  mbar. Under these conditions they certainly do not exhibit bare surfaces. This also applies to the *P*-implanted sample with a thin SiO<sub>2</sub> layer on top.

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In conclusion, our experiments on self- and dopant diffusion in Ge under proton irradiation clearly demonstrate that interstitial-mediated diffusion is favored under irradiation that promotes B diffusion and retards P diffusion. These findings open up new strategies to effectively suppress vacancy-mediated diffusion in Ge that prevails under thermal equilibrium conditions. Thermal treatments under irradiation are proposed to solve the diffusion and also the doping issues that limit today the fabrication of Ge n-channel MOSFET's.

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#### Fano Signatures in the Intersubband Terahertz Response of Optically Excited Semiconductor Quantum Wells

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Absorption and transmission spectra of broadband terahertz pulses are measured to probe the intersubband response of an optically excited quantum-well heterostructure. While the terahertz absorption shows the single peak of the resonant intersubband transition, the transmission spectra display strong Fano signatures due to the phase sensitive superposition of ponderomotive and terahertz currents as predicted by our microscopic theory.

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Terahertz (THz) experiments on optically excited or doped semiconductors provide unique opportunities to analyze and manipulate low-energy excitations or quasiparticle states, like intersubband transitions between quantum confined states [1-5], excitons, or plasmons, and monitor their dynamical evolution [6-10]. With sufficiently strong THz pulses, one can even reach the regime of "extreme nonlinear optics" leading to effects such as Rabi flopping [5,11,12], ac-Stark splitting [13], or the dynamical Franz-Keldysh effect [14,15]. The microscopic analysis of these experiments shows that the THz wave propagating through the semiconductor is determined by the combined response of the so-called ponderomotive current and the true THz transitions. Here, the ponderomotive contribution refers to the charge current generated by the classical field that causes a wiggling motion of the carriers according to the acceleration theorem  $\hbar \mathbf{k} =$  $-e\mathbf{E}_{\text{THz}}$  [16,17]. This part of the light-matter interaction, often thought to be relevant only at high intensities [15], produces the simple response  $\chi(\omega) = -\frac{\omega_{\rm PL}^2}{\omega^2}$  with the plasma frequency  $\omega_{\rm PL}$ . Because of the factor  $\omega^{-2}$ , the ponderomotive contribution can usually be neglected in the analysis of interband optical excitation; however, it is of relevance in the THz regime even at the lowest intensities. Thus, the THz response of an excited semiconductor is influenced by ponderomotive effects plus the true THz transitions. When both contributions are of equal strength, one expects a strong interplay. In the experiments reported in Refs. [12,18] the evidences for the role of the ponderomotive current are rather indirect. Their influence could only be seen by virtue of a theoretical switch-off analysis.

To clearly expose the interplay of ponderomotive and true THz response, one needs an experimental method that makes it possible to observe both effects directly. In this Letter, we study the intersubband transition of photoexcited semiconductor quantum wells using linear THz spectroscopy. Monitoring the transmission of a broadband THz pulse, we directly observe signatures of the ponderomotive motion of the excited carriers. We show that interference of ponderomotive and resonant contributions produces a characteristic Fano-like line shape in the differential transmission spectrum.

Asymmetric Fano line shapes are known to result from quantum interference of discrete energy levels coupled to a nearby continuum [19] and have been observed in semiconductor heterostructures under various conditions [20– 23]. Whereas the relevant continuum in typical Fano configurations is an integral part of the probed quantum object, in our system an effective continuum is provided by the light-matter interaction through the ponderomotive contribution.

Figure 1(a) shows the basic concept of our experiment where spectrally broadband THz pulses probe the sample's intersubband transition after resonant photoexcitation. The THz pulses are generated in a 55  $\mu$ m thin z-cut GaSe crystal by phase-matched difference frequency mixing [24] within the broad spectrum of 12 fs optical pulses, delivered by a 78 MHz Ti:sapphire oscillator (Femtolasers: Femtosource Scientific sPro). The THz beam is focused by off-axis parabolic mirrors on the multi-quantum-well (MQW) sample, with the THz field having a strong component perpendicular to the MQW plane to couple effectively to the intersubband transition [25]. For THz-fieldresolved detection, phase-matched electro-optic sampling [26,27] is applied. To this end, a weak component of the 12 fs laser beam goes through a delay stage and samples the THz-field induced polarization change in a second 30  $\mu$ m thin z-cut GaSe crystal as a function of the time delay between THz pulse and sampling pulse [Fig. 1(c)].

The sample studied consists of 60 periods of 8.2-nmthick undoped GaAs quantum wells, separated by 19.6-nmthick barriers of  $Al_{0.34}Ga_{0.66}As$ . The sample was prepared

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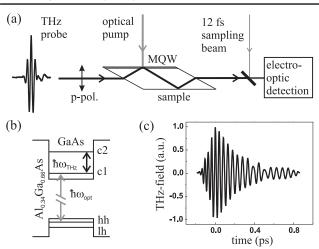


FIG. 1. (a) Optical interband pump intersubband THz probe experiment. (b) The transition from the heavy-hole (hh) state to the first conduction state (c1) in the  $Al_{0.34}Ga_{0.66}As/GaAs$  multiquantum-well (MQW) sample is resonantly excited, either by a ps or a fs laser. THz pulses probe the c1-to-c2 transition 25 ps after photoexcitation. (c) The field transients of the transmitted THz pulses are detected by phase-matched electro-optic sampling with a sampling beam. The reference without photoexcitation is shown.

in a 38-degree wedged-waveguide geometry, as shown in Fig. 1(a). To ensure optimized overlap between MQW region and THz standing wave [25], an additional  $Al_{0.34}Ga_{0.66}As$  spacer layer of 300 nm thickness was grown on top of the sample. The experiments are performed at a temperature of 6 K.

The photoexcitation scheme is illustrated in Fig. 1(b). An optical interband pump pulse excites the MQW resonantly at the 1s heavy-hole exciton and creates carriers in the first conduction band. We choose either spectrally narrow 2.5 ps or broadband 100 fs optical excitation centered at  $\hbar\omega_{opt} = 1.56$  eV. Both lasers are 78 MHz Ti: sapphire oscillators (Spectra Physics: Tsunami), locked to the repetition rate of the THz generating laser. In each case, only the first conduction subband of the MQW is populated. After a time delay of 25 ps during which the optically induced interband coherences disappear, the weak broadband THz-pulse probes the c1-to-c2 intersubband transition. In all the experiments, the photoexcited carrier density is  $2 \times 10^{10}$  cm<sup>-2</sup> per quantum well.

A two-lock-in technique is employed to measure the transmitted THz transients with and without excitation. The visible pump and THz probe beams are chopped simultaneously at different frequencies around 2 kHz. The first lock-in amplifier locks on the modulation of the optical pump beam. Since the THz beam is also chopped at the same time, twice the signal obtained by the first lock-in amplifier yields the differential transmission  $\Delta E(t)$ , i.e., the pump-induced change in the transmitted THz field. The second lock-in amplifier detects the mean value between transmission with and without photoexcitation, leading

to the reference  $E_{\rm ref}(t)$  without excitation by subtracting the signal of the first lock-in amplifier. This allows us to record  $\Delta E(t)$  and  $E_{\rm ref}(t)$  simultaneously under the same conditions, preventing a drift in the relative phase between them. Such a phase drift would strongly affect the computed total THz absorption of the quantum wells  $\alpha(\omega)$ , where the complex valued Fourier transforms of  $\Delta E(t)$ and  $E_{\rm ref}(t)$  enter in the following way:  $\alpha(\omega) =$  $2 {\rm Im}[-i\Delta E(\omega)/E_{\rm ref}(\omega)]$ . This relation follows from Maxwell's equations and the assumptions that (i)  $|\Delta E| \ll |E_{\rm ref}|$ , (ii) the unexcited sample is nonabsorptive in the investigated frequency range, and (iii) only the single-pass signal is measured.

Figure 1(c) shows the reference transient  $E_{ref}(t)$ . Compared with the incident THz transient [Fig. 1(a)], the field oscillations last longer in time due to dispersion in the sample. The differential THz transmission transients  $\Delta E(t)$  are presented in Figs. 2(a) and 2(b) for ps and fs excitation, respectively. First, one observes that the signal has 440 fs

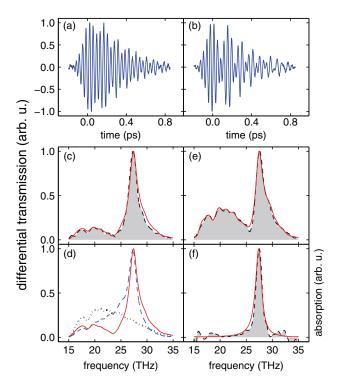


FIG. 2 (color online). Experiment-theory comparison of the THz intersubband response. (a),(b) Measured time-resolved differential transmission signals  $\Delta E(t)$  for ps and fs excitation, respectively. (c) Measured spectral amplitude of differential transmission  $|\Delta E(\omega)|$  (shaded area) and calculated current density  $|J_{tot}(\omega)|$  (red solid line) after optical ps excitation. (d) shows the decomposition of the computed total current density (red solid line) into the ponderomotive contribution  $|J_A|$  (black dotted line) and the intersubband current  $|J_{THz}|$  (blue dashed line). (e), (f) Measured (shaded area) and computed (red solid line) differential transmission and THz absorption, respectively, after fs excitation. Here,  $J_A$  has been enhanced by 1.85 in order to account for substrate excitations.

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decay constant for both ps and fs excitation, whereas a decay of 240 fs is observed for the reference  $E_{ref}(t)$  [see Fig. 1(c)]. This reveals the dephasing of the reradiated THz-induced intersubband polarization [2,3,28]. Second and more interesting, one observes a clear beating compared to the reference  $E_{ref}(t)$ , superimposed on the dephasing and being more pronounced for fs photoexcitation.

We first address the ps excitation to analyze the origin of the observed beating. Figure 2(c) shows the spectral amplitude of the measured differential THz transmission  $|\Delta E(\omega)|$  after optical excitation at the 1s position of the heavy-hole exciton resonance. We see that the spectrum consists of two major contributions: (i) a sharp resonance peak at the transition frequency of the conduction subbands  $\nu_{1,2} = 27.3$  THz and (ii) a broad contribution centered at roughly 20 THz, being responsible for the beating in the time domain. These features remind us of a typical Fano spectrum, i.e., an undershoot at the low-frequency side of the peak followed by an asymmetric line shape.

When the system is excited with a 100 fs pulse, the broad contribution and the Fano signatures are more pronounced than in the ps case [Fig. 2(e)]. To check whether the broad feature in the differential transmission is caused by an additional carrier transition besides the intersubband resonance, we measure the THz absorption. Figure 2(f) shows that the absorption is single peaked; i.e., only the intersubband resoband resonance appears and no Fano-like signature is observed.

In order to explain the experimental findings, we compute the THz transmission of a quantum well positioned at z = 0 (where z is the growth direction of the quantum well). The THz response follows from the wave equation

$$\left(\nabla^2 - \frac{n_b^2}{c^2} \frac{\partial^2}{\partial t^2}\right) E(\mathbf{r}, t) = \mu_0 \delta(z) \frac{\partial}{\partial t} (J_{\rm A}(t) + J_{\rm THz}(t)), \quad (1)$$

where  $n_b$  is the background refractive index, c is the speed of light, and  $\mu_0$  is the permeability of free space. The delta function arises due to the fact that the quantum-well width is much smaller than the wavelength of the THz field. The induced current density appearing as source term on the right-hand side of Eq. (1) consists of two terms:  $J_{\text{THz}}$  is the current due to the intersubband transitions and  $J_A$  describes the ponderomotive motion of the excited carriers due to the THz field. Solving Eq. (1), one finds that the differential transmission, i.e., the field that is reemitted by the current density, is directly proportional to the induced current [29]:  $\Delta E \propto J_A + J_{\text{THz}}$ .

The ponderomotive contribution to the current density is given by  $J_{\rm A} = -\sum_{\lambda} \frac{e^2 n_{\lambda}}{m_{\lambda}} A_{\rm THz}$  [30] where  $n_{\lambda}$  is the carrier density in band  $\lambda$  and  $m_{\lambda}$  is the effective mass. The vector potential  $A_{\rm THz}$  of the THz pulse is defined via  $E_{\rm THz} = -\frac{\partial}{\partial t} A_{\rm THz}$ . Here,  $\lambda$  includes both bulk-band index and subband index. Obviously, the ponderomotive current directly follows  $A_{\rm THz}$  with an opposite phase due to the minus sign in  $J_{\rm A}$ . The linear susceptibility  $\chi_{\rm A}(\omega) = -\frac{\omega_{\rm Pl}^2}{\omega^2}$  following from the ponderomotive current is a real-valued quantity. Consequently,  $J_A$  does not contribute to the absorption  $\alpha(\omega) \propto \omega \text{Im}[\chi(\omega)]$ , but simply introduces a refractive index change to the excited material.

The THz current is computed via  $J_{\text{THz}} = \frac{1}{S} \sum_{\lambda,l,l',\mathbf{k}} j_{l',l}^{\lambda} p_{l,l',\mathbf{k}}^{\lambda}$  with the quantization area *S* and the matrix elements  $j_{l,l'}^{\lambda}$ . The quantity  $p_{l,l',\mathbf{k}}^{\lambda}$  represents the microscopic intersubband polarization between subbands *l* and *l'* of bulk-band  $\lambda$ . The intersubband matrix element is given by  $j_{l,l'}^{\lambda} = -\frac{i\hbar e}{m_{\lambda}} \int dz \xi_{\lambda,l}^*(z) \frac{\partial}{\partial z} \xi_{\lambda,l'}(z)$  where  $\xi_{\lambda,l}(z)$  is the confinement wave function of the carriers.

The intersubband polarization  $p_{l,l',\mathbf{k}}^{\lambda}$  in  $J_{\text{THz}}$  is computed microscopically with an equation-of-motion approach. Since we are interested only in the c1-to-c2 transition, we can restrict the sum to  $\lambda = c$  and  $l, l' \in \{1, 2\}$ . In analogy to the semiconductor Bloch equations [31], one finds for the time evolution of the intersubband polarization

$$i\hbar \frac{\partial}{\partial t} p_{1,2,\mathbf{k}}^{c} = (\tilde{\varepsilon}_{2,\mathbf{k}}^{c} - \tilde{\varepsilon}_{1,\mathbf{k}}^{c}) p_{1,2,\mathbf{k}}^{c} + \frac{\partial}{\partial t} p_{1,2,\mathbf{k}}^{c} \bigg|_{\text{scatt}} + (f_{\mathbf{k}}^{c1} - f_{\mathbf{k}}^{c2}) \bigg[ j_{2,1}^{c} A_{\text{THz}} - \sum_{\mathbf{q} \neq \mathbf{k}} V_{\mathbf{k}-\mathbf{q}} p_{1,2,\mathbf{q}}^{c} \bigg] + S_{\mathbf{k}}^{\text{coh}}, \qquad (2)$$

where  $\tilde{\varepsilon}_{l,\mathbf{k}}^c$  are the renormalized single-particle energies,  $f_{\mathbf{k}}^{c,l}$  are the optically excited carrier distributions in the conduction bands, and  $V_{\mathbf{q}}$  is the Coulomb matrix element. The term  $\frac{\partial}{\partial t} p_{1,2,\mathbf{k}}^c |_{\text{scatt}}$  formally contains all scattering effects and  $S_{\mathbf{k}}^{\text{coh}}$  includes the coupling to optically excited interband coherences via the Coulomb interaction. The scattering terms are modeled by a phenomenological dephasing constant  $\gamma$  to match the measured 440 fs decay, i.e.,  $\frac{\partial}{\partial t} p_{1,2,\mathbf{k}}^c |_{\text{scatt}} \approx -i\gamma p_{1,2,\mathbf{k}}^c$ . This approximation is justified since in our case, the intersubband transitions are not affected by excitonic effects due to large experimental linewidths (roughly 3.3 meV). For large time delays of optical pump and THz probe pulses, the optically excited coherences have decayed such that  $S_{\mathbf{k}}^{\text{coh}}$  does not contribute.

In our numerical evaluations, we compute the singleparticle energies of the quantum-well system using standard  $\mathbf{k} \cdot \mathbf{p}$  perturbation theory. For the THz response, we include the first two conduction subbands and the first heavy-hole and light-hole subband [Fig. 1(b)]. As input for the time-dependent fields, we use the experimental THz-pulse shapes of the reference pulse.

The theoretical results are superimposed to the experimental results in Fig. 2. The (red) solid line in Figs. 2(c) and 2(e) represents the absolute value of the computed current density, i.e., the differential transmission for the ps (c) and fs excitation (e). Figure 2(d) separates the two contributions  $J_A$  (dotted line) and  $J_{THz}$  (blue dashed line) to the total current for the case of ps excitation. The solid line in Fig. 2(f) shows the computed absorption spectrum.

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In all cases, an excellent experiment-theory agreement is obtained.

As in the measurement, we clearly notice the double peaked Fano-like feature in the transmission spectra, whereas the absorption is only single peaked. In our theory, the origin of the Fano-like feature can be investigated looking at the individual contributions separately.  $J_{\Delta}$ directly reflects the vector potential of the THz probe pulse while  $J_{THz}$  consists of the intersubband resonance weighted with the pulse spectrum. The broad feature in the transmission spectrum is a direct consequence of the broad probe pulse. The Fano-like line shape is caused by the phase sensitive superposition of the broad ponderomotive and the sharp intersubband contribution,  $|J_{tot}| =$  $|J_{\rm A} + J_{\rm THz}|$ . A further analysis reveals that for frequencies smaller than  $\nu_{1,2}$ , both contributions partially compensate each other while they interfere constructively for larger frequencies. As for typical Fano situations, this kind of superposition leads to narrowing of the resonance at the low-frequency side and a broadening at the high-frequency side, resulting in the characteristic asymmetric Fano line shape. Since the ponderomotive current leads to a realvalued susceptibility, there is no Fano-like behavior in the THz absorption [Fig. 2(f)].

In order to explain the more pronounced ponderomotive feature in the fs experiment, we had to extend our model beyond the single-quantum-well response. Since the fs pump pulse is spectrally significantly broader than the line width of the 1s exciton resonance (18.2 meV vs 3.3 meV), some frequency components are transmitted through the quantum wells and excite carriers in the substrate. These carriers contribute to  $J_A$  but not to  $J_{THz}$ , since there are no subbands in bulk material. A quantitative investigation of the interband optical excitation shows that 46% of the pump pulse is transmitted through the quantum wells and absorbed by the substrate. Hence, the total carrier density (in the wells plus substrate) is 1.85 times larger than the density in the quantum wells alone. Since  $J_A$  is proportional to the carrier density, we can model the substrate contribution to the THz response simply by enhancing  $J_A$  relative to  $J_{THz}$  by 1.85. The resulting current density is shown as the red solid line in Fig. 2(e). In the ps experiment, substrate excitations can be neglected since only 1.4% of the pump pulse reaches the substrate.

In summary, we have shown how the emission by the ponderomotive motion of the excited carriers can directly be identified in the linear THz response. Using a microscopic theory for the THz response, the observed Fano-like features in the transmission spectrum can unambiguously be attributed to the phase sensitive superposition of the intersubband resonance and the ponderomotive carrier dynamics. Our results are connected to the typical Fano situation because the system has a sharp discrete intersubband resonance that interferes with a broad continuous contribution, i.e., the ponderomotive current density. However, one of the contributions in our case is nonabsorptive such that we do not observe the Fano resonance in the absorption spectrum but in the differential transmission spectrum only.

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#### Transition from smoothing to roughening of ion-eroded GaSb surfaces

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During ion sputtering of GaSb(100) surfaces a transient behavior from initial smoothing to roughening accompanied by self-organized pattern formation has been observed using *in situ* x-ray reflectivity and grazing incidence small angle scattering. The induced patterns show hexagonally ordered nanodot arrays with a spatial periodicity of 30 nm. The correlation length of the pattern increases with ion fluence. In the framework of the Bradley–Harper model [R. M. Bradley and J. M. E. Harper, J. Vac. Sci. Technol. A **6**, 2390 (1988)], where the dot pattern formation results from an interplay of surface roughening due to sputtering and surface smoothing due to diffusion, the initial smoothing behavior is explained by the same surface diffusion processes as the pattern formation. © 2009 American Institute of Physics. [DOI: 10.1063/1.3136765]

The roughness of surfaces and interfaces strongly influences many properties of thin films and surfaces, among them the electrical, optical and magnetic properties.<sup>1</sup> Therefore controlling the roughness is an important issue in a broad variety of technological applications. In addition, by controlling the roughness down to the nanoscale the structure and properties of films deposited on these surfaces can be tuned. For example, nanostructured surfaces can be used as templates for the growth of thin magnetic films with a predefined anisotropy<sup>2</sup> and metallic thin films exhibiting dichroitic plasmonic properties.<sup>3</sup> When materials are bombarded with energetic ions the surface is eroded by the sputtering process. Ion beam sputtering (IBS) is often used to modify the roughness of solid surfaces on lateral scales ranging from a few nanometers up to micrometers.<sup>4</sup> Under certain sputtering conditions (energy, ion mass, incidence angle, fluence, etc.), IBS leads to a smoothing of the surface, whereas at other parameters the surface roughness is increased, leading eventually to periodic patterns, i.e., ripple or dot patterns.<sup>5</sup> Recently, it has also been shown that the pattern formation might be accompanied by an actual decrease of the surface roughness.<sup>6</sup>

In this letter we report on *in situ* x-ray scattering measurements of the morphology of GaSb (100) surfaces during Ar<sup>+</sup> IBS at normal incidence. Especially, the early time regime is addressed, which gives insight into the detailed mechanisms governing the dynamics in the beginning of the pattern formation process. The roughness and morphology of the sample surface are measured by *in situ* x-ray reflectivity (XRR) and grazing incidence small angle x-ray scattering (GISAXS). This experimental approach, i.e., *in situ* x-ray scattering, has proven to be a valuable tool to investigate the surface evolution and transient behaviors in the early time Ion erosion was performed in a compact high vacuum chamber with a base pressure of  $10^{-7}$  mbar with a Kaufman type ion source. The chamber, equipped with a 360° beryllium window for the x-ray scattering measurements,<sup>10</sup> is mounted on a goniometer at the ID01 synchrotron beam line at the ESRF in Grenoble (France). Sputtering was done with Ar<sup>+</sup> ions at normal incidence with an energy of 450 eV and ion flux of  $1 \times 10^{15}$  cm<sup>-2</sup> s<sup>-1</sup>. XRR and GISAXS spectra were measured at an x-ray energy of 8 keV after consecutive steps of sputtering, stopping the erosion process during the measurements. The sample temperature was always below 60 °C during each sputtering step. At these conditions the GaSb surface should be amorphized and in sputter equilibrium after an ion fluence of approx.  $1 \times 10^{16}$  cm<sup>-2</sup>.<sup>11</sup>

Figure 1(a) shows XRR measurements of the initial, untreated surface and after sputtering with fluences between  $3.6 \times 10^{16}$  and  $6.8 \times 10^{17}$  cm<sup>-2</sup>. In general, the decay of specular reflected intensity with increasing incidence angle  $\alpha$  above the critical angle  $\alpha_c \approx 0.32^\circ$  reflects the penetration of the x-ray beam in the material and is, for a perfectly flat surface, given by the Fresnel equations. Surface roughness leads to offspecular diffuse scattered intensity and hence to a faster decay of the specular signal with incidence angle. Finally, the presence of a layer with different optical properties

regime of the ion erosion process, which would be difficult to access with other *ex situ* techniques.<sup>7,8</sup> In contrast to earlier investigations of the roughening behavior of ion eroded GaSb surfaces<sup>9</sup> where only an increase in the roughness with the formation of a dot pattern has been measured, we observe a transition behavior of the surface morphology: an initial smoothing regime is followed by a subsequent roughening of the GaSb surface and the formation of a dot pattern. Simulations of the dynamics of the surface morphology with a continuum approach show that the initial smoothing results from the same relaxation mechanism leading to the formation of the periodic dot structure.

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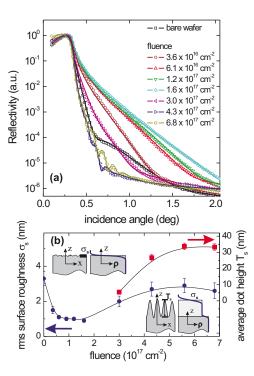


FIG. 1. (Color online) (a) XRR (symbols) of the GaSb surface sputtered with 450 eV Ar<sup>+</sup> ions at different ion fluences. The rough initial surface is smoothed in the beginning, followed by a strong roughening process. (b) Evolution of the roughness  $\sigma_s$  and the thickness  $T_s$  of the transition layer, respectively, as obtained from a simulation [solid lines in (a)] of the reflectivity curves. Insets illustrate the nature of the transition layer at low and high fluences, respectively.

on top of the surface leads to interference fringes whose angular distance is inversely proportional to the layer thickness.

Prior to sputtering, the XRR measurements reveal a rather rough surface with a thin surface layer which probably corresponds to the native oxide on top of the GaSb.<sup>19</sup> During ion erosion up to a fluence of  $1.6 \times 10^{17}$  cm<sup>-2</sup> the reflectivity at angles  $\alpha > \alpha_c$  increases significantly indicating a smoothing of the surface due to the ion irradiation. The roughening observed for higher fluence can be associated with the onset of a dot pattern formation as visible by GISAXS measurements (see text below). Moreover, the interference fringes observed in the XRR curves in this fluence region indicate the development of a layer with an electron density reduced with respect to the bulk value of GaSb. In order to quantify the surface evolution the reflectivity data are analyzed assuming a transition layer on top of a GaSb substrate that is characterized by a constant (lower) electron density, by its thickness  $T_s$ , and by introducing a surface roughness  $\sigma_s$  as a Gaussian-like density gradient into the Fresnel formula [insets in Fig. 1(b)]. Figure 1(b) shows the evolution of  $\sigma_s$  and of  $T_s$ , respectively.

At the beginning of sputtering the oxide layer is removed almost immediately and the GaSb surface can be modeled by the surface roughness only, i.e.,  $T_s=0$ . The initial decrease of  $\sigma_s$  reflects the reduction of the surface roughness. With the onset of pattern formation at a fluence of 3  $\times 10^{17}$  cm<sup>-2</sup>  $T_s$  starts to grow until it reaches a stable value of 30 nm, giving a measure for the averaged height of the dot pattern. The simultaneous increase of  $\sigma_s$  can be interpreted as height fluctuations of the dot pattern.

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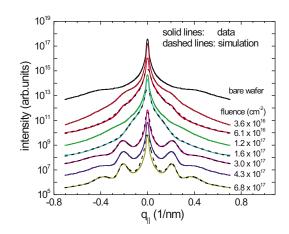


FIG. 2. (Color online) GISAXS spectra of the GaSb sample at different ion fluences.

In addition to XRR, GISAXS spectra were recorded between the sputtering cycles (Fig. 2). Complementary to XRR, these measurements contain information about the lateral height correlations along the surface. At the beginning of the erosion process, the strong diffuse scattering of the substrate is reduced, reaching a minimum at a fluence of 1.6  $\times 10^{17}$  cm<sup>-2</sup>. At a fluence of  $3 \times 10^{17}$  cm<sup>-2</sup>, corresponding to the increase of surface roughness as obtained by XRR, satellite peaks appear, indicating the onset of the formation of a periodic pattern. The position of these peaks is related to the mean interdot distance (wavelength) *l*, whereas number and width of the satellite peaks are related to the correlation length  $\xi$ .<sup>12</sup> The dashed lines in Fig. 2 show simulations of the scattered intensity. During the smoothing regime, the diffuse scattering can be modeled taking into account the nonspecular component of the structure factor of a rough surface.<sup>13</sup> For a quantitative analysis of the GISAXS spectra from the periodic dot pattern, the data has been fitted with the program ISGISAXS,<sup>14</sup> using a model of cone-shaped particles and the two-dimensional hexagonal paracrystal model to describe their correlation in a short-range order scenario.<sup>14</sup> The fitting results for the mean wavelength l and the correlation length  $\xi$ (not shown) revealed that the wavelength increases only slightly from 30 to 32 nm, whereas the correlation length increases with fluence by about a factor of 2 from roughly 10 to about 20 periods. This increase of  $\xi$  is reflected in the decrease of FWHM of the first-order satellite peak and the appearance of second-order peaks in Fig. 2.

In the Bradley–Harper (BH) model of IBS, pattern formation is explained by the interplay of two processes: first, roughening by the curvature dependent sputter yield and second, surface relaxation by surface diffusion.<sup>15</sup> In order to elucidate the smoothing and pattern formation mechanisms at the beginning of the ion sputtering, numerical integrations of a nonlinear extension of the BH model, the Kuramoto– Sivashinsky (KS) equation,<sup>16</sup>

$$\frac{\partial h}{\partial t} = -v_0 + \nu \nabla^2 h - D_{\text{eff}} \nabla^4 h + \frac{\lambda}{2} (\nabla h)^2, \qquad (1)$$

have been performed. Here,  $v_0$  is the constant erosion velocity of the planar surface. The second term represents the curvature dependence of the erosion velocity and the third term the effective surface relaxation. The nonlinear fourth term incorporates the dependence of the local erosion velocity on the surface slopes and is responsible for the formation



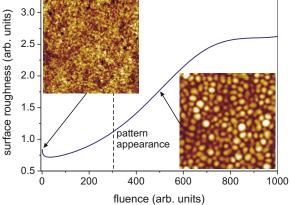


FIG. 3. (Color online) Root mean square roughness and surface morphology obtained by numerical integration of the KS Eq. (1). Note the good qualitative agreement with the measured results [Fig. 1(b)]. *X* and *y* scales depend only on simulation parameters and can be rescaled arbitrarily. The size of the AFM type images is  $500 \times 500$  nm<sup>2</sup>.

of ordered dot patterns at intermediate times.<sup>17,18</sup> However, for long sputtering times, in the nonlinear regime the pattern evolves to kinetic roughening which is not observed experimentally for GaSb. In our integration of the continuum equation a real atomic force microscopy (AFM) image of the virgin GaSb(100) wafer was used as the starting surface (left inset in Fig. 3). The coefficients  $\nu$ =0.15,  $D_{\rm eff}$ =1, and  $\lambda$ =0.1 were chosen such that the periodicity *l* is close to the experimental measured periodicity of 30 nm and that the initial dynamics of the pattern evolution can be followed easily. In this way the scales of the initial surface are mapped to the simulation.

Figure 3 shows the evolution of the surface roughness in the simulation. In the initial phase of the simulation the diffusion term leads to a reduction of the roughness at length scales smaller than the wavelength l of the system, whereas at larger length scales roughness is not affected. Therefore, the net effect of the diffusion term is to reduce the rms roughness, calculated as the integral of the power spectral density (PSD), in the beginning. At longer simulation times, the KS instability starts to grow and the pattern exhibits dot features with the wavelength l (right inset in Fig. 3). At fluences around 300 an ordered dot pattern appears which can be identified by a pronounced peak in the PSD. Now the surface is dominated by the pattern and the roughness increases again due to the formation of dots. The wavelength l is given by the ratio between the roughening coefficient  $\nu$  and the diffusion coefficient  $D_{\text{eff}}$ . Thus, the same diffusion mechanism that determines the formation of periodic patterns is also responsible for the initial surface smoothing.

In summary, we presented *in situ* measurements of the nanodot formation on an Ar-eroded GaSb(100) surface using XRR and GISAXS. We showed that the sputtering process leads to an initial smoothing of the surface, followed by a roughening process associated with the formation of nanodots. Both the smoothing and roughening behavior can be modeled using the KS equation and are the result of the same diffusion mechanisms.

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- <sup>19</sup>The initial root mean square (rms) roughness of commercially available GaSb(100) has a value of  $\sim$ 0.8 nm and is thus much higher than that of, e.g., commercial Si(100) with  $\sim$ 0.2 nm.

# Achieving high free electron mobility in ZnO:AI thin films grown by reactive pulsed magnetron sputtering

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The study is focused on the improvement of the free electron mobility in Al-doped ZnO films grown by reactive pulsed magnetron sputtering. At optimum growth conditions low-absorbing films are obtained with a Hall mobility of 46 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, a free electron density of  $6.0 \times 10^{20}$  cm<sup>-3</sup>, and an electrical resistivity of  $2.26 \times 10^{-4}$   $\Omega$  cm. The relation between the mobility and free electron density for different growth conditions is discussed in terms of ionized impurity scattering, impurity clustering, and grain boundary limited transport. © 2009 American Institute of Physics. [DOI: 10.1063/1.3074373]

The need to establish a cost-effective replacement of tindoped indium oxide as a transparent electrode material for thin-film solar cells has stimulated substantial research efforts to decrease both optical absorption in the near infrared (down to 1 eV) and electrical resistivity of Al-doped ZnO (AZO).<sup>1-6</sup> This can be accomplished by increasing the free electron mobility  $(\mu_e)$  while maintaining the maximum free electron density  $(N_e)$  below  $1 \times 10^{21}$  cm<sup>-3</sup>. The combination of lowest electrical resistivity ( $\rho \sim 0.85 - 4 \times 10^{-4} \ \Omega \text{ cm}$ ), highest mobility ( $\mu_e \sim 40-50 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ), and a low optical absorption coefficient  $\alpha \sim 500 \text{ cm}^{-1}$  in the visible spectral range is usually achieved by pulsed laser deposition (PLD)<sup>2,3</sup> or radio frequency magnetron sputtering (rf MS)<sup>4</sup>, using ceramic targets and deposition at substrate temperatures  $T_S > 200$  °C. Films grown by reactive pulsed MS (RP MS), which would override both PLD and rf MS in terms of scalability and cost efficiency, respectively, by now showed mobilities limited to  $\mu_e \sim 28 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1} \cdot \overset{6,7}{.}$  In contrast to PLD and rf MS, the RP MS uses metallic targets being sputtered in a mixture of Ar and O<sub>2</sub>. This offers the possibility to precisely vary the oxygen to metal flux ratio, which may serve as an additional optimization parameter to increase  $\mu_{\rho}$ by controlling the film stochiometry and the local bonding arrangement around the dopant atom. Nevertheless, this potential advantage remained unexplored due to limited understanding of the mechanisms that control the generation and transport of the free electrons in AZO and their relation to the deposition conditions. The present study focuses on the effect of *in situ* controlled variation in the oxygen partial pressure during RP MS on the free electron mobility of AZO thin films grown at different Al concentrations and substrate temperatures.

AZO films were deposited by RP MS using two 2 in. unbalanced magnetrons (FHR, Germany) operated in parallel.<sup>8</sup> Three sets of Zn–Al alloy targets were used with different Al concentrations ( $c_{Al}$ ) of ~1.7, 4.7, and 8.7 at. %, as determined by energy dispersive x-ray spectrometry (EDX). All films were grown on fused silica 7×7 ×0.5 mm<sup>3</sup> substrates, whose temperature was varied in the range of  $T_s$ =22-520 °C using a substrate holder with a boralectric heater (Tectra, Germany). Before each deposition run, the chamber was baked for several hours at  $\sim 90$  °C and then cooled down to RT to reach a base pressure of 1.0  $\times 10^{-7}$  mbar. With the substrate covered by a closely mounted shutter, the magnetron targets were initially presputtered in pure Ar for 5 min, followed by a stabilization of the discharge current and the gas partial pressures under the addition of O2. A fixed Ar flow rate of 105 sccm was employed. Using a capacitance gauge (Pfeiffer CMR 275) the total Ar and O<sub>2</sub> pressure was monitored precisely. For each run, the total pressure during presputtering in Ar only  $(p_1)$ , during deposition in Ar and  $O_2(p_2)$ , and after deposition in Ar and  $O_2$  with switched off magnetrons  $(p_3)$  was determined. Depositions were carried out at  $p_2$  values in the range of  $1.85-2.00 \times 10^{-2}$  mbar. The oxygen partial pressure during film growth was calculated as  $p_{O_2}=p_2-p_1$ . An error of ~5% in  $p_{O_2}$  is attributed mainly to a limited stability of the gas flow controllers. The long-term stability of the Ar and  $O_2$ partial pressures during deposition was controlled by a differentially pumped quadrupole mass spectrometer (HIDEN RGA 201).

The magnetrons were operated in constant voltage mode (cathode voltage  $U \le 750$  V) with a pulse frequency of f=2 kHz. At fixed pumping speed the resulting discharge current and power depend strongly on the oxygen partial pressure  $p_{O_2}$ . While the oxygen gas flow  $(\Phi_{O_2})$  controls the total amount of oxygen available for the reactive process, a fine tuning of  $p_{O_2}$  was performed by varying the magnetron voltage at constant  $\Phi_{O_2}$ . This is based on the gettering of reactive gas by the sputtered metal, a characteristic feature of reactive MS. The final  $p_{O_2}$  value depends on the total amount of sputtered metal, which can be controlled by changing *U*, because the sputtering yield of Zn and Al monotonically increases with rising energy of the Ar ions below 1 keV.<sup>9,10</sup> After determining the optimum  $p_{O_2}$  value, denoted as  $p_{O_2}^*$ , corresponding to a certain magnetron voltage at given  $\Phi_{O_2}$ .

The film thickness (300–400 nm), the refractive index, and the extinction coefficient were determined by spectroscopic ellipsometry (M-2000, J.A Woolam, Inc.). Hall effect measurements in van der Pauw geometry (HMS 3000, ECO-

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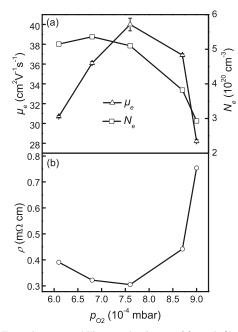


FIG. 1. Free electron mobility  $\mu$ , density  $N_e$  (a), and film resistivity  $\rho$  (b) vs oxygen partial pressure at a fixed substrate temperature of  $T_S$  = 350 °C, a target Al-content of  $c_{\rm Al}$ =1.7 at. %, and an oxygen flow of  $\Phi_{\rm O_2}$ =3.1 SCCM.

PIA) were carried out at a magnetic flux density of 0.51 T and RT, from which the free electron density  $N_e$ , the Hall mobility  $\mu_e$ , and the film electrical resistivity  $\rho$  were derived. The samples were contacted by gold-plated clamps providing Ohmic contacts without any additional contact deposition or bonding. Compositional analysis of the films was performed by means of elastic recoil detection analysis (ERDA) using 35 MeV Cl<sup>7+</sup> ions. The film structure was analyzed by x-ray diffraction (XRD) using a D8 (BRUKER AXS) step scan diffractometer with Cu  $K\alpha$  radiation The size of coherently diffracting domains, i.e., the coherence length *L* indicative of the grain size, was estimated from the wurtzite ZnO (002) peak using the Scherrer formula.

Figure 1 shows a typical dependence of the AZO film  $N_e$ ,  $\mu_e$  (a), and  $\rho$  (b) on the oxygen partial pressure  $p_{O_2}$  adjusted by a variation in the target voltage in the range from 540 to 660 V using a constant oxygen flow of  $\Phi_{O_2}$  = 3.1 SCCM. The data are shown for films grown at  $T_s$  = 350 °C from targets with  $c_{Al}$ =1.7 at. %. In this case, a maximum of  $\mu_e \sim 40 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  together with a minimum resistivity of  $\rho \sim 3 \times 10^{-4} \Omega$  cm is achieved at  $p_{O_2}^* \sim (7.6 \pm 0.4) \times 10^{-4}$  mbar. The electrical resistivity of the AZO films increases rapidly when increasing  $p_{O_2} > p_{O_2}^*$  due to a decrease in both  $\mu_e$  and  $N_e$ . This can be attributed either to Al dopant deactivation or to a decreasing density of oxygen vacancies acting as electron donors caused by an excess of oxygen during film formation. Similar dependences on  $p_{O_2}$  were observed at other  $\Phi_{O_2}$  and  $T_s$ .

Figure 2 shows the dependence of the electrical properties [(a) and (b)] and coherence length L (c) on  $T_S$  for another set of films grown at  $c_{\rm Al}=1.7$  at. % with optimum parameters of  $p_{O_2}^{*}=(8.7\pm0.4)\times10^{-4}$  mbar, U=680 V, and  $\Phi_{O_2}=3.5$  SCCM. The best electrical properties,  $\rho$  $\sim 2.26\times10^{-4} \ \Omega \ {\rm cm}, \ \mu_e \sim 46 \ {\rm cm}^2 \ {\rm V}^{-1} \ {\rm s}^{-1}$ , and  $N_e \sim 6.0$  $\times 10^{20} \ {\rm cm}^{-3}$ , are achieved at an optimum substrate temperature of  $T_S^{*}=350 \ {\rm °C}$ . The present free electron mo-

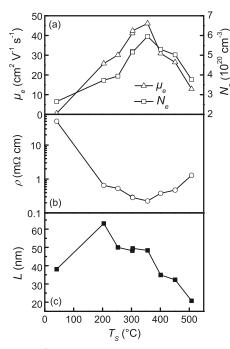


FIG. 2. [(a) and (b)] As for Fig. 1(c) coherence length L vs substrate temperature at an oxygen partial pressure of  $p_{O_2}^*=8.7\pm0.4\times10^{-4}$  mbar, an oxygen flow of  $\Phi_{O_2}=3.5$  SCCM, and a target composition of  $c_{\rm Al}$  = 1.7 at. %.

bility is significantly higher than the highest value of 28 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> reported before for AZO films grown by reactive MS (Ref. 7) and is essentially identical to the best values achieved for PLD (47 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>) (Ref. 3) and rf MS (44 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>).<sup>4</sup> A steep decrease in the mobility is observed both below and above the optimum temperature with a similar trend for the free electron density. This leads to a characteristic  $\rho(T_S)$  dependence with a minimum resistivity at  $T_S^*$ . Strongly absorbing films form at  $T_S \leq 100$  °C, while at higher temperatures low resistivity and transparent films are obtained. The AZO film with the lowest resistivity exhibits an absorption coefficient of  $\alpha$ =460 cm<sup>-1</sup> at a wavelength of 550 nm.

According to the XRD data all AZO films show a pronounced *c*-axis texture, resulting in a strong (002) peak corresponding to wurtzite ZnO. The dependence of the coherence length *L* on  $T_S$  [Fig. 2(c)] qualitatively resembles that of electrical properties, but the maximum *L* is reached already at  $T_S < T_S^*$ . Thus, although the decreasing  $\mu_e$  values above  $T_S^*$  are consistent with a decreasing crystallite size, the observed  $\mu_e(T_S)$  cannot be directly related to the change in crystallinity.

Figure 3 sorts all present data for different target compositions and depositions conditions in a plot of the free electron mobility versus the free electron density. The figure shows a semiempirical model for ionized impurity scattering and impurity clustering developed by Masetti *et al.*,<sup>11</sup> which has been evaluated using parameters for AZO given in Ref. 5. At free electron densities between 10<sup>19</sup> and 10<sup>21</sup> cm<sup>-3</sup>, the result sets a practical limit of  $\mu_e \sim 55$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> to the ionized impurity scattering mobility, which is consistent with the highest value achieved in our work. The model even slightly underestimates the attainable mobility at  $N_e \sim 6$  $\times 10^{20}$  cm<sup>-3</sup>, while at higher free electron densities the calculated mobility is limited to  $\sim 15$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> due to the onset of ionized impurity clustering. A competing description 042103-3

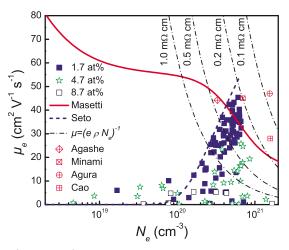


FIG. 3. (Color online) Scatter plot of free electron mobility vs free electron density from present data at different target compositions and from literature for PLD (Ref. 3), rf MS (see Refs. 4 and 16), and RP MS (Ref. 7). The solid line represents a semiempirical curve for ionized impurity scattering and impurity clustering from Ref. 12 with parameters from Ref. 5; the dashed line is from an approximate description of grain barrier limited transport (see Ref. 13). The dash-dotted lines represent contours of constant resistivity.

of grain barrier limited transport in polycrystalline materials has been published by Seto.<sup>12</sup> Depending on the areal density of traps  $N_T$  in the disturbed regions between the grains and the carrier density inside the grains, the traps can be partly or completely filled, leading to varying barrier heights  $\varphi_{B}$  and widths. Electrons can surmount these barriers by thermionic emission. In the case of TCOs  $(LN_e > N_T)$ , the mobility is given by<sup>12</sup>

$$\mu_{\text{Seto}} = \mu_0 \exp(-\varphi_B/kT) = eL(2\pi m^* kT)^{-1/2} \exp(-e^2 N_T^{-2}/8\varepsilon\varepsilon_0 N_e kT),$$
(1)

where  $\mu_0$  represents the in-grain mobility. In contrast to the model of Masetti et al.,<sup>11</sup> Eq. (1) results in a decreasing mobility with decreasing  $N_e$  values and is valid only at  $N_e$  $< 6 \times 10^{20}$  cm<sup>-3</sup>, where impurity clustering does not occur.

As can be seen in Fig. 3, most of the  $\mu_e(N_e)$  data for the target composition of  $c_{Al}=1.7$  at. % are limited by either mechanism. The limiting data points for  $c_{Al}=1.7$  at. % below  $N_e = 6 \times 10^{20}$  cm<sup>-3</sup> have been fitted using the Seto<sup>12</sup> model (1). Although  $\mu_0$  and  $N_T$  are interdependent, it is possible to extract both values because they affect slope and position of  $\mu_{\text{Seto}}(N_e)$ , respectively. A rough best fit yields  $\mu_0 = 75-90 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and  $N_T = (1.60-1.75) \times 10^{13} \text{ cm}^{-2}$ . The latter is in good agreement with data reported by Ellmer and co-worker<sup>5,13</sup> for AZO films grown by MS. Still, our highest mobility values are about a factor of two smaller than the in-grain mobility  $\mu_0$  due to the contribution of electron scattering inside the bulk of the grains at dislocations and point defects, which is not considered in the idealized Seto<sup>12</sup> model. Similar experimental in-grain mobilities of ~100 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, which are close to our estimated  $\mu_0$  values, have been obtained by spectroscopic ellipsometry.<sup>14</sup> Finally, the significant decrease in the maximum attainable mobility at  $N_e \ge 6 \times 10^{20}$  cm<sup>-3</sup> for the larger Al fractional compositions is qualitatively consistent with the harmful role of Al impurity clustering.

A general trend of increasing optimum  $\mu_e$  values along with improving of the film crystallinity toward lower  $c_{A1}$  has

TABLE I. Optimum values of the AZO film electrical parameters and corresponding coherence length values obtained for different target Al concentrations.

<i>c</i> <sub>Al</sub> (at %)	L (nm)	$\mu_e \ ({ m cm}^2 { m V}^{-1} { m s}^{-1})$	$N_e (10^{20} \text{ cm}^{-3})$	$(10^{-4} \Omega \text{ cm})$
$8.7 \pm 1.7$ $4.7 \pm 1.7$	19 34	7.0 24.6	4.3 6.9	20.7 3.62
$1.7 \pm 0.5$	48	46.6	6.0	2.26

been observed (Table I). According to ERDA data, the Al concentration in the films at  $T_S = T_S^*$  agrees with that in the target within the error limits. Thus, the Al donor activation rate even decreases with increasing  $c_{A1}$  because higher  $c_{A1}$ does not lead to higher optimum  $N_e$  values. Taking into account recent studies suggesting a solubility limit of 3 at. % for Al in ZnO,<sup>15</sup> the trend in  $\mu_e$  can be understood assuming that at  $c_{Al} > 3$  at. %, there is an excess of electrically inactive Al, which might locally drive the formation of ZnO-Al<sub>2</sub>O<sub>3</sub> homologous phases<sup>15</sup> and affect film electrical properties by increasing the free electron scattering.

In summary, a fine tuning of the oxygen partial pressure using gettering of  $O_2$  by sputtered metal atoms, along with substrate temperature variation, yielded AZO films with the highest mobility values so far reported for the present deposition technique. The best value of 46 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> is comparable to the highest values achieved in AZO films grown by techniques that are less efficient in terms of either scalability or cost, and is approaching the practical limit of 55 cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>. Depending on the free electron density and the aluminum content, the electron mobility is either controlled by ionized impurity scattering in connection with impurity clustering or grain boundary limited transport.

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## Highly dense amorphous Nb<sub>2</sub>O<sub>5</sub> films with closed nanosized pores

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This study is focused on tailoring the porosity of Nb<sub>2</sub>O<sub>5</sub> films during reactive pulsed magnetron sputtering. Dense amorphous films containing nanopores only in deeper regions have been grown at a high rate using substrate temperatures below 60 °C. The films exhibit a high refractive index,  $n_{400}=2.54$ , a low extinction coefficient,  $k_{400}\sim 6\times 10^{-4}$ , a low mechanical stress (-90 MPa), and a negligible thermal shift. The specific depth distribution of the nanopores is believed to be the reason for the optimum trade-off between a high refractive index and low mechanical stress. © 2009 American Institute of Physics. [DOI: 10.1063/1.3212731]

The need for cost-effective materials combining a high refractive index, low optical extinction, low mechanical stress, and amorphous homogeneous microstructure for optical applications has stimulated active research on thin films of oxides such as HfO<sub>2</sub>, Ta<sub>2</sub>O<sub>5</sub>, and Nb<sub>2</sub>O<sub>5</sub>.<sup>1-3</sup> Films of  $Nb_2O_5$  produced by magnetron sputtering remain amorphous up to a temperature of 500 °C.<sup>4,5</sup> In addition, their refractive index  $n(\lambda = 400 \text{ nm}) \sim 2.60$  (Refs. 6 and 7) is significantly higher than that of HfO<sub>2</sub> and Ta<sub>2</sub>O<sub>5</sub>. However, Nb<sub>2</sub>O<sub>5</sub> films with the highest refractive index, produced by either reactive pulsed magnetron sputtering (RPMS)<sup>4</sup> or ion plating,<sup>8</sup> often exhibit a relatively high compressive stress ( $\sigma > 300$  MPa) (Ref. 9) and an extinction coefficient  $k(\lambda = 400 \text{ nm}) > 0.002$ (Refs. 6 and 7) leading to inferior performance of the resulting optical devices. There is evidence in the literature that the incorporation of nanopores may cause relaxation of the mechanical stresses in amorphous films,<sup>4</sup> but the pores may absorb atmospheric water, which will then lead to a thermal shift, i.e., to a thermally variable refractive index. Tailoring film porosity would enable one to achieve an optimum tradeoff between mechanical and optical film properties. The volume fraction of pores in the growing film may be reduced by increasing surface adatom mobility.<sup>10</sup> Such an increase can be accomplished by either energetic particle bombardment or raising substrate temperature. The present study focuses on the porosity control of Nb<sub>2</sub>O<sub>5</sub> films using high-growth rate RPMS with variable oxygen partial pressure, different magnetron-substrate configurations, and substrate temperatures.

Nb<sub>2</sub>O<sub>5</sub> films of a thickness of 490 to 550 nm were deposited by RPMS using two 2 in. unbalanced magnetrons (MightyMAK, U.K.) operated in parallel<sup>11</sup> and equipped with high purity (99.95%) Nb metallic targets (Kurt J. Lesker, U.K.). The films were grown using either two magnetrons with axes directed to the sample holder edges (offnormal deposition, with the angle between the magnetron axis and the substrate normal being  $17^{\circ}$ )<sup>11</sup> or a single magnetron with an axis perpendicular to the substrate. The sub-

strates were either  $(10 \times 10 \times 0.5)$  mm<sup>3</sup> plates of UV-grade fused silica,  $(20 \times 20 \times 0.3)$  mm<sup>3</sup> plates of crystalline Si(100), or 3 -in. wafers of crystalline Si. The substrates were at floating potential while their temperature,  $T_S$ , was kept either below 60 °C (no intentional heating) or at a fixed value of 370 °C by means of a sample holder with a boralectric heater (Tectra, Germany). In both cases, the distance from the magnetron to the substrate was 55 mm. The magnetrons were operated in a constant power mode (400-625 W per magnetron), with voltage and current values in the range of 175-500 V and 0.8-3.6 A, respectively, and frequencies of 5 and 16.67 kHz. The chamber was baked to reach a base pressure of  $3.0 \times 10^{-7}$  mbar before deposition. The magnetron targets were initially presputtered in pure Ar for 5 min followed by stabilization of the discharge current and gas partial pressures upon addition of  $O_2$ . Before striking the magnetron discharge, the partial pressures of Ar  $(p_{Ar}=2.29\times10^{-2} \text{ mbar})$  and  $O_2$   $[p_{O_2}=(3.03-6.68)$  $\times 10^{-3}$  mbar] were monitored precisely with a capacitance gauge (Pfeiffer CMR 275).

An automated Langmuir probe (SmartProbe, Scientific Systems Ltd., USA) was employed to determine the spatial distribution of the plasma ion density,  $N_i$ , with a resolution of 1 cm in the plane parallel to the substrate at a distance of 3 cm from the latter. The film optical properties were characterized using an M-2000 spectroscopic ellipsometer (J.A. Woolam Co Inc., USA) at a photon energy of 1.24-3.4 eV. Spectroscopic ellipsometry (SE) data were processed with the aid of WVASE software, and were fitted to a four-layer model involving air, surface roughness, the oxide film, and the substrate. The surface roughness was modeled using a 50% air and 50% bulk oxide Bruggeman effective medium approximation.<sup>12</sup> The Nb<sub>2</sub>O<sub>5</sub> complex refractive index, N=n+ik, being consistent with the Kramers–Kronig relations, was obtained using a Cody-Lorentz parameterized model.<sup>3</sup> Adding a linear grading of *n* and *k* values does not improve substantially fit results for optimized Nb<sub>2</sub>O<sub>5</sub> films; however, it decreases the fit mean square error by a factor of 2-3 for unoptimized ones. In the latter case, the film parameters, including thickness-averaged n values, match (within the experimental error) those obtained by a simpler model. The

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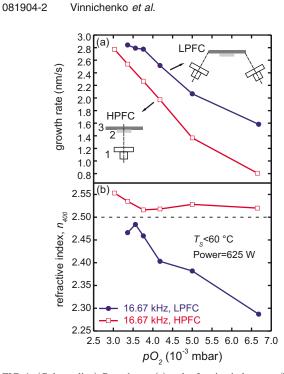


FIG. 1. (Color online) Growth rate (a) and refractive index,  $n_{400}$  (b), of films produced at different oxygen partial pressures, a substrate temperature of  $T_s < 60$  °C, and magnetron power of 625 W. The insets show the configuration of the magnetron (1) with respect to the substrate (2) fixed to the substrate holder (3).

values of the refractive index and the extinction coefficient at a wavelength of 400 nm (3.1 eV),  $n_{400}$  and  $k_{400}$ , respectively, were compared for different samples, and the error involved in the former did not exceed  $\Delta n_{400} \sim \pm 0.01$ . The sensitivity limit of the SE measurement of the extinction coefficient was estimated to be about  $k_{400} \sim 5 \times 10^{-4}$ . For selected samples, the thermal shift was determined from transmission measurements with an OptiMon spectrophotometer as the relative change in the refractive index due to heating in a vacuum from room temperature to 100 °C.<sup>13</sup> The film stress was evaluated with Stoney's equation measuring the change in the curvature of the 3 in. Si wafers.<sup>13</sup> Negative and positive values correspond to tensile and compressive stress, respectively.

The hydrogen depth profile in the films was determined by nuclear reaction analysis (NRA) using  ${}^{15}N^{2+}$  ions at energies above 6.3 MeV. The microstructure of selected films was characterized by high-resolution cross-sectional transmission electron microscopy (X-TEM) using a CM20 instrument (Philips, Netherlands) at an acceleration voltage of 200 kV, resulting in a point-to-point resolution of 0.25 nm. X-TEM specimen preparation followed a standard procedure described in more detail elsewhere.<sup>14</sup> The ion energy and the angle were chosen in such a way as to minimize ion milling artifacts.

Figure 1 shows the dependence of the growth rate (a) and the refractive index,  $n_{400}$ , (b) on the oxygen partial pres-

sure  $p_{O_2}$  for films grown using two different magnetronsubstrate configurations. The films were deposited on unheated substrates  $(T_S < 60 \degree \text{C})$  at the highest magnetron power of 625 W. In the case of two magnetrons and offnormal deposition, the ion density at the substrate center is  $N_i \sim 3 \times 10^{10}$  cm<sup>-3</sup>. This is denoted as a low plasma flow configuration (LPFC). In the high plasma flow configuration (HPFC) for a single magnetron with an axis perpendicular to the substrate surface, the central beam of the unbalanced magnetron provides an ion density at the substrate of  $N_i$  $\geq 2.8 \times 10^{11}$  cm<sup>-3</sup> for the entire range of  $p_{O_2}$  and at the two frequencies used. Increasing  $p_{O_2}$  causes a reduction in the film growth rate from 2.84 to 1.58 nm/s for LPFC, and a decrease from 2.77 to 0.80 nm/s for HPFC due to stronger coverage of the Nb magnetron target with an oxide layer resulting from an increased supply of oxygen.<sup>15</sup> For LPFC, the films grown at  $p_{O_2}=3.56 \times 10^{-3}$  mbar exhibit a highest refractive index of  $n_{400}=2.48$  and a relatively high extinction coefficient  $k_{400}=0.02$  (not shown). These parameters decrease to  $n_{400}=2.29$  and  $k_{400}<0.001$  at  $p_{O_2}=6.68$  $\times 10^{-3}$  mbar. In contrast, films grown at HPFC have a refractive index  $n_{400} = 2.52 - 2.55$  and  $k_{400} < 6 \times 10^{-4}$  over the whole range of  $p_{O_2}$ . In case of deposition onto heated substrates ( $T_s$ =370 °C) at LPFC, a reduced magnetron power of 500 W and operation frequency of 5 kHz ( $p_{O_2}$ =3.36  $\times 10^{-3}$  mbar) were necessary to achieve an optimum combination of a high refractive index ( $n_{400}$ =2.46) and a low extinction coefficient  $(k_{400} < 5 \times 10^{-4})$ .

The films grown for HPFC without substrate heating (Table I) have a low tensile stress of -90 MPa and a negligible thermal shift in +0.06% in addition to high *n*. In contrast, the films grown for LPFC, but at an elevated substrate temperature, show a higher tensile stress (-132 MPa) and a much larger thermal shift in -2.0%.

Figure 2 shows X-TEM images of the films grown without heating for HPFC [(a)-(c)] and onto heated substrates for LPFC [(d)-(f)]. The common feature of both types of films is the dense nonporous amorphous layer near the  $Nb_2O_5/Si$ interface, with a thickness of  $\sim 5$  nm [sample A, Fig. 2(c)] and  $\sim 5$  nm [sample B, Fig. 2(f)]. Then, in the first case, a 10 nm thick layer consisting of isolated pores [Fig. 2(c)] forms followed by a dense amorphous microstructure with discontinuous vertical arrays of pores [Fig. 2(a)]. The characteristic pore size is 2 nm. The NRA depth profile shows that the hydrogen content in the film decreases from 1.2 at. % at the surface to values below the detection limit inside the film. The low hydrogen content is mainly attributed to atmospheric water adsorption at the surface rather than to water uptake by the pores. Accordingly, the thermal shift becomes negligible.

Conversely, for LPFC on heated substrates, no such a porous layer is observed near the film-substrate interface, and vertical arrays of pores start to form directly from the

TABLE I. Deposition parameters, thermal shift, and stress of the two different series of Nb<sub>2</sub>O<sub>5</sub> samples.

Sample	$T_S$ (°C)	$P_m$ (W)	$V_m$ (V)	$f_m$ (kHz)	No. of magnetrons	$\binom{N_i}{(10^{11} \text{ cm}^{-3})}$	Configuration	Thermal shift (%)	Stress (MPa)
A	<60	625	175	16.67	1	2.8	HPFC	+0.06	-90
В	370	500	500	5	2	0.08	LPFC	-2.0	-132

FIG. 2. X-TEM images of films grown onto unheated substrates in HPFC mode [(a)–(c)], and at  $T_S$ =370 °C in LPFC mode [(d)–(f)].

dense area [Figs. 2(d) and 2(f)], then continue through the film and, finally, open at the film surface [Fig. 2(e)]. The characteristic pore size is a little larger than described above, i.e.,  $\sim 3$  nm, while the pore density is significantly higher. These findings are in agreement with the NRA depth profile showing a high H concentration decreasing from 11 to 7 at. % with film thickness. The significant uptake of atmospheric water by the open pores explains the high thermal shift observed in these films.

The increased particle bombardment for HPFC at normal incidence is the reason for the observed formation of a dense amorphous microstructure with a small volume fraction of closed nanosized pores. In our case of reactive unbalanced magnetron sputtering, positive plasma ions contribute appreciably to the thermal power at the substrate<sup>16</sup> for plasma ion densities higher than 10<sup>11</sup> cm<sup>-3</sup>. In addition, energetic reflected neutral particles of Ar might play a role because their mass is appreciably lower than that of Nb target atoms. Since the refractive index of the material correlates with its density,<sup>5</sup> the densification of the amorphous film produces the high *n* values obtained irrespectively of  $p_{O_2}$ , and occurs for  $N_i > 10^{11}$  cm<sup>-3</sup>. The decreased particle bombardment for LPFC, as indicated by the low plasma ion density  $(N_i \sim 10^{10} \text{ cm}^{-3})$ , in conjunction with off-normal deposition, favors shadowing effects which in turn lead to the porous microstructure observed even in the case of growth at an elevated substrate temperature  $T_S$ =370 °C. A higher degree of porosity explains the lower refractive index values of the films grown for LPFC in comparison with those for HPFC (Fig. 1).

The influence of the nanosized pores on the film refractive index, thermal shift, and mechanical stress in the amorphous Nb<sub>2</sub>O<sub>5</sub> films is interpreted in light of a recently developed model.<sup>17</sup> It is assumed that nonporous films show a high refractive index and a zero thermal shift while having a high compressive stress. The model suggests that there is an optimum volume fraction of closed pores of about 1% and, while the refractive index is still high, the film has a low Journal Reprint

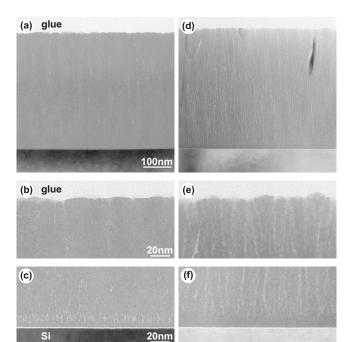
stress and a thermal shift close to zero. The properties of the  $Nb_2O_5$  samples deposited for HPFC match closely the predictions of this model, thus providing an optimum trade-off between the discussed optical and mechanical film parameters. Further enhancement in porosity and dominance of open pores increase the tensile stress, reduce significantly the refractive index, and lead to a substantial negative thermal shift. This situation corresponds to the films produced at LPFC.

 $n_{400}$ =2.54 ( $n_{550}$ =2.36) is among the highest refractive index values achieved to date using magnetron sputtering<sup>4</sup> and is only a little below the value  $n_{550}$ =2.40 for films produced by ion plating.<sup>8</sup> However, the films with the highest refractive index in Ref. 4 exhibit a mechanical stress of -200 MPa while ion plated films show an extinction coefficient in the visible spectral range of  $k_{515}$ =5×10<sup>-4</sup> (Ref. 8) which is commensurate with our values in the UV spectral range. In addition, the growth rate of the films studied in the present work is the highest compared to the values available in literature.<sup>4,8</sup>

In summary, adjusting the deposition geometry and plasma ion density during RPMS enables one to tailor the porosity of amorphous Nb<sub>2</sub>O<sub>5</sub> thin films using a threedimensional growth mode. Films with a low extinction coefficient, a high refractive index, low tensile stress and negligible thermal shift have been obtained during deposition for plasma ion densities above  $10^{11}$  cm<sup>-3</sup> and substrate temperatures below 60 °C. These properties have been explained by the formation of a dense amorphous microstructure with a specific depth distribution of closed nanopores. In contrast, films grown at 370 °C using low plasma ion densities (~ $10^{10}$  cm<sup>-3</sup>) are less dense with pores being open at the film surface, and show a significant thermal shift, higher tensile stress and lower refractive index.

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## Anomalous Hall resistance in Ge:Mn systems with low Mn concentrations

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Taking Mn doped Germanium as an example, we evoke the consideration of a two-band-like conduction in diluted ferromagnetic semiconductor (FMS). The main argument for claiming Ge:Mn as a FMS is the occurrence of the anomalous Hall effect (AHE). Usually, the reported AHE (1) is observable at temperatures above 10 K, (2) exhibits no hysteresis, and (3) changes the sign of slope. We observed a similar Hall resistance in Mn implanted Ge with the Mn concentration as low as 0.004%. We show that the puzzling AHE features can be explained by considering a two-band-like conduction in Ge:Mn. © 2009 American Institute of Physics. [doi:10.1063/1.3257363]

Diluted ferromagnetic semiconductors (FMSs) exhibit strong magnetotransport effects, namely negative magnetoresistance (MR) and anomalous Hall effect (AHE),<sup>1-3</sup> and provide the possibility to control the spin by an external electric field. Ferromagnetic GaMnAs reveals hysteretic AHE, which mimics its magnetization, and allows the determination of its magnetic parameters by measuring the Hall resistance. The observation of AHE is considered as one of the important criteria for FMS to be intrinsic.<sup>4</sup> Mn doped Germanium provides an alternative of FMS, as predicted by Dietl et al.<sup>5</sup> Its compatibility with conventional microelectronics makes it more promising for industry application. We notice that pronounced MR and AHE have been reported in the Ge:Mn system<sup>6-13</sup> independent of the formation of MnGe precipitates or not, as well as in Cr doped Ge.<sup>14</sup> By scrutinizing the published data on Ge:Mn, one can observe three features in the reported AHE. First, most of the AHE curves shown were recorded at temperatures above 10 K. Indeed, Riss et al.<sup>9</sup> reveal only ordinary Hall effect below 10 K. Second, no hysteresis in AHE curves has been observed, despite the observation of a clear hysteresis in magnetization, which is much different from the case of III-Mn-V and ZnMnTe.  $^{1\!-\!3}$ Third, the Hall curve changes the sign of slope at lower temperatures, usually between 10 and 50 K. Obviously, the correlation between magnetization, MR, and AHE, which is a hallmark of III-Mn-V and ZnMnTe FMS, 1-3 has not been proven for Ge:Mn. In this letter, we report the observation of an anomalous Hall resistance in Mn implanted Ge with the Mn concentration as low as 0.004%, in which neither ferromagnetism nor paramagnetism has been measured. By considering two types of carriers, all the puzzling Hall-resistance features can be explained. Moreover, the two-band-like conduction probably also explains the Hall resistance at large fields for InMnAs (Ref. 15) and InMnSb.<sup>16</sup>

Intrinsic Ge(001) wafers were implanted with Mn ions at 300 °C to avoid amorphization. We varied the ion fluence to get samples with a large range of Mn concentrations and correspondingly different structural and magnetic properties (see Table I). Magnetotransport properties were measured using Van der Pauw geometry with a magnetic field applied perpendicular to the film plane.

As shown in Table I, we examined samples with a wide range of Mn concentration. Mn<sub>5</sub>Ge<sub>3</sub> precipitates have been observed in sample Ge02 and Ge03 by synchrotron radiation x-ray diffraction (SR-XRD) (Ref. 17) at the Rossendorf beamline (BM20) at the ESRF, but not in sample Ge01, and are not expected in sample Ge19. SR-XRD has been proved to be sensitive for detecting nanocrystalline precipitates.<sup>18,1</sup> Correspondingly, ferromagnetism was observed only in sample Ge02 and Ge03. Down to 5 K, only diamagnetism was probed for sample Ge01, identical to a virgin Ge sample. Note that independent of the formation of precipitates, a fraction of Mn ions has been confirmed by spectroscopic methods to be diluted inside the Ge matrix, resulting in p-type doping,<sup>20-22</sup> as well as by electrical transport measurements as shown in Fig. 1(a). In sharp contrast to the structural and magnetic properties, similar Hall effects are probed for all samples as shown in Fig. 1. Indeed, the shape and temperature dependence of the Hall-resistance curves are similar to those reported in literature.<sup>7–13</sup> Deng *et al.*<sup>12</sup> attribute the sign change in Hall curves to the hopping conductivity. However, this cannot explain the vanishing of AHE below 10 K.

The nonferromagnetic nature of sample Ge19 and Ge01 indicates that the observation of anomalous Hall resistance is not necessarily related to ferromagnetism. Actually, similar Hall curves have been observed in materials with a twoband-like conduction.<sup>23–25</sup> Figure 2 shows the resistance versus temperature for all samples. Basically, below 10 K the resistance is weakly dependent on temperature and a transition occurs above 10 K. The activation energy is only several meV for all samples. It cannot be the thermal activation of holes from the Mn acceptor levels in Ge since the Mn single and double acceptor level is as deep as 160 meV from the valence band and 370 meV from the conduction band,<sup>26</sup> re-

TABLE I. Sample identification (ID), Mn concentration (Mn conc.), sheet hole concentration (hole conc.), and magnetic properties.

	Mn conc.	Hole conc.	Properties		
ID	(%)	$(cm^{-2})$	Precipitates?	Ferromagnetic?	
Ge19	0.004		No	No	
Ge01	0.2	$6.5 \times 10^{12}$	No	No	
Ge02	2	$1.1 \times 10^{13}$	Mn <sub>5</sub> Ge <sub>3</sub>	Yes	
Ge03	10	$2.0 \times 10^{13}$	Mn <sub>5</sub> Ge <sub>3</sub>	Yes	

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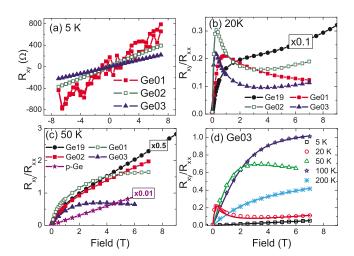


FIG. 1. (Color online) (a) Hall resistance  $(R_{xy})$  at 5 K: only ordinary Hall effect has been observed. [(b) and (c)] The ratio between  $R_{xy}$  and sheet resistance at zero field  $(R_{xx})$  at 20 and 50 K, respectively. An anomalous Hall resistance appears and the sign of slope is changed at lower fields (20 K) or at larger fields (50 K). (d)  $R_{xy}/R_{xx}$  at different temperatures for sample Ge03: the symbols are experimental data, while the solid lines are fits using Eq. (1). A Ga-doped Ge wafer with a hole concentration of 1.5  $\times 10^{16}$  cm<sup>-3</sup> was measured for comparison and only ordinary Hall effect is observed as shown in (c). For better visibility some curves are multiplied by the factors indicated.

spectively. The small activation energy also cannot be a shallow acceptor level due to the large difference in the magnitude and temperature-dependence of resistance compared to Ga or In-doped Ge.<sup>27</sup> Moreover, between 10 and 50 K the temperature dependence of the resistance is rather a universal feature in Mn doped Ge.<sup>6,7,9,28,29</sup> An interpretation of the transition at around 10 K is plausible by considering the ground and the first excited states of  $Mn^{2+}(d^5+2h)$ .<sup>30</sup> Within such a scenario, a two-band-like conduction (or two types of carriers) is expected at above 10 K or even lower if the Mn concentration is very small as shown in Fig. 2. In the next step we fit the Hall curves using a two-band model described in Refs. 23 and 24.

The dependence of the ratio between Hall and sheet resistance  $(R_{xy}/R_{xx})$  on the magnetic field *B* is given by

$$R_{xy}/R_{xx} = \frac{x_1\mu_1(1+\mu_1^2B^2)+x_2\mu_2[1+\mu_2^2B^2]}{1+(x_1\mu_2+x_2\mu_1)^2B^2}B,$$
 (1)

where

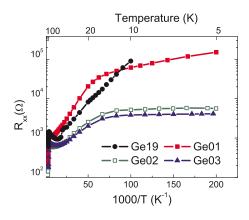


FIG. 2. (Color online) Temperature dependent sheet resistance at zero field  $(R_{xx})$ . For sample Ge19 the resistance is too large and can only be measured down to 10 K. Basically, below 10 K the resistance of sample Ge02 and Ge03 is weakly dependent on temperature.

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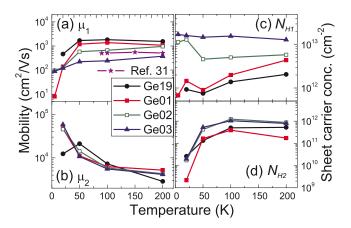


FIG. 3. (Color online) Fitting results of Hall curves at different temperatures. [(a) and (b)] Temperature dependent mobilities, data extracted from Ref. 31 is shown for comparison. [(c) and (d)] Temperature dependent sheet carrier concentrations. Lines are guides for eyes.

$$x_1 = \frac{G_1}{G_1 + G_2}, \quad x_2 = \frac{G_2}{G_1 + G_2},$$
 (2)

*G* and  $\mu$  are conductance and mobility, respectively. The subscript numbers denote the two types of carriers. Numbers "1" and "2" label the carriers in the ground and the first excited state, respectively. In this formula, we assume spherical Fermi surfaces and use the zero-field sheet resistance ( $R_{xx}$ ) to relate the two-carrier conductance,<sup>23</sup>

$$1/R_{xx} = G = G_1 + G_2, (3)$$

where  $x_1$ ,  $\mu_1$ , and  $\mu_2$  in Eq. (1) are fitting parameters. At 5 K, for sample Ge01, Ge02, and Ge03 we only have carriers of type 1. With increasing temperature carriers of type 2 are activated and  $x_1$  decreases. Taking this as a restriction, we can fit all measured results. Figure 1(d) shows the comparison between experiments and fitting. All the features are reproduced. In Fig. 3 we list the fitting results, i.e., temperature dependent mobility and sheet carrier concentration for two types of carriers. Despite the number of fitting parameters, it is plausible to draw conclusions according to the overall trend by examining all samples. The sheet carrier concentration  $(N_{H1})$  of type 1 carriers is weakly dependent on temperature [Fig. 3(c)], while  $N_{H2}$  is increased above 10 K and becomes saturated at high temperatures [Fig. 3(d)]. The dependence of  $\mu_1$  on temperature is nonmonotonic. This is expected given the fact of a large hole concentration in those samples. The ion implantation results in an effective Ge:Mn layer thickness of around 100 nm. Then the concentration of type 1 carriers is in the range between  $10^{17}$  and  $10^{18}$  cm<sup>-3</sup>. In this range a peak of hole mobility in Ge depending on temperature was observed.<sup>31</sup> The concentration of type 2 carriers is mostly below  $10^{17}$  cm<sup>-3</sup> and  $\mu_2$  increases monotonically with decreasing temperature.

Equation (1) neglects the influence of a possible paramagnetism inside the samples. The paramagnetism can be included as an anomalous Hall term using a Brillouin function.<sup>24</sup> However, we have not observed any paramagnetic component using superconducting quantum interference device magnetometry down to 5 K with fields up to 7 T for sample Ge19 and Ge01. The quench of the magnetic moments from Mn ions is somehow surprising, and has also been observed in Mn doped Si.<sup>32</sup> Nevertheless, we also refined Eq. (1) by including a Brillouin function in order to 172103-3 Zhou et al.

account for possible magnetic field dependent mobilities. However, using the Brillouin function the fitting of the Hall curve at large fields is not possible because the fitted Hall curve changes the sign of slope with increasing field. This is in contrast to the Hall data probed on Mn implanted Ge where the sign of slope remains positive at large fields.<sup>9</sup> The simple two-band-like picture described by Eq. (1) also qualitatively explains the saturation of the positive MR at large fields. Namely, above 10 K a large positive MR is usually observed in Ge:Mn (also in our samples) and saturates at large fields,<sup>7,9,13,28</sup> which is a typical feature of the two-band MR.<sup>23</sup>

In summary, we observed an anomalous Hall resistance in Mn implanted Ge with the Mn concentration as low as 0.004%. By considering two types of carriers participating in the conduction, we can explain the puzzling Hall-resistance curves reported in the literature and in this letter. A multiplepath-like conduction can mislead the interpretation of materials as FMS. Possibly, it also can make an add-on effect to the AHE observed in InMnAs (Ref. 15) and InMnSb,<sup>16</sup> as well as in Zn<sub>0.9</sub>Mn<sub>0.075</sub>Cu<sub>0.025</sub>O.<sup>33</sup>

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# In situ observation of secondary phase formation in Fe implanted GaN annealed in low pressure $N_2$ atmosphere

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The formation of secondary phases in Fe implanted GaN upon annealing in low pressure  $N_2$ -atmosphere was detected by means of *in situ* x-ray diffraction and confirmed by magnetization measurements. A repeatable phase change from Fe<sub>3</sub>N at room temperature and Fe<sub>3-x</sub>N at 1023 K was observed *in situ*. The phase transformation is explained by the change of lattice site and concentration of nitrogen within nitrides. The diffusion of Fe toward sample surface and oxidation with increasing annealing cycles limits the availability of secondary phase and hence the repeatability. At high temperature GaN dissolves and Ga as well as Fe oxidize due to presence of residual oxygen in the process gas. The ferromagnetism in the samples is related to nanometer sized interacting Fe<sub>3-x</sub>N crystallites. © 2009 American Institute of Physics. [doi:10.1063/1.3271828]

GaN is a wide band gap semiconductor that has been studied intensively in the last years because of its potential field of applications like in optoelectronics, plasmonics, as well as for high power electronics. By doping with transitional metals like Fe it might also be a diluted magnetic semiconductor (DMS) with a Curie temperature above room temperature (RT) and could then be used for spintronics.<sup>1</sup> There are many experimental studies reporting ferromagnetism at RT in Fe doped GaN. In a real DMS, with magnetic atoms randomly substituting cation sites, ferromagnetic coupling is supposed to be due to the indirect exchange coupling between magnetic impurities mediated by holes.<sup>1–4</sup> Experimental observation of strong-coupling effects in a DMS  $Ga_{1-x}Fe_xN$  was reported by Pacuski *et al.*<sup>5</sup> Robust ferromagnetism in the region of insulator-to-metal transition was predicted for high hole densities. However, there are also other possible sources of ferromagnetism like spinodal decomposition of Fe or ferromagnetic secondary phases. The detection of those is rather difficult. Bonanni et al.<sup>6</sup> prepared GaN:Fe layers by metalorganic chemical vapor deposition (MOCVD) and observed ferromagnetism that was partially accounted to the spinodal decomposition and non-uniform distribution of Fe-rich magnetic nanocrystals. Kuwabara et al.' reported the formation of nanoclusters and superparamagnetic behavior in GaN:Fe epilayers prepared by rfplasma-assisted molecular beam epitaxy. In case of ion implantation the reports from different groups are quite controversial. Theodoropoulou et al.<sup>8</sup> and Shon et al.<sup>9,10</sup> did not relate ferromagnetic response to secondary phases after transition ion implantation into semiconductors. In our experiments, however, the formation of  $\alpha$ -Fe nanoclusters, that were responsible for ferromagnetic response, was observed.<sup>11</sup> Though the appearance of such precipitates is not desired in a DMS they might be useful for certain applications.<sup>12</sup> Li *et al.*<sup>13</sup> detected co-occurrence of  $\alpha$ -Fe and ε-Fe<sub>3</sub>N in MOCVD prepared GaN:Fe films and pointed out the role of nitrogen pressure and structural disorder in the formation of Fe-rich phases. Bonanni et al.<sup>14</sup> have shown

that the controlled aggregation of magnetic ions in a semiconductor can be affected by the growth rate and doping with shallow impurities.

Recently we reported predominant formation of epitaxially oriented  $\alpha$ -Fe nanoclusters if Fe-doped samples were annealed in a N<sub>2</sub> flow at 1.1 bar pressure.<sup>11</sup> In this paper we report the formation of  $\varepsilon$ -Fe<sub>3-x</sub>N with x < 1 that builds up during annealing at 1073 K in 0.5 bar N<sub>2</sub> and the reversible transformation to  $\varepsilon$ -Fe<sub>3</sub>N during cooling down to RT.

P-type (Mg) doped ( $\sim 2 \times 10^{17}$  cm<sup>-3</sup>) single crystalline wurtzite GaN(001) films of about 3  $\mu$ m thickness epitaxially grown by metal organic vapor phase epitaxy on sapphire (001) were used. Samples, 7° tilted relative to the ion beam to avoid channeling, were implanted with 195 keV <sup>57</sup>Fe ions with fluence  $\Phi = 4 \times 10^{16}$  cm<sup>-2</sup> (peak Fe concentration of 4 at. % at the projected range  $R_p = 85$  nm according to TRIM<sup>15</sup>), keeping the samples at RT. In order to reduce the implantation damage and to investigate the formation of secondary phases the implanted samples were annealed at 1073 K in a low pressure  $N_2$ -atmosphere(0.5 bar) within several minutes. The annealing experiments along with the in situ x-ray diffraction characterization were performed at the Rossendorf beamline at the ESRF in Grenoble with a x-ray wavelength of  $\lambda = 0.124$  nm. The annealing chamber was equipped with a boron nitride heater, controlled by a Eurotherm controller, gas inlet and a half sphere beryllium dome. The temperature was measured by a PtRh/Pt thermocouple placed on top of the heater. The gas pressure in the chamber was limited to 0.5 bar with a flow of about 40  $1 \cdot \text{min}^{-1}$ . The purity of the N<sub>2</sub> gas (99.9999%) was limited by the setup with an oxygen contamination in the ppm range.

A Pilatus 100 K two-dimensional (2D) pixel detector was used to record 2D diffraction pattern. Additionally, a scintillation counter was used for  $2\theta$ - $\omega$ -scans. Generally, for clusters in the range of some nm the signal to background ratio is very low. In order to increase the signal to background ratio to an acceptable level the acquisition time of the 2D detector was set to 10 s and those of the scintillation counter to 5 s per point. The investigations of the magnetic properties were performed with a Quantum Design MPMS

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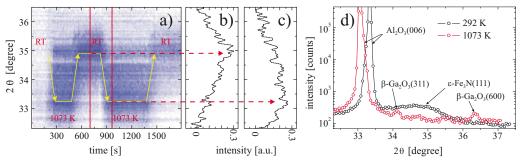


FIG. 1. (Color online) (a) Time dependent  $2\theta$ -scans between  $32.3^{\circ}$  and  $36.5^{\circ}$  at  $\omega = 17.7^{\circ}$ . The presentation is based on the line profiles from the 2D detector with an 10 s acquisition time each. Intensity is color/grayscale coded with dark color representing higher intensity. On the right two profiles are presented for the (b) RT and (c) 1073 K scans. (d)  $2\theta$ - $\omega$  coupled scans at RT and 1073 K recorded after third cycle with a scintillation counter.

superconducting quantum interference device magnetometer. For the evaluation of the 2D detector exposures rectangular areas with dimensions  $2\theta$  from  $32.3^{\circ}$  to  $36.5^{\circ}$  and  $\chi$  $=\pm 0.2^{\circ}$  (angle  $\perp$  to the scattering plane) were integrated over  $\chi$  and assumed as line scans. Those line scans are represented over annealing/cooling time with the color/ grayscale coded intensity in Fig. 1(a). Single examples of line scans are given in Figs. 1(b) and 1(c) for RT and 1073 K, respectively. Prior to the annealing procedure (time t=0 at RT) no reflexes from secondary phases were detected [see Fig. 1(a)]. After about 200 s annealing, at 1073 K, a broad reflex starts to evolve in the region between 32.5° and 35° with a local maximum at about 33.3°. The peak shift due to the lattice expansion from RT to 1073 K is in the order of 0.1° and can be neglected. The position of the maximum fits well to the pattern of FeN(200) (33.46° at RT). However, because of the broadness of the reflex, other nitrides like disordered  $\varepsilon$ -Fe<sub>3-x</sub>N(002) or  $\zeta$ -Fe<sub>2</sub>N(102) can also be taken into account. Disordered in this sense means the redistribution of mainly N atoms within the structure and can be described by the transfer of N from Fig. 2(b) and 2(c) Wyckoff site position in the  $\varepsilon$ -Fe<sub>3-x</sub>N-phase.<sup>16</sup> From symmetry reasons the formation of  $\varepsilon$ -Fe<sub>3-x</sub>N is more probable since it features the same type of structure (wurtzite) as GaN. The strain caused by the lattice mismatch is supposed to relax by generating misfit dislocations, as was shown in Ref. 13.

During cooling down (t=500 s) from 1073 K to RT the reflex shifts consistently toward higher angular values as represented by arrows. At RT the maximum of the peak ends up at about 34.9° and can be assigned to  $\varepsilon$ -Fe<sub>3</sub>N(111). This is shown in a selected plot at RT in Fig. 1(b). The presence of  $\varepsilon$ -Fe<sub>3</sub>N correspond with the results in Refs. 13 and 14 How-

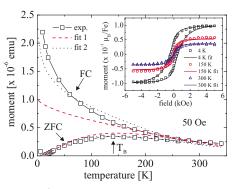


FIG. 2. (Color online) ZFC/FC measurement performed at 50 Oe on the sample implanted with  $3 \times 10^{16}$  cm<sup>-2</sup> and annealed at 1073 K for one cycle along with the fitting results using Preisach model. The inset shows magnetization curves recorded at 4, 150, and 300 K.

ever the change of symmetry cannot be explained up to now. The mean size of clusters, calculated with Scherrer formula using the full width at half maximum, is about 5 nm. However, the values calculated by means of Scherrer formula should be treated carefully. Because of the weak scattering intensity of the only secondary phase reflex no statement about the microstrain or mosaicity can be made.

The following annealing sequence, starting now from the already formed  $\varepsilon$ -Fe<sub>3</sub>N(111) phase lead to a peak shift from 34.9° at RT to 33.3° at 1073 K; cooling down to RT reverses the phase transformation again. Hence, the process of phase transformation from a partially disordered  $\varepsilon$ -Fe<sub>3-x</sub>N to an ordered  $\varepsilon$ -Fe<sub>3</sub>N is reproducible.

The process of secondary phase formation is suggested as follows. After the ion implantation a highly defective GaN with broken Ga–N bonds is present. During annealing in low pressure N<sub>2</sub> atmosphere atomic nitrogen can escape from the surface region leading to a further dissolution of GaN. Considering the energies of formation if atomic nitrogen is available the formation of iron nitrides rather than of GaN is preferable. Once  $\varepsilon$ -Fe<sub>3-x</sub>N is formed the transformation between the  $\varepsilon$ -Fe<sub>3-x</sub>N and  $\varepsilon$ -Fe<sub>3</sub>N phases occur over the N exchange and lattice site change. In contrast to that, the annealing in a high pressure N<sub>2</sub> atmosphere (at 1.1 bar) prevents the out-diffusion of nitrogen out of the damaged region, thus reducing the dissolution of GaN. In this case bcc-Fe is formed predominantly.

At this point it should be mentioned, that the reversibility is limited by the out-diffusion of Fe and gradual oxidation of Fe and Ga. During annealing the reflex intensities of the secondary phases decreases and after some repetitions reflexions from nitrides disappeared completely. This can be seen in Fig. 1(d), that shows two coupled  $2\theta$ - $\omega$  line scans recorded with the scintillation counter at RT after first annealing cycle and at 1073 K during the third annealing cycle. The shift of Al<sub>2</sub>O<sub>3</sub> is due to its thermal expansion and is consistent with Ref. 17 this confirms proper temperature control. The additional small reflexes in the 1073 K spectrum are related to the  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> reflexes. The full set of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> reflexes was found in this sample (not shown), referring to the polycrystallinity of the oxide layer.<sup>18</sup>

Magnetic measurements were performed on the samples in virgin and as implanted state, then after annealing at 1073 K for one cycle and after three cycles annealing accordingly. Virgin as well as implanted samples show no ferromagnetic response. The sample annealed for one cycle show a characteristic behavior of superparamagnetic nanoparticles. Figure 2 shows a zero-field-cooled/field-cooled (ZFC/FC) 232506-3 Talut et al.

measurement along with the magnetization curves (inset) of this sample recorded at 4, 150, and 300 K. The broad maximum in the ZFC part of the curve gives a hint of a large size distribution of the nanoparticles with the mean blocking temperature  $T_B$  of about 130 K. Using a formalism for the relaxation time  $\tau$  of the particle magnetization with the anisotropy energy density  $E_A$  (2×10<sup>4</sup> J/m<sup>3</sup>),<sup>19</sup> the attempt frequency  $f_0$  (typically 10<sup>9</sup>-10<sup>11</sup> Hz), measurement time 100 s, Boltzmann constant  $k_B$  the volume V of the mean sized cluster can be calculated. From that a diameter of about 16 nm is estimated, assuming a spherical shape of clusters

$$\tau^{-1} = f_0 \cdot \exp\left(-\frac{E_A V}{k_B T_B}\right). \tag{1}$$

However, the blocking temperature may be influenced by magnetic interactions between the particles.<sup>6</sup> This can lead to an overestimation of cluster size. A Preisach model was applied to fit the ZFC/FC as well as the magnetization loops.<sup>20,21</sup> The model assumes the decomposition of magnetic system into an ensemble of bistable units characterized by a temperature dependent spontaneous moment  $\mu$ , two possible magnetic states  $\pm \mu$ , and two characteristic values of coercive  $h_c$  and asymmetry  $h_i$  fields. Best fitting results were obtained by using Lorentz type functions for the coercive and asymmetry field distributions. The average spontaneous moment of the clusters at 0 K  $\mu_0$ , mean coercive field  $h_{c0}$ , power law temperature dependence parameter  $\sigma_c$  and  $\sigma_i$  as well as the critical temperature  $T_c$  were best fitted using  $\mu_0 = 7.7 \times 10^{-16}$  emu ( $\cong 8.3 \times 10^4 \ \mu_B$ ),  $h_{c0} = 150$  Oe,  $\sigma_c$ =800 Oe,  $\sigma_i$ =900 Oe, and  $T_c$ =480 K, respectively (fit 1). With 1.9  $\mu_{B}$ /Fe for the magnetic moment of Fe in  $\varepsilon$ -Fe<sub>3</sub>N (Ref. 22) and 3 Fe atoms in a unit cell the number of unit cells and out of it the cluster volume can be calculated. Again, assuming the spherical shape of the cluster its diameter can be calculated to about 9 nm. This model describes the ZFC curve as well as the hysteresis loops very precise. An indication of superparamagnetic behavior of clusters is the breakdown of the coercive field with increasing temperatures (see inset in Fig. 2). It has to be noted that the moment per Fe atom in  $\mu_B$  is by a factor 20 lower than of pure  $\varepsilon$ -Fe<sub>3</sub>N.<sup>16</sup> This means that not all Fe are incorporated in the formation of  $\varepsilon$ -Fe<sub>3</sub>N. Unfortunately, the mean size cluster approach is not able to describe the enhanced magnetization in the FC curve at low temperatures, related to the response from those magnetic clusters which are larger than 9 nm. Using two mean moments  $\mu_0 = 7 \times 10^{-16}$  and 14  $\times 10^{-16}$  emu ( $\cong$  mean sizes 9 and 11 nm) and different cluster concentrations the fit approaches the experimental curve (fit 2). This bimodal size distribution hint that clusters are in a coalescent regime, since the volume of a 11 nm sized cluster is close to the volume of two merged 9 nm sized clusters.

After annealing for three cycles at 1073 K no ferromagnetic response was detected. Because of the oxidation of Ga as well as of Fe the layer can be regarded as  $Ga_2O_3$  with inclusions of some iron oxide. This result is in contrast to the results from Lee *et al.*,<sup>23</sup> where Fe doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> was found to be ferromagnetic.

In summary, reversible phase transition of Fe implanted GaN was observed upon annealing in 0.5 bar  $N_2$  at 1073 K by means of *in situ* x-ray diffraction and discussed in terms of N exchange. After annealing  $\varepsilon$ -Fe<sub>3</sub>N was found to be formed predominantly and is responsible for ferromagnetic

response. It demonstrates that by variation of annealing conditions specific secondary phases can be created, leading to different electronic, magnetic and other properties. Preisach model yields an reasonable estimation of magnetic quantities and cluster size. The cluster size is in a good agreement with the calculation using Scherrer formula. No DMS related phenomena have been observed.

The annealing process is accompanied by a strong diffusion of N and dissolution of GaN, mediated by the formation of nitrides and oxides. Oxygen contamination further promotes the dissolution of GaN and the formation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. Hence, it acts as a further limiting aspect and need to be excluded in the following experiments. Fe doped  $\beta$ -Ga<sub>2</sub>O<sub>3</sub> was found to be paramagnetic which is in contrast with the findings in Ref. 12.

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# Millisecond flash lamp annealing of shallow implanted layers in Ge

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Shallow  $n^+$  layers in Ge are formed by phosphorus implantation and subsequent millisecond flash lamp annealing. Present investigations are focused on the dependence of P redistribution, diffusion and electrical activation on heat input into the sample and flash duration. In contrast to conventional annealing procedures an activation up to  $6.5 \times 10^{19}$  cm<sup>-3</sup> is achieved without any dopant redistribution and noticeable diffusion. Present results suggest that independently of pretreatment the maximum activation should be obtained at a flash energy that corresponds to the onset of P diffusion. The deactivation of P is explained qualitatively by mass action analysis which takes into account the formation of phosphorus-vacancy clusters. © 2009 American Institute of Physics. [doi:10.1063/1.3276770]

Previous investigations on the formation of shallow junctions in Ge by ion beam processing have shown that  $p^+$ doping using boron yields junctions that meet the requirements for the 22 nm technology node, whereas the formation of  $n^+$  junctions by P or As is complicated by the high diffusivity and the low activation of these dopants.<sup>1</sup> The present work deals with millisecond flash lamp annealing (FLA) of shallow layers implanted by P. Dopant redistribution, diffusion, and activation are investigated. The dependence of these effects on flash energy and flash duration is discussed. Furthermore, the influence of pre-amorphization implantation (PAI) and preannealing as well as the solid phase epitaxial recrystallization (SPER) of amorphous layers are studied.

(100) Ge wafers (p-type) with a resistivity between 0.2 and 0.5  $\Omega$  cm were used. In order to prevent surface degradation by implantation and outdiffusion during annealing the wafers were capped with 10 nm  $SiO_2$  by sputter deposition. Selected samples were preamorphized by Ge implantation at an energy of 200 keV and a fluence of  $10^{15}$  cm<sup>-2</sup>. All samples were implanted by P at 30 keV and  $3 \times 10^{15}$  cm<sup>-2</sup>. The ion beam was tilted by 7° with respect to the normal of the sample surface. Some samples were first preannealed in N<sub>2</sub> ambient at 400 °C for 240 s. Flash-lamp annealing of all samples was carried out in Ar ambient using flash durations of 3 and 20 ms and different electrical energies required to produce the flash. The electrical energy is determined by the capacitors used in the flash lamp facility and the applied voltage. Details of the FLA equipment were elucidated elsewhere.<sup>2</sup> In a limited range the temperature of the sample is assumed to be proportional to the electrical energy of the flash  $E_{\rm FLA}$  and, therefore, can be estimated by

 $T = cE_{FLA} + T_0$ . The parameters c and  $T_0$  are determined by complete SPER (Refs. 3 and 4) at about 600 °C and by melting of the sample surface at about 937  $^\circ C$  (3 ms flash: c=5.81 kJ<sup>-1</sup> K,  $T_0=373$  °C; 20 ms flash: c=2.31 kJ<sup>-1</sup> K,  $T_0$ =401 °C). In the following the flash energy  $E_{\rm FLA}$  is always used as a measure for the heat transfer to the sample.

Analysis by channeling Rutherford backscattering spectrometry (RBS/C) and cross-sectional transmission electron microscopy (XTEM) showed that the thickness of the amorphous layer formed by the PAI is about 178 nm, whereas the P implantation produces an amorphous layer of about 78 nm thickness. Furthermore, RBS/C and XTEM revealed that preannealing leads to complete SPER of the amorphous layers. If preannealing is not applied, samples recrystallize during FLA. The only exception is that treatment of preamorphized samples by 3 ms flashes leads to the formation of a polycrystalline layer by spontaneous nucleation and recrystallization.<sup>4,5</sup> In the recrystallized samples end-ofrange defects cannot be found by XTEM. This is in contrast to silicon where such defects are always observed after SPER.

Depth profiles of P before and after annealing are shown in Fig. 1. The data were obtained by secondary ion mass spectrometry (SIMS). The difference between the asimplanted profiles in single-crystalline and preamorphized Ge is due to suppressed channelling in the latter case. Preannealing at 400 °C for 240 s leads to fast phosphorus redistribution by the snow plough effect [Figs. 1(a) and 1(b)]. A certain amount of P is incorporated into crystalline Ge, and the surplus is pushed ahead from the moving amorphouscrystalline interface. This leads to P loss of about 25% and the level of incorporation is about  $3 \times 10^{20}$  cm<sup>-3</sup>. This value can be obtained from the plateau in the profiles shown in Figs. 1(a) and 1(b). The level of P incorporation found during SPER at 400 °C is higher than the maximum equilibrium solubility known from literature  $(2 \times 10^{20} \text{ cm}^{-3}, \text{ cf. Refs. 1}, 6, \text{ and } 7)$  and is therefore called metastable solubility.<sup>8,9</sup> On the other hand, SPER during FLA [Figs. 1(c) and 1(d)] does not show any significant snow plough effect. Obviously,

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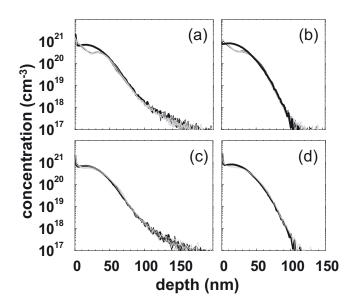


FIG. 1. Depth profiles of P in samples prepared without [(a) and (c)] and with [(b) and (d)] PAI as well as with [(a) and (b)] and without [(c) and (d)] preannealing. The as-implanted profiles are shown by thick black lines. Thin black and gray lines show P depth distributions after flash lamp annealing (FLA) for 3 and 20 ms, respectively. In the cases of 3 and 20 ms flashes the flash energy varied between 39 and 80 kJ as well as between 86 and 139 kJ, respectively.

nearly all phosphorus atoms are incorporated into crystalline Ge. This may be due to the fact that the temperature during SPER at millisecond FLA is higher than during preannealing, leading to a metastable solubility of at least  $5-7 \times 10^{20}$  cm<sup>-3</sup>. The temperature dependence of the metastable solubility can be explained by the competition between the mobility of the impurity in the amorphous phase and the velocity of SPER (cf. Refs. 9 and 10, and references therein). The higher the temperature, the higher the speed of the amorphous-crystalline interface, the less likely the impurity can stay in front of it and, therefore, incorporation into the crystalline phase prevails.

Figures 1(a)-1(d) clearly demonstrate that no P diffusion occurs if the flash energy does not exceed 80 and 139 kJ for 3 and 20 ms flash duration, respectively. This is in striking contrast to conventional annealing with durations of seconds to hours. Significant concentration-dependent P diffusion is already observed in the case of annealing at 500 °C for 60 s.<sup>8,11</sup> However, some diffusion is also found at higher flash energies of 88 kJ (3 ms) and 183 kJ (20 ms) as shown in Fig. 2(a). In this case the temperature of the sample is so high that diffusion can even occur during some milliseconds. If the flash energy is further increased the near surface region of the sample melts and a huge diffusion of phosphorus is found [Fig. 2(b)].

The variable probe spacing technique<sup>12</sup> and micro-fourpoint-probe measurements<sup>13</sup> were applied to determine the sheet resistance of the samples.<sup>14</sup> Figures 3(a) and 3(c) depict the sheet resistance of the samples annealed by FLA for 3 and 20 ms, respectively. The flash energy  $E_{FLA}$  is given on the abscissa. In Fig. 3 data points obtained by the same preparation method are marked by the same symbols. These points are connected by straight lines if no P diffusion is observed [cf. Figs. 1(a)–1(d)] whereas the isolated points indicate diffusion in the solid phase [cf. Fig. 2(a)]. Since it is not useful to compare sheet resistance data for active layers with different depth distributions of charge carriers, the elec-

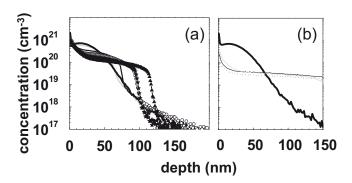


FIG. 2. P depth distributions obtained after different pretreatments and FLA at higher flash energies than in the case of Fig. 1. (a) Thin gray and black lines show the case of 3 ms FLA at 88 and 97 kJ, respectively, for samples without any pretreatment. The curves with lines and symbols were obtained for 20 ms FLA at 182 kJ (open circles – without any pretreatment, triangles-PAI, stars-PAI and preannealing). (b) Thin lines depict results obtained for 20 ms FLA at 232 kJ (black line – without any pretreatment, dotted line – PAI, gray line – PAI and preannealing).

trical properties are discussed in terms of the activation level of dopants. This quantity was roughly estimated using both the SIMS profile obtained after annealing and the corresponding value of sheet resistance.<sup>15</sup> The relation by Cuttriss<sup>16</sup> was used in order to transform resistivity to carrier concentration data. The resulting values for the activation are shown in Figs. 3(b) and 3(d). The figures demonstrate that below a certain threshold the activation level increases with increasing heat transfer to the sample, independently of the pretreatment. This effect is more pronounced for 3 ms flash duration. Below a certain flash energy preannealed samples show a lower activation than those without this treatment. If preannealing is used, samples that underwent PAI show a higher activation. In the case of 20 ms flash duration samples that did not undergo preannealing have a higher activation if they were preamorphized. The similar case for 3 ms duration was not investigated since a polycrystalline layer was formed.

The highest activation level of about  $6.5 \times 10^{19}$  cm<sup>-3</sup> is achieved for the 3 ms flash and 97 kJ electrical energy. This corresponds to a case where slight P diffusion is observed

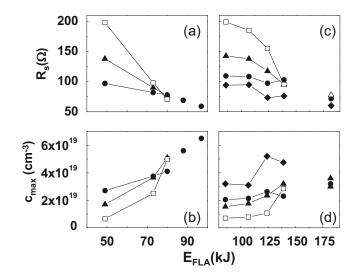


FIG. 3. Sheet resistance  $R_s$  and maximum carrier concentration  $c_{max}$  in the case of 3 ms [(a) and (b)] and 20 ms [(c) and (d)] FLA, for different flash energies and pretreatments. Circles: without any pretreatment, open squares: preannealing, diamonds: PAI, triangles: PAI and preannealing.

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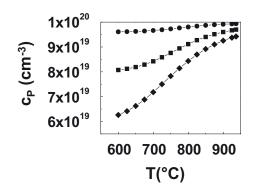


FIG. 4. Result of mass action analysis taking into account the formation of  $P_nV$  clusters (n=1...4): concentration of unbound or active P vs temperature. Different values are assumed for the total vacancy concentration (circles:  $10^{18}$  cm<sup>-3</sup> squares:  $5 \times 10^{18}$  cm<sup>-3</sup>, diamonds:  $10^{19}$  cm<sup>-3</sup>), and the total concentration of P is  $10^{20}$  cm<sup>-3</sup>.

[cf. Fig. 2(a)]. However, the comparison with results for the 20 ms flash shows that the activation level does not increase significantly or even decreases if P diffusion becomes stronger [cf. Figs. 3(d) and 2(a)]. Therefore, the optimum should be found at that electrical energy of the flash that corresponds to the onset of P diffusion. Taking into account the data depicted in Figs. 3(b) and 3(d) it may be concluded that this optimum should not strongly depend on the pretreatment. However, the highest activation level of P obtained in this work is far below the metastable and the equilibrium solubility. The fact that solubility returns to the equilibrium value during annealing after the completion of SPER or in subsequent annealing steps was also reported for dopants in Si. The reduction of solubility leads to less dopants on substitutional sites and to a concentration of activated dopants below or equal to equilibrium solubility. The decrease of solubility and activation may be accompanied by the forma-tion of dopant-defect clusters.<sup>9,17–19</sup> However, in the considered samples such clusters were not found by XTEM. Therefore, it is assumed that deactivation is due to the formation of P-vacancy (PV) acceptor pairs<sup>1</sup> or other tiny clusters containing vacancies and dopant atoms<sup>20</sup> which are hardly detectable by standard XTEM. Indeed calculations using density functional theory and mass action analysis showed that the formation of  $P_nV$  clusters  $(n=1,\ldots,4)$  may be responsible for the deactivation of P.<sup>21</sup> Using the data from Ref. 21 a similar mass action analysis was performed. The temperature dependence of the concentration of unbound P is depicted in Fig. 4. In order to demonstrate qualitatively the effect of deactivation a total P concentration  $(10^{20} \text{ cm}^{-3})$  below equilibrium solid solubility is assumed and different values for the total concentration of vacancies were considered. These values are much higher than the equilibrium vacancy concentration due to defect formation by ion implantation. Figure 4 can be used to interpret the dependence of the P activation on flash energy and pretreatment: At low temperatures (or flash energies) and/or at high vacancy concentration many  $P_nV$  clusters are formed and, therefore, a relatively low level of unbound or electrically active P is found. At higher temperature and/or lower concentration of vacancies the concentration of active P increases. It should be emphasized that present mass action analysis considers equilibrium states as well as a homogeneous distribution of P and vacancies. Kinetics could prevent the ability of the system to reach such states so that the above interpretation should be used with some caution.

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### The use of nanocavities for the fabrication of ultrathin buried oxide layers

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A continuous buried oxide layer with a thickness of only 58 nm is formed in silicon by oxygen implantation at 185 keV with a very low ion fluence of  $1 \times 10^{17}$  cm<sup>-2</sup> and subsequent He implantation. Due to the implanted He efficient oxygen gettering occurs at the implantation induced damage and results in the accumulation of the implanted oxygen as well as of oxygen indiffused from the annealing atmosphere. The morphology and the resistivity of the resulting silicon-on-insulator structure are analyzed by cross section transmission electron microscopy and by cross section scanning spreading resistance microscopy. © 2009 American Institute of Physics. [DOI: 10.1063/1.3065478]

Point defects in silicon induced by ion implantation have been widely investigated with regard to doping in silicon device processing.<sup>1,2</sup> The forward momentum transfer from the implanted ions to the silicon atoms in the collision cascades results in the presence of two distinct layers of vacancy excess close to the surface and interstitial excess at larger depth.<sup>3–6</sup> These implantation induced excess defects may act as gettering centers for, e.g., impurities.<sup>7,8</sup> Excess vacancy defects induced by oxygen implantation also serve as trapping centers for the implanted atoms by acting as nucleation sites for SiO<sub>2</sub> precipitation. The empty volume generated in the vacancy excess region assists the growth of the precipitates by balancing their volume expansion (by about 120%).<sup>9,10</sup> This mechanism is employed in this study for ion beam synthesis of a buried oxide (BOX) layer in Si, which is commonly known as the separation-by-implanted-oxygen (SIMOX) process. In SIMOX, silicon-on-insulator (SOI) substrates are fabricated by high-fluence oxygen implantation at elevated temperature and subsequent high temperature annealing.<sup>11</sup> Most of the oxide precipitates formed during SIMOX processing are distributed around the mean projected ion range  $(R_p)$  where the oxygen concentration reaches its maximum. However, many large SiO<sub>2</sub> precipitates are also formed at a shallower depth position around the damage maximum  $(D_p)$  of about  $0.8R_n$ , where the change is from the vacancy rich region to the interstitial rich one.<sup>12-14</sup> This is a problem especially for the low-fluence SIMOX processing (at fluences well below the stoichiometric limit) as a discontinuous oxide layer forms consisting of isolated precipitates. In order to create a homogeneous continuous BOX layer, the accumulation of the implanted oxygen in only one narrow layer is essential.<sup>9</sup> This shrinking of the BOX layer thickness in the SOI structure is required in order to enable downscaling of the SOI complementary metal-oxidesemiconductor architecture at suppressed short channel effect<sup>15</sup> and in order to improve the thermal conductivity.<sup>16</sup>

Also implanted He is known to be trapped mainly by vacancy defects to form a He-filled bubble layer at the posi-

tion of the vacancy dominated layer.<sup>17–20</sup> Such a layer of He bubbles or of empty cavities (after He outdiffusing during high temperature annealing<sup>17</sup>) is efficient for gettering of implanted oxygen into a narrow layer<sup>21</sup> and also for gettering of oxygen indiffused from the annealing atmosphere.<sup>22</sup> In our study, a subsequent helium implantation is employed after the oxygen implantation to stabilize the excess vacancy defects formed during oxygen implantation by He-induced bubbles and to better define the depth of oxide seeding.

Czochralski-Si (100) wafers were implanted at 550 °C with 185 keV O<sup>+</sup> ions to a fluence of  $1 \times 10^{17}$  cm<sup>-2</sup>. For this fluence, the maximum concentration of the as-implanted oxygen profile is only 10% of the oxygen concentration in stoichiometric SiO<sub>2</sub> ( $4.4 \times 10^{22}$  cm<sup>-3</sup>). Subsequently 45 keV He<sup>+</sup> ions were implanted at room temperature to fluences of  $4 \times 10^{16}$ ,  $8 \times 10^{16}$ , and  $2 \times 10^{17}$  cm<sup>-2</sup>. High temperature annealing was preformed at 1300 or 1350 °C for 3 h in an atmosphere containing Ar and  $O_2$  at the ratio Ar/ $O_2$ = 100/3 or 100/30. In addition, a reference sample was fabricated by only oxygen implantation and subsequent annealing. The oxygen depth distribution was analyzed by means of secondary ion mass spectrometry (SIMS) using a Cameca IMS 7f instrument. The morphology and the resistance of the SOI structure were investigated by cross section transmission electron microscopy (XTEM) and cross section scanning spreading resistance microscopy (SSRM), respectively. For the cross section SSRM, the sample was cut manually and measured using a multimode atomic force microscopy (from Veeco instruments) equipped with a conductive Si tip coated with diamond.

Figure 1(a) presents the oxygen profiles obtained after the above processing. After annealing at 1300 °C, the oxygen profile of the reference sample shows only one peak located at the depth of 460 nm, which is close to  $R_p$  at 440 nm as calculated by the TRIM code.<sup>23</sup> For a low- and medium-fluence postimplantation of He, there are additional front peaks close to the depth of the calculated  $D_p$  position. The broadened  $R_p$  peak at lower He fluence may result from oxygen gettering at a certain amount of He, which is trapped by interstitial defects induced by the oxygen implantation,<sup>20</sup> which therefore cause the oxygen gettering there. As the He

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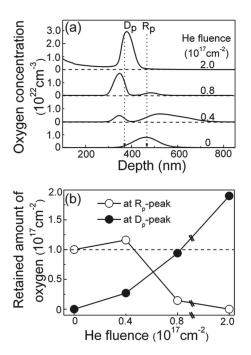


FIG. 1. (a) SIMS profiles of the oxygen distributions after postimplantation with He at different fluences and annealing at 1300 °C in 3% O<sub>2</sub> atmosphere for 3 h. The calculated positions of  $R_p$  and  $D_p$  are indicated. (b) Retained amount of the oxygen accumulated in the  $R_p$  peak and the  $D_p$  peak of the oxygen profiles vs He postimplantation fluence. The fluence of implanted oxygen is indicated by the dashed line.

fluence increases, more excess vacancies are generated in the vacancy rich region around  $D_p$  by the He implantation, in addition to those generated by the oxygen implantation. At the highest He fluence, all the oxygen is accumulated in the front peak at a depth of 370 nm. Figure 1(b) shows the retained amount of oxygen calculated by integrating the corresponding peaks of the profiles. The oxygen accumulation at the  $R_p$  region vanishes toward high He fluence, whereas the amount found around  $D_p$  even increases significantly above the implanted fluence. This indicates additional incorporation of oxygen by indiffusion from the annealing atmosphere. The corresponding XTEM image for the highest He fluence [Fig. 2(a)] shows a discontinuous BOX layer with a thickness of

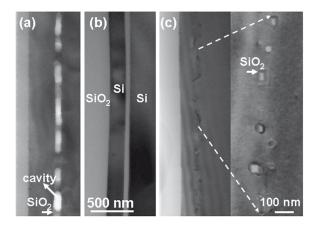


FIG. 2. Comparison of XTEM micrographs showing SOI structures formed by implantation of 185 keV ions to  $1 \times 10^{17}$  O cm<sup>-2</sup>. (a) After 45 keV He postimplantation at  $2 \times 10^{17}$  cm<sup>-2</sup> and annealing at 1300 °C in an atmosphere containing 3% O<sub>2</sub>. The bright spots indicate cavities in the BOX layer. (b) Same, but after annealing at 1350 °C under 30% O<sub>2</sub>, resulting in a continuous narrow BOX layer. (c) Reference sample annealed at 1350 °C in 30% O<sub>2</sub> showing widely distributed SiO<sub>2</sub> precipitates.

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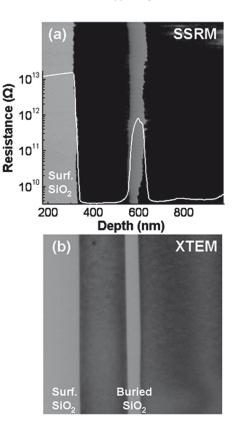


FIG. 3. (a) Cross section SSRM image of silicon implanted with 185 keV ions to  $1 \times 10^{17}$  O cm<sup>-2</sup> plus 45 keV He ions to  $2 \times 10^{17}$  cm<sup>-2</sup> and annealed at 1350 °C in 30% O<sub>2</sub>. The dark and light colors indicate low and high resistance, respectively. A laterally averaged resistance scan curve is inserted. The scanning size is  $0.8 \times 0.8 \ \mu m^2$ . (b) Corresponding XTEM image with the same imaging area.

80 nm containing extended cavities (bright spots), which are similar to those recently reported by Ogura.<sup>22</sup> A continuous BOX layer [Fig. 2(b)] with a thickness of only 58 nm was achieved by enhanced oxygen indiffusion during annealing at increased temperature (1350 °C) and oxygen concentration (30%). Here, the total amount of retained oxygen of  $2.6 \times 10^{17}$  cm<sup>-2</sup>, which is derived from the thickness of the BOX layer, exceeds the initially implanted fluence of  $1 \times 10^{17} \ \text{cm}^{-2}$  even more. Only few and disperse cavities remain in the BOX layer, and few dislocations in the top Si layer are observed by XTEM. The thickness of the BOX layer is not perfectly uniform, being slight larger at the positions of the remaining cavities. In comparison, a layer of isolated SiO<sub>2</sub> precipitates distributed over a thickness of about 110 nm is formed in the reference sample [see Fig. 2(c)]. The cross sectional SSRM analysis in Fig. 3 reveals the thermally formed surface oxide and confirms that the continuous BOX layer formed at the highest He postimplantion fluence [see Fig. 2(b)] is fully insulating. Any SiO<sub>2</sub> precipitates that would be larger than the tip diameter<sup>24</sup> cannot be seen in the top Si layer. The position in depth (at 400 nm) and the thickness (about 58 nm) of the BOX layer measured by SSRM are in agreement with the XTEM result shown in Fig. 2(b). Figure 3(a) demonstrates that the upper  $Si/SiO_2$ interface of the BOX layer is as smooth as the SiO<sub>2</sub>/Si interface obtained by thermal surface oxidation. The inserted curve shows the resistance laterally averaged over the scanned area. The top Si layer has the same resistance as the Si substrate, whereas the measured resistance of the BOX

layer is one order of magnitude lower than the surface oxide. This may be attributed to defects such as Si rich precipitates embedded in the BOX, which is usually obtained in the SIMOX process.<sup>25</sup> Together with this, the apparent less uniform BOX layer shown in the SSRM image may be attributed to artifacts of the SSRM measurement, such as electrical defects or partial oxidation of the cleaved surface.

The enhancement in oxygen accumulation around  $D_p$  caused by subsequently implanted He can be explained by vacancy clusters originally generated by the oxygen implantation, which form stable complexes<sup>6</sup> that are precursors of nanosized He-filled bubbles.<sup>18–20</sup> The empty volume and the internal surfaces associated with such bubbles act as the preferential trapping centers for He and oxygen, respectively (the latter is accumulated during the annealing process).<sup>17,19,21,22</sup> He escapes from bubbles leaving the Si substrate at the temperature above 300 °C.<sup>26</sup> An increase in the He fluence may result in a higher pressure inside the bubbles, which en-

hances their stability against the dissolution that may occur during the temperature ramping for high temperature annealing. A longer lasting oxygen gettering effect is achieved due to the stabilization of the gettering centers. By the conventional low-fluence SIMOX process for implantation energy of 185 keV, a continuous BOX layer was never achieved at such a low oxygen fluence and small thickness. BOX layers with a thickness of 100–125 nm were formed with oxygen fluences of  $(4-5) \times 10^{17}$  cm<sup>-2</sup>.<sup>25,27</sup> Nevertheless, the BOX thickness can also be decreased by reducing the ion energy according to the so-called "energy-dose" window,<sup>9,28,29</sup> which, however, would decrease the thickness of the superficial Si layer.

To summarize, oxygen gettering at the damage induced by the implantation of a very low implanted oxygen fluence of  $1 \times 10^{17}$  cm<sup>-2</sup> at 185 keV is enhanced by using a subsequent He implantation and employing additional oxygen indiffusion from the annealing atmosphere, which benefits the SOI structure formation at  $D_p$ . In this way, an extremely thin continuous BOX layer can be formed, which is demonstrated by cross section SSRM together with XTEM.

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# Light emitting field effect transistor with two self-aligned Si nanocrystal layers

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Light emitting field effect transistors based on narrow layers of silicon nanocrystals (NCs) in the gate oxide were fabricated. Direct quantum mechanical electron and hole tunneling into NCs was achieved by self-alignment of NCs-interface-distances to  $\sim 2$  nm. The direct tunneling reduces oxide degradation, prolongs device lifetime and increases operation speed. Self-alignment occurs during thermal treatment of ion irradiated stacks of 50 nm polycrystalline silicon/15 nm SiO<sub>2</sub>/(001)Si substrate. An alternating voltage (ac) was applied to the gate to inject charges into the NCs. Due to injection by direct tunneling, electroluminescence extends to higher ac frequencies than reported so far. © 2009 American Institute of Physics. [doi:10.1063/1.3242379]

Ion beam synthesis (IBS) of nanocrystals (NCs) is compatible with modern complementary metal oxide semiconductor (CMOS) technology as it is a combination of ion implantation and subsequent annealing. Great effort is currently devoted to the IBS of semiconducting or metallic NCs for micro- and optoelectronic applications, respectively. An example is the nonvolatile multinanodot floating-gate memory.<sup>1-4</sup> Recently, Walters *et al.*<sup>5</sup> demonstrated a field effect driven electroluminescence (EL) by excitonic emission from Si NCs which are embedded in a gate oxide. An alternating current (ac) voltage is applied at the gate electrode of a MOS transistor device in order to charge the Si NCs sequentially with electrons and holes. Thereby, both charge carriers are injected from the transistor channel via Fowler-Nordheim (FN) tunneling through the oxide barrier. The excitons formed in the Si NCs recombine radiatively; the emitted wavelength depends on the NC bandgap energy, i.e., the NC size.<sup>6</sup>

Here, using an innovative fabrication process, a light emitting field effect transistor (LEFET) is demonstrated with two self-aligned luminescent layers of Si NCs. Compared to conventional Si NC synthesis by Si<sup>+</sup> ion implantation into the gate oxide (see Refs. 1 and 5, for instance), we take advantage of a self-alignment process, i.e., the Si NCs are formed in SiO<sub>2</sub> at a well-controlled small distance of  $\sim 2$  nm from the Si/SiO<sub>2</sub> interfaces. This allows charge carrier injection into the NCs by direct tunneling, which leads to lower operation voltage and longer device life time. Additionally, excitonic light emission comes from two Si NC layers at the gate poly-Si/SiO<sub>2</sub> and the channel Si/SiO<sub>2</sub> interfaces. Figures 1(a)-1(c) illustrate the fabrication process. Ion irradiation through a MOS-like poly-Si/SiO<sub>2</sub>/Si substrate stack [Fig. 1(a)] causes an ion-irradiation-induced mixing of both  $SiO_2/Si$  interfaces. At the formerly sharp  $SiO_2/Si$  interfaces nonstoichiometric  $SiO_x$  (x < 2) regions are formed [Fig. 1(b)]. Subsequent annealing restores the upper and lower  $SiO_2/Si$  interfaces by phase separation (of  $SiO_r$  into  $SiO_2$ ) and Si) [Fig. 1(c)]. In the tails of the ion beam mixed profile NCs form due to slow diffusion to the distance interfaces.

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The competition between interface restoration and nucleation self-aligns the NCs into narrow layers parallel to the Si/SiO<sub>2</sub> interfaces. The competition between interface restoration and nucleation aligns the NCs into narrow layers parallel to the SiO<sub>2</sub>/Si interfaces.<sup>7</sup> In comparison to the device reported by Walters *et al.*,<sup>5</sup> here, the light emission efficiency is improved due the additional second Si NC layer charged with opposite polarity from the poly-Si gate [see Fig. 1(d)]. The self-alignment of the NC layers with the SiO<sub>2</sub>/Si interfaces allows to control shorter (direct) tunneling distances between the NCs and the Si electrodes with the potential of faster devices operating at reduced voltages. The charge storage

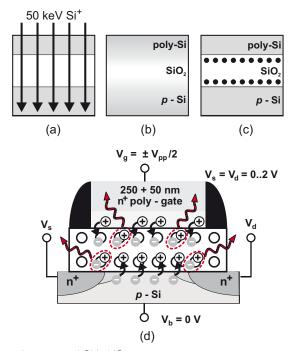


FIG. 1. (Color online) [(a)-(c)] Scheme of the fabrication of Si NCs in the gate oxide by ion irradiation through the Si/SiO<sub>2</sub> interfaces and subsequent annealing. (d) Schematic of light emission where an ac voltage is applied to the gate in order to inject charges of both polarities into the lower and upper Si NC layer from the channel and the poly-Si gate of the transistor, respectively.

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behavior of such a structure used as NCs based memory device is described elsewhere.  $^{10,11}\,$ 

In this article the performance of the LEFET devices as well as options of light emission with a NC double layer configuration are reported. A 14.5 nm thin SiO<sub>2</sub> layer was thermally grown at 900 °C for 50 min in dry O<sub>2</sub> on (001) *p*-type Si (1–10  $\Omega$  cm). A 50 nm thick poly-Si layer fabricated by low pressure chemical vapor deposition (LPCVD) covers this oxide. After 50 keV <sup>28</sup>Si<sup>+</sup> ion irradiation of the poly-Si/SiO<sub>2</sub>/p-Si layer stack to a fluence of 7  $\times 10^{15}$  cm<sup>-2</sup> at room temperature [Fig. 1(a)], a highly  $n^+$ -doped 250 nm thick LPCVD poly-Si capping layer was deposited on top to form the poly-Si gate of the transistor. Rapid thermal annealing (RTA) was carried out at temperatures between 1000 and 1150 °C in N2 for 10-160 s to prepare the Si NCs embedded in SiO<sub>2</sub> [see Fig. 1(c)]. The existence of the NCs was confirmed by EFTEM imaging using a FEI Tecnai 20 FEG microscope and a Gatan energy filter. Due to this protecting poly-Si layer the discussed fabrication approach profits by its insensitiveness to detrimental impacts.<sup>4,8</sup> The LEFETs were fabricated as nMOSFET devices with an active area of  $20 \times 20 \ \mu m^2$  (gate length  $\times$  gate width) using the standard 0.6  $\mu$ m CMOS technology line of the Zentrum Mikroelektronik Dresden company. Originally prepared to study the memory characteristics of the embedded Si NCs, several layers of in total 3.2  $\mu$ m thick  $Si_3N_4$  and  $SiO_2$  layers were deposited on top of the transistors. This device protection is usual in modern memory device fabrication but clearly not suitable for LEFET applications. Therefore, a second set of samples was prepared to study the photoluminescence (PL) properties of the embedded Si NCs, which was not possible on the integrated CMOS devices due to this multilayer coverage. For this sample series the preparation stopped after the 50 nm poly-Si layer deposition and subsequent ion irradiation. Here, the RTA treatment was carried out for 120 and 750 s and also for 3 h in a furnace, both at 1050 °C in N2. Excited by the secondharmonics, 532 nm, of a Nd:YAG laser the PL spectra of the Si NCs were obtained by a liquid nitrogen cooled CD camera. ac square-wave voltage signals were applied to the gate with the *p*-Si substrate as the reference electrode and an amplitude between  $\pm 7$  and  $\pm 11$  V (peak-to-peak voltage  $V_{pp}$  of 14 to 22 V) by a 20 MHz NF1930 arbitrary function generator. The source and drain voltages were set to  $V_s = V_d = 0$  V. The EL spectra were recorded using an Andor DU401A-UV-BR-DD CCD camera working at T=-70 °C (thermoelectrical cooling), a Shamrock 303i grating spectrometer, and an optical microscope.

TRIDYN ion collision cascade simulations reveal<sup>7,12</sup> that the 50 keV Si<sup>+</sup> ion irradiation of the LEFET layer stack causes an ion beam mixing of the poly-Si/SiO<sub>2</sub> and SiO<sub>2</sub>/(001)Si interfaces with a considerable Si excess in the gate oxide. As the projected ion range is located deeply inside the Si substrate, the majority of Si excess results from ion beam mixing of the interfaces. As shown in Fig. 2 during annealing well-aligned narrow layers of Si NCs with 2–3 nm in diameter form adjacent to the recovered interfaces. The layers are separated to the poly-Si gate and *p*-Si substrate, respectively, by an oxide of about 2 nm thickness, i.e., within a direct tunneling distance for electrons and holes. In the middle of the gate oxide an about 5 nm thin SiO<sub>2</sub> layer is found sandwiched between the NC arrays. Applying an ac

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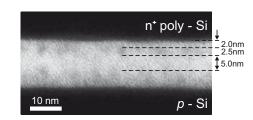


FIG. 2. Cross section EFTEM image of the poly-Si/SiO<sub>2</sub>/p-Si layer stack revealing tiny Si NCs of about 2–3 nm size on both sides of the gate oxide in the late stage of ripening after 1100 °C, 160 s annealing.

voltage to the gate electrode-following the model of Walters et al.<sup>5</sup>—excitons are formed within the Si NCs due to alternating electron and hole injection from both electrodes. Since the emitted wavelength depends on the NC bandgap energy, i.e., the NC size, the luminescence with a center wavelength of about 750 nm as shown in Fig. 3 is attributed to radiative recombination of excitons within Si NCs of 2-3 nm in diameter which is consistent with the TEM result (Fig. 2).<sup>5,6</sup> The EL spectra in Figs. 3(a) and 3(c)were obtained from integrated devices of  $20 \times 20 \ \mu m^2$  size. As confirmed by light transmission calculations (not shown here), the undulating modulations of the typically Gaussianshaped luminescence profiles are related to multiple internal reflections within the  $Si_3N_4/SiO_2$  multilayer stack covering the LEFET devices. These deviations are clearly not correlated with the NCs fabrication method since the PL spectra in Fig. 3(d), which are obtained at simple 50 nm poly-Si/14.5 nm SiO<sub>2</sub>/Si stacks, reveal a typical behavior of tiny Si NCs. With the electro- and PL results in Fig. 3 different stages of NC evolution can be traced as a function of the annealing temperature and/or time. During the annealing at 1000 °C the overall EL intensity is close to its maximum value and

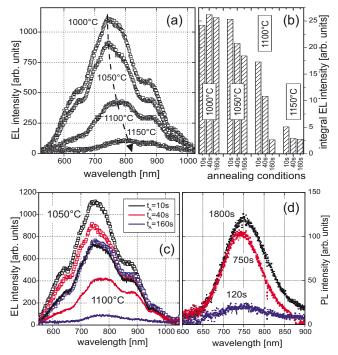
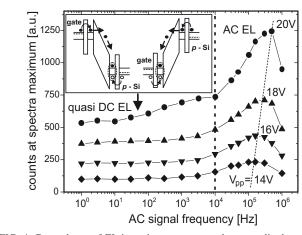


FIG. 3. (Color online) (a) EL spectra after 40 s annealing at different temperatures { $V_{pp}$ =22 V and f=10 kHz in [(a)–(c)]}. (b) Bar graph plot of the integral EL intensities according to the total sample set (annealing for 10, 40, and 160 s at each temperature). (c) EL spectra after isothermal annealing at 1050 °C (squares) and 1100 °C (lines). The different annealing times are indicated by different colors. (d) PL spectra obtained at simplified structures after 120, 750 (RTA) and 1800 s (furnace) annealing at 1050 °C.



20

AC EL

FIG. 4. Dependence of EL intensity on ac gate voltage amplitude and frequency for a LEFET annealed at 1050 °C for 40 s. A schematic band diagram illustrates the quasi DC mode EL mechanism in the inset (f  $\lesssim$  10 kHz) for negative and positive ac square-wave gate voltage periods.

still slightly increasing [see the bar graph plot of Fig. 3(b)]. With a higher thermal budget (1050 °C) the intensity is decreasing especially during the 1100 °C annealing down to a very low but stable level such as for 1150 °C thermal treatment. Whereas an increasing intensity indicates that the process of precipitation and Si NC formation has not been finished up to this state (1000 °C), the dissolution of NCs is beginning at 1050 °C; the evolution of the NC size follows Gibbs-Thomson's relation, i.e., a diffusion controlled ripening process.<sup>7,9</sup> NCs adjacent to the Si substrate and poly-Si gate dissolve the faster, the closer they are located to the Si/SiO<sub>2</sub> interfaces.<sup>7–9</sup> This accelerates the dissolution of small, close NCs (1100 °C) and stabilizes bigger more distant ones (related to the Si substrate and gate, respectively) like for 1100 °C, 160 s and 1150 °C annealings in Fig. 3(b). During Ostwald ripening the Si NCs compete with each other in plane whereas some Si NCs grow at the expense of smaller ones. This corresponds at the same time to (i) the redshift of the EL spectra in Fig. 3(a), i.e., a radiative exciton recombination in larger NCs (which are characterized by a smaller bandgap), and (ii) the quenched luminescence due to a considerably reduced number of light emitting sites. It has to be mentioned that the denoted LEFET annealing temperatures represent only a part of the total thermal budget necessary for complete CMOS device fabrication. This explains why the overall PL intensity in Fig. 3(d) is still increasing during isothermal annealing indicating that the process of Si formation has not been finished after 1050 °C, 750 s, while seemingly the EL intensity already decreases with 1050 °C, 40 s thermal treatment [Fig. 3(c)].

The LEFET has two luminescence options depending on the applied gate signal frequency (Fig. 4). With similar EL spectra we refer both modes to radiative recombination of excitons in the Si NCs. At low frequencies, where both NC layers are saturated with charges of opposite sign, a quasidirect current (dc) related EL is dominating (see the inset in Fig. 4). The applied gate voltage drops mainly across the remaining 5 nm thin SiO<sub>2</sub> layer sandwiched between the NC arrays. This enables a dc leakage current due to FN tunneling of electrons which recombine radiatively with stored holes at the NCs of the opposite side. The quasi-dc current can be easily suppressed increasing the gate oxide thickness to quench the tunneling probability (with positive consequences

for the device long time reliability). In Fig. 4 for frequencies  $f \gtrsim 10$  kHz an ac related EL emerges out of the quasi DC luminescence background. This type of EL-schematically illustrated in Fig. 1(d)—has been invented by Walters *et al.*<sup>2</sup> as a field effect driven mechanism. The luminescence intensity increases with increasing number of switching cycles up to 100-500 kHz and collapses at 1 MHz AC signal frequency. The highest EL intensity occurs at much higher AC frequencies than obtained by Walters et al.,<sup>5</sup> or predicted by Carreras *et al.*<sup>13</sup> ( $f \le 50$  kHz). Here, the gate capacitive charging time constant is clearly reduced due to our very small transistor. In addition the 2 nm thin direct tunneling oxide enables higher charging currents with shorter charging times (as confirmed by their memory device characteristics) $^{10,11}$  in comparison to FN tunneling as reported by Walters *et al.*<sup>5</sup> where the NCs are located closer to the oxide center. The dependence of the EL intensity maximum on the ac voltage amplitude reveals the significance of the charge transfer rate. The EL quenches at about f=1 MHz which corresponds supposedly to the physical limit of radiative recombination of excitons in tiny Si NCs, i.e., their radiative lifetime.<sup>5,13</sup>

In conclusion, very small  $(20 \times 20 \ \mu m^2)$  Si NC based light emitting nMOSFET devices are demonstrated where charge carriers are injected into NCs by direct tunneling, not by FN tunneling as in devices reported so far. The direct tunneling leads to a faster device operation which was proven by EL at one order of magnitude higher ac frequencies. Another advantage of direct tunneling is the lower operation voltage and the reduced oxide degradation which increases the device life time. The direct tunneling distances are achieved by a self-alignment process which yields an additional NC layer leading to a further increase of luminescence.

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# Enhanced blue-violet emission by inverse energy transfer to the Ge-related oxygen deficiency centers via Er<sup>3+</sup> ions in metal-oxide semiconductor structures

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It is generally believed that the 1.5  $\mu$ m Er luminescence is enhanced by transferring energy from Si nanocrystals to the nearest Er<sup>3+</sup> ions in Er-doped Si-rich SiO<sub>2</sub> layers during optical pumping. Here, the influence of Ge nanocrystals instead of excess Si in the same environment is studied using electroluminescence technique on metal-oxide-semiconductor structures. An increase of the 400 nm electroluminescence intensity with a concomitant reduction of the Er-related emission is observed. This is explained in the light of an inverse energy transfer process from Er<sup>3+</sup> to the Ge-related oxygen-deficiency centers. © 2009 American Institute of Physics. [DOI: 10.1063/1.3077169]

Recently, Si nanocrystals (NCs) in SiO<sub>2</sub> received much attention, since they can act as sensitizers for increasing the intensity of the 1.53  $\mu$ m Er luminescence in layers codoped with Er ions.<sup>1-4</sup> The natural question arises, whether and/or how this behavior will change by using NCs of Ge, which is chemically very similar to Si. The optoelectronic properties of Ge NCs in SiO<sub>2</sub> have been investigated<sup>5,6</sup> to a much lesser extent than that of Si. The underlying physics behind such light emitting devices (LEDs) has generally been expressed in terms of recombination of charge carriers either in Ge NCs through bandgap opening' according to the quantum confinement model<sup>5</sup> or in defect states<sup>6</sup> during electrical/optical pumping. Emission of light at  $\sim 400$  nm has been reported<sup>6</sup> as a result of optical and/or electrical excitation of the Gerelated oxygen-deficiency center (GeODC) into the first singlet state  $(S_1)$ , followed by the intersystem crossing to the first triplet state  $(T_1)$ , and a radiative transition back to the ground singlet state  $(S_0)$ . Recent photoluminescence studies<sup>8,9</sup> revealed that this system could be used for visible range pumping of Er<sup>3+</sup> ions where Ge nanoclusters behave as sensitizers. In fact, the application of such a system in Sibased electronic platform requires the fabrication of a metaloxide semiconductor (MOS) structure while the electroluminescence (EL) efficiency is crucial for device performance.

In this letter, we present the fabrication of Si-based MOSLEDs where Ge NCs and/or  $Er^{3+}$  ions are dispersed into the SiO<sub>2</sub> layer. In particular, we demonstrate a pronounced increase in the 400 nm EL intensity by Er-doping with a concomitant reduction in the Er emission, implying an *inverse energy transfer* from  $Er^{3+}$  to the GeODCs. This is contrary to the phenomenon commonly accepted for Er-doped Si-rich SiO<sub>2</sub> layers where energy is known to transfer from Si NC to the nearby  $Er^{3+}$  ions, <sup>1-4</sup> keeping in mind the spatial location of the Er sites.<sup>10</sup>

Initially 130 keV Ge ions were implanted at room temperature (RT) with a dose of  $2 \times 10^{16}$  ions/cm<sup>2</sup> into a thermally grown SiO<sub>2</sub> layer of thickness 200 nm on *n*-type (100)Si wafers. The samples were subjected to rapid thermal annealing at 1050 °C for 180 s according to Ref. 11 in nitrogen ambience to produce Ge NCs. Subsequently, 250 keV

Er ions were implanted with a dose of  $1 \times 10^{15}$  ions/cm<sup>2</sup> into the Ge-rich SiO<sub>2</sub> layer, followed by short-time annealing at 1050 °C for 6 s not only to remove ion-beam induced defects, but also to activate Er<sup>3+</sup> ions. Both Ge and Er ions provide Gaussian-like profiles with maximum concentrations of  $\sim 3.5\%$  and 0.3% at  $R_p$  of  $\sim 112$  and 115 nm, respectively. Two reference samples were also prepared by implanting either Ge or Er ions into the SiO<sub>2</sub> layers followed by annealing at 1050 °C for 180 and 6 s, respectively. A semitransparent indium tin oxide and aluminum contacts were sputter deposited in the front and rear surfaces, respectively, to achieve a MOS structure. Circular electrodes with a diameter of 0.2-1.1 mm were subsequently patterned in the front surface by photolithography. The EL spectra were recorded at RT with a single grating monochromator and a photomultiplier, a liquid-nitrogen cooled InGaAs detector, or a charge-coupled device detector. The InGaAs detector or the photomultiplier in combination with a photon counting system were employed to study the time-resolved EL dynamics. Crosssectional transmission electron microscopy (TEM) images were taken by means of a FEI Titan 80-300 S/TEM.

Formation of Er/Er-oxide clusters with dimension in the range of 2–5 nm (indicated by white arrows) [Fig. 1(a)] in Er-doped SiO<sub>2</sub> layers, called Er:SiO<sub>2</sub>, was seen using high-resolution TEM (HRTEM), while Ge NCs with an in-plane diameter of ~4 nm (marked by black arrows) were observed in Ge-rich SiO<sub>2</sub> layers, called Ge-NCs:SiO<sub>2</sub> [Fig. 1(b)]. Although the composition of the clusters in Er:SiO<sub>2</sub> is not yet known, on the basis of the thermodynamic properties of Er and its oxide,<sup>12</sup> we feel that the formation of Er oxide is more favorable than that of Er. However, no specific variation in size and distribution of NC was observed in Er-doped Ge-NCs:SiO<sub>2</sub> layers, named Er:Ge-NCs:SiO<sub>2</sub> [Fig. 1(c)]; the corresponding HRTEM image is displayed in Fig. 1(d).

Figure 2 shows the EL spectra of the Ge-rich MOSLEDs with or without Er ions for a constant current density (*J*) of 0.17 mA/cm<sup>2</sup>. The spectrum for the Er-doped MOSLED is characterized by four EL bands, namely,  $P_1$  to  $P_4$ , in the spectral range between blue violet and infrared, which are assigned as the radiative transitions in Er<sup>3+</sup> from the <sup>2</sup>H<sub>11/2</sub>, <sup>4</sup>S<sub>3/2</sub>, <sup>4</sup>F<sub>9/2</sub> and <sup>4</sup>I<sub>13/2</sub> states to the ground state (<sup>4</sup>I<sub>15/2</sub>), respectively.<sup>13</sup> Nevertheless, the Er emission in the short wavelength region, especially the <sup>2</sup>H<sub>9/2</sub>  $\rightarrow$  <sup>4</sup>I<sub>15/2</sub> transition in-

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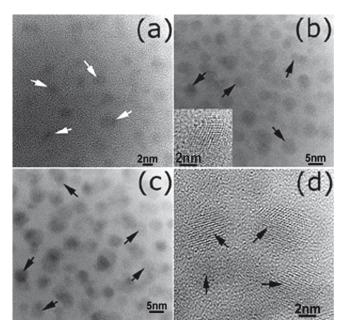


FIG. 1. Bright-field TEM images, with electron beam direction along the [110] zone axis for the (a)  $\text{Er}:SiO_2$ , (b)  $\text{Ge-NCs}:SiO_2$ , [(c) and (d)] and  $\text{Er}:\text{Ge-NCs}:SiO_2$  are shown. Inset of (b) displays the HRTEM of a Ge NC of the Ge-NCs:SiO<sub>2</sub>, where crystalline Ge core shows the {111} faceting. HRTEM of the  $\text{Er}:\text{Ge-NCs}:SiO_2$  (d) depicts randomly oriented Ge-NCs (denoted by black arrows).

duced ~410 nm EL (inset of Fig. 2) can be distinguished for  $J=7.1 \text{ mA/cm}^2$ . The EL peak of ~400 nm in Ge-NCs:SiO<sub>2</sub> is attributed to the GeODC (as a consequence of the  $T_1 \rightarrow S_0$  transition).<sup>6</sup> Our key result is the increase in the 400 nm EL intensity at the expense of Er-related emission upon Er doping. Quantitative measurements show that the 1532 nm EL yield of the Er<sup>3+</sup> is suppressed by a factor of ~3 in presence of Ge NCs. Whereas other Er EL bands are hardly distinguishable due to the broadening of the 400 nm peak and possible decrease in intensity of those Er emissions by a factor similar to the 1532 nm Er EL.

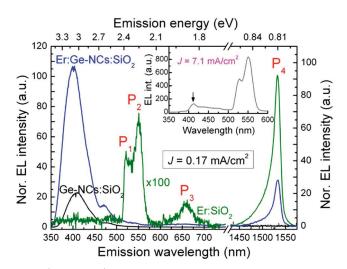


FIG. 2. (Color online) The visible and infrared EL spectra, plotted by solid lines in black, green, and blue for the Ge-NCs:SiO<sub>2</sub>, Er:SiO<sub>2</sub>, and Er:Ge-NCs:SiO<sub>2</sub>, respectively, with  $J=0.17 \text{ mA/cm}^2$ . Note that the scale given in the left and right ordinates are independent of each other as the signals in the visible and infrared regions were collected by two different detectors. 100 for better projection, where  $P_1$  to  $P_4$  represent radiative transitions from Inset shows an additional EL spectrum for Er:SiO<sub>2</sub> with  $J = 7.1 \text{ mA/cm}^2$ ; a prominent peak at ~410 nm is indicated by the downward arrow.

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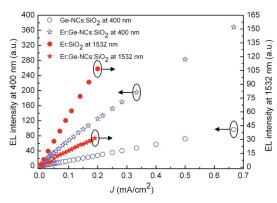


FIG. 3. (Color online) Relative intensities of the 400 nm peak for Ge-NCs:SiO<sub>2</sub> and Er:Ge-NCs:SiO<sub>2</sub> are denoted by blue open circles ( $\bigcirc$ ) and blue solid stars ( $\Rightarrow$ ), respectively. Similarly, the relative 1532 nm peak intensities for Er:SiO<sub>2</sub> and Er:Ge-NCs:SiO<sub>2</sub> are depicted by red solid circles ( $\bullet$ ) and red solid stars ( $\star$ ), respectively.

The current-voltage (*I-V*) characteristics of the working devices have also been tested (not shown), confirming that the device integrity is unaffected during device processing; the *I-V* profiles can be interpreted in the light of injection of electrons from the conduction band (CB) of the Si to the CB of SiO<sub>2</sub> via the Fowler–Nordheim or trap-assisted tunneling.<sup>14</sup> Furthermore, the 400 and 1532 nm EL intensities are found increasing linearly as a function of *J* (Fig. 3), where each of them can be fitted by EL(*J*) = EL<sub>max</sub>[ $\sigma \tau \phi / (\sigma \tau \phi + 1)$ ];<sup>15</sup>  $\phi = J/q$ . The product of the excitation cross section ( $\sigma$ ) and lifetime ( $\tau$ ) for the former peak increases from 2.3 × 10<sup>-20</sup> to 5.4 × 10<sup>-17</sup> cm<sup>2</sup> s by Er doping, while it varies from 6.5 × 10<sup>-17</sup> to 1.7 × 10<sup>-16</sup> cm<sup>2</sup> s for the later by incorporating Ge NCs.

It is assumed that during implantation Si and oxygen (O) atoms are released from the SiO<sub>2</sub> network. In the following phases of annealing, displaced O can either be reintegrated into the SiO<sub>2</sub> network or be used in oxidizing implanted element(s), where the formation enthalpies  $(\Delta G_f)$  of GeO, GeO<sub>2</sub>, SiO<sub>2</sub>, and Er<sub>2</sub>O<sub>3</sub> are -237.2, -521.4, -856.3, and -1808.7 kJ/mol, respectively.<sup>12</sup> Indeed, reconstruction of SiO<sub>2</sub> is favorable in Ge implanted SiO<sub>2</sub>, where Ge atoms try to form NCs by Ostwald ripening and eventually triggers the formation of GeODCs near the NC/SiO<sub>2</sub> interface. The  $Er_2O_3$  configuration is preferable in  $Er: SiO_2$  due to high  $\Delta G_f$ and leads to the formation of Er oxide clusters during annealing [Fig. 1(a)]. We should note here that in contrary to Ref. 16, HRTEM results reveal that Er doping does not give rise to (i) preferential nucleation of amorphous Ge, (ii) fragmentation of NCs by sputtering, (iii) evolution of bigger NCs, or (iv) dissolution of NCs via ion-beam-mixing for Ge-NCs:  $SiO_2$  layers (Fig. 1). Therefore, even if we assume a variation in the cross-section without any change in microstructure, the two *independent* changes in Fig. 2 (quenching of the 1532 nm Er EL and the rise of the Ge-related 400 nm EL intensity) cannot be interpreted, unless we consider an energy transfer mechanism to the GeODC via Er<sup>3+</sup>, called inverse energy transfer process.

The energy levels up to  ${}^{2}H_{11/2}$  of an Er<sup>3+</sup> ion lay below the level  $T_1$  of GeODC and hence cannot take part in energytransfer process. Although the high energy level Er transitions are not evident in case of Er:SiO<sub>2</sub> by using a low J value (0.17 mA/cm<sup>2</sup>), upon injection of electrons with a moderate J (~7.1 mA/cm<sup>2</sup>), other Er-related EL peaks, es051903-3 Kanjilal et al.

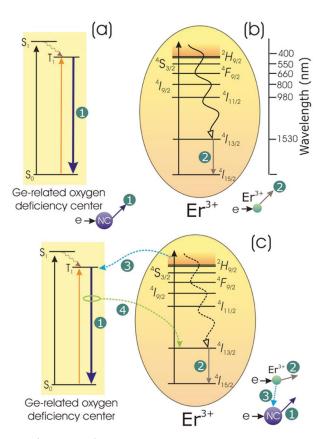


FIG. 4. (Color online) A hot electron excited Ge-related defect center in Ge-NCs:SiO<sub>2</sub> is shown in (a), where the radiative transition from the level  $T_1$  is marked by 1. Electronic excitation of an Er<sup>3+</sup> in Er:SiO<sub>2</sub> is shown in (b), while the downward arrow labeled as 2 represents the infrared emission of Er<sup>3+</sup>. The downward wavy arrow up to  ${}^4I_{13/2}$  represents a nonradiative transition. Both the radiative defect center and Er<sup>3+</sup> are excited by colliding with hot electrons in Er:Ge-NCs:SiO<sub>2</sub> (c). Here, the downward wavy dashed arrow up to  ${}^4I_{13/2}$  signifies nonradiative transition, which is either absent or strongly reduced. In all three cases, the corresponding impact-excitation processes are elucidated in the lower right corner of (a)–(c), respectively.

pecially the  ${}^{2}H_{9/2} \rightarrow {}^{4}I_{15/2}$  transition mediated ~410 nm EL, can be visible (inset of Fig. 2). Clearly, the Er energy levels lying almost in the same height or above than that of  $T_1$  in GeODC [schematically shown in Fig. 4(a)] can contribute to the energy transfer process. For 3.5% of Ge, the 400 nm EL yield reaches its maximum by doping Er up to 0.8%, while the peak intensity quenches by further increase in Er due to concentration quenching.<sup>1</sup> To follow the proposed energy transfer process, the EL lifetime of the  $Er^{3+}$  excited states has further been examined using a stretched exponential function,  $^{17}$  which gives  $\tau^{\rm decay}$  of  ${\sim}214$  and 228  $\mu s$  in Er:SiO<sub>2</sub> for the 410 and 550 nm EL, respectively. Because of close proximity, as the 410 nm Er peak is submerged into the Ge-related 400 nm EL intensity in Er: Ge-NCs: SiO<sub>2</sub>, we estimate the corresponding rise (  $au^{
m rise}$  ) and decay (  $au^{
m decay}$  ) times for both Ge-NCs:SiO<sub>2</sub> and Er:SiO<sub>2</sub> layers (not shown). While the values of  $\tau^{rise}$  and  $\tau^{decay}$  are 46 and 66  $\mu$ s for the former, the respective values are 27 and 214  $\mu$ s for the latter. As expected from the energy transfer process, the radiative lifetime of the 400 nm EL hardly changes by Er doping,<sup>2</sup> revealing an increase in  $\sigma$  of the 400 nm EL by an order of three by introducing Er ions.

Based on our results, we believe that the transitions from the  ${}^{2}H_{9/2}$  level or above to the ground  ${}^{4}I_{15/2}$  level are likely to

be accompanied by a nonradiative relaxation to the lower lying levels of  $\text{Er}^{3+}$  in  $\text{Er}: \text{SiO}_2$  and as a consequence intensify the green, red, and infrared EL [Fig. 4(b)]. However, the decay time measurements indicate that the  $\text{Er}^{2}H_{9/2}$  level is excited faster than that of the GeODC and can initiate an energy transfer process in  $\text{Er}:\text{Ge-NCs}:\text{SiO}_2$  from  $\text{Er}^{3+}$  to the level  $T_1$  of Ge-related defects (process 3) based on their respective lifetimes [Fig. 4(c)], analogous to the energy back transfer process as reported in Ref. 18. The 1532 nm EL quenching signifies either absence of an additional excitation path from the higher-energy levels to the state  ${}^{4}I_{13/2}$  of  $\text{Er}^{3+}$ or excitation of the  $\text{Er}^{3+}$  ions staying apart from Ge NCs. A possible energy back transfer process (process 4) in Fig. 4(c) plays a minor role only.

In summary, we experimentally demonstrated an increase in the Ge-related 400 nm EL intensity at the expense of the Er emission in Er-doped SiO<sub>2</sub> layers containing Ge NCs, showing a strong coupling between the  $\text{Er}^{3+}$  ions and GeODCs. Since the microstructure of the Ge-rich SiO<sub>2</sub> layers is found unaffected by Er doping, based on the decay time measurements, we conclude that an energy transfer from  $\text{Er}^{3+}$  to the triplet state  $T_1$  of the Ge-related defect centers can only justify the observed electroluminescence.

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# Resonant enhancement of second order sideband generation for intraexcitonic transitions in GaAs/AIGaAs multiple quantum wells

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We present an experimental study on efficient second order sideband generation in symmetric undoped GaAs/AlGaAs multiple quantum wells. A near-infrared laser tuned to excitonic interband transitions is mixed with an in-plane polarized terahertz beam from a free-electron laser. The terahertz beam is tuned either to the intraexcitonic heavy-hole 1s-2p transition or to the interexcitonic heavy-hole light-hole transition. We find strong evidence that the intraexcitonic transition is of paramount influence on  $n=\pm 2$  sideband generation, leading to an order-of-magnitude resonant enhancement of the conversion efficiency up to 0.1% at low temperature. At room temperature, the efficiency drops only by a factor of 7 for low terahertz powers. © 2009 American Institute of Physics. [DOI: 10.1063/1.3155189]

Strong ac fields in the terahertz (THz) region are known to modify interband absorption of semiconductor heterostructures. Besides shifts of the excitonic energies, for instance due to the ac-Stark effect<sup>1</sup> or dynamical Franz– Keldysh effect,<sup>2</sup> strong THz fields can lead to spectral sidebands of an interband excitation. Sidebands have been studied in bulk GaAs,<sup>3</sup> but especially in semiconductor heterostructures using magnetoexcitons and excitonic and electronic intersubband transitions.<sup>4–8</sup> Besides high conversion efficiencies, semiconductor heterostructures have the advantage that intersubband energies can be tuned by applying a dc field,<sup>9</sup> making these devices potentially attractive for electrically controlled switches and modulators.

In THz sideband generation, the ac THz field with a frequency  $\omega_{\text{THz}}$  modulates the polarization induced by a near-infrared (NIR) beam of frequency  $\omega_{\text{NIR}}$ . New frequencies result at  $\omega = \omega_{\text{NIR}} \pm n \omega_{\text{THz}}$ . In symmetric structures, *n* is restricted to even integer numbers,<sup>4</sup> whereas in asymmetric quantum wells also odd sidebands are allowed.<sup>10</sup> Sideband generation at weak THz fields can be modeled using a nonlinear susceptibility and low-order perturbation theory.<sup>10,11</sup> Highest reported efficiencies in quantum wells for an *n*=+1 process were around 0.2% in a THz waveguide geometry with the THz beam tuned to an excitonic hole intersubband transition.<sup>12</sup> For *n*=+2, slightly lower efficiencies were reported for magnetoexcitons in strong magnetic fields.<sup>5</sup>

Here, we report on efficient  $n = \pm 2$  sideband generation up to room temperature in a relatively simple geometry. We employ an in-plane THz field in an effective three-level system, consisting of the 1s and 2p states of the heavy-hole (hh) exciton, and the 1s state of the light-hole (lh) exciton in multiple quantum wells (MQWs). Note that no magnetic field is needed to separate the energy levels. We demonstrate experimentally how the  $n = \pm 2$  sideband efficiency scales when the THz beam is tuned between interexcitonic hh-lh and intraexcitonic hh(1s-2p) transitions. The experimental scheme is indicated in Fig. 1(a). A tunable NIR Ti:sapphire laser delivering 2.5 ps long pulses is transmitted through the sample and detected by a charge coupled device camera attached to a grating spectrometer. The sample studied consists of 60 periods of undoped GaAs quantum wells, each 8.2-nm-thick and separated by 19.6-nm-thick barriers of  $Al_{0.34}Ga_{0.66}As$ . To allow transmission measurements, the sample was glued to NIR-transparent (100)-oriented ZnTe and the semi-insulating GaAs substrate has been etched away. The sample was kept in a liquid He flow cryostat equipped with a diamond window for the strong THz beam which is overlapped with the NIR beam near normal incidence on the sample. We employ FELBE, the free-electron laser (FEL) at the Forschungszentrum Dresden-

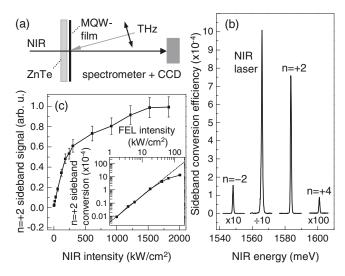


FIG. 1. (a) Experimental setup. (b) Transmitted sideband spectrum at 10 K for  $\hbar\omega_{\text{THz}}$ =8.9 meV near the hh(1*s*-2*p*) transition. For clarity, the *n*=-2 and *n*=+4 sidebands are multiplied by 10 and 100, respectively, and the NIR laser is divided by 10. The THz peak intensity was around 65 kW/cm<sup>2</sup>. The NIR intensity was 3 kW/cm<sup>2</sup>. (c) For an FEL peak intensity of about 8 kW/cm<sup>2</sup> the *n*=+2 sideband signal depends linearly on NIR intensity until saturation is reached. The inset shows the quadratic behavior of the *n*=+2 sideband with FEL intensity (the dotted line is a quadratic fit).

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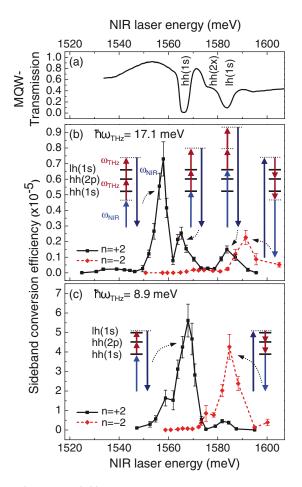


FIG. 2. (Color online) (a) MQW film transmission spectrum at 10 K with hh(1s), hh(2x), and hh(1s) transition (x marks the s and p state). (b) Sideband spectra for n=+2 (black line) and n=-2 (red dashed line) for an NIR intensity of 180 kW/cm<sup>2</sup>, an FEL intensity around 18 kW/cm<sup>2</sup> and an FEL energy of 17.1 meV, corresponding to the hh(1s)-lh(1s) transition. Schematic level diagrams indicate the involved transitions. (c) Same as (b) for an FEL energy of 8.9 meV near the estimated hh(1s-2p) resonance.

Rossendorf. This THz source can be tuned continuously from 6 to 300 meV and delivers pulses at a repetition rate of 13 MHz with pulse durations of 10–25 ps for the energies used here. The NIR laser repetition rate was also reduced down to 13 MHz by an acousto-optical pulse picker to temporally overlap each NIR pulse with an FEL pulse. The polarizations of the FEL and NIR laser were linear and parallel to each other for maximum sideband signal.

Figure 2(a) shows the transmission spectrum of the thin MQW film, obtained through normalizing the signal by the ZnTe substrate transmission. hh and lh exciton transitions can be distinguished. At an energy of 1575 meV, the onset of the hh continuum transitions can be seen, starting with the interband allowed hh(2s) exciton state. The nearby 2p state is optically interband forbidden but should lie around 9 meV above the hh(1s) transition.<sup>13</sup> However, the exciton states is allowed and couples strongly to THz radiation.<sup>2,5</sup>

In Fig. 1(b), a typical low-temperature sideband spectrum is measured under FEL illumination with the NIR laser tuned near the hh(1s) exciton energy. Different even orders of sidebands could be observed. Due to slight asymmetry of the quantum wells resulting from MBE growth, also the odd sidebands  $n = \pm 1$  appear with typical intensities 1000 times smaller than  $n = \pm 2$  (not shown here). Corrected for the

losses in the ZnTe substrate, the n=+2 sideband reveals a conversion efficiency between incoming NIR power and emitted sideband of 0.13% for an FEL intensity of 145 kW/cm<sup>2</sup>, which is high for an n=+2 process.<sup>12</sup>

In Fig. 1(c), the n=+2 sideband power with the NIR laser at the hh(1s) resonance depends linearly on the NIR intensity until saturation is reached. The following experiments were performed at an NIR peak intensity of 180 kW/cm<sup>2</sup> on the quantum well film, resulting in an exciton density at the strongly absorbing hh(1s) exciton of  $3 \times 10^{10}$  cm<sup>-2</sup>. A measurement with 10% of this relatively high exciton density was also taken but the trends presented here stayed the same and only a reduced linewidth was observed. The inset of Fig. 1(c) displays the quadratic dependence of the n=+2 power with FEL intensity, as expected for this  $\chi^{(3)}$  process.

Now, in Fig. 2(b), the NIR laser energy was tuned at a fixed FEL energy, and at each position, the n=+2 (black line) and n=-2 (red dashed line) sideband power was recorded. First, we address the case where the FEL energy is fixed at 17.1 meV, corresponding to the interexcitonic hh(1s)-lh(1s)separation. For the n=+2 sideband, three pronounced resonances are observed and their origin can be seen qualitatively from the schematic energy level diagrams. The strongest resonance at 1557 meV lies slightly below the band edge. One FEL-photon energy above we find the hh(2s) and hh(2p) state in the transmission spectrum, serving as an intermediate state for the mixing. The relative strength of the resonance peak shows that the 1s-2p transition couples strongly to the THz radiation, although the NIR laser is only resonant with a virtual level and the final state lies in the continuum above the lh. The sideband resonance signal at the hh(1s) state is smaller, although one FEL photon is resonant with the hh(1s)-lh(1s) transition. However, for small inplane wave vectors, this transition is weak.<sup>14</sup> The third resonance at the lh(1s) position is less pronounced since the lh exciton is coupled to continuum states and not to sharper excitonic states like in the aforementioned cases. Note that the intraband optically allowed hh(2s) state does not lead to a pronounced feature in the sideband resonance scan. The n=-2 sideband spectrum is consistent with this picture, showing highest conversion signal when the NIR laser is tuned 17.1 meV above the hh(2p) state. Thus we find that the hh(2p) state as an intermediate state for the nonlinear mixing dominates both the n=+2 and n=-2 sideband spectra.

We now address the case where the FEL energy is fixed at 8.9 meV near the estimated hh(1s-2p) resonance. The corresponding NIR sideband spectrum is shown in Fig. 2(c). One observes an increase in the n=+2 sideband signal at the hh exciton by a factor of 20. Now the FEL is resonant with the hh(1s-2p) transition and also approximately with the hh(2p)-lh(1s) transition, leading to an efficient n=-2 generation as well. The n=+2 spectrum shows a weak resonance at 1583 meV at the lh(1s) state, similar to Fig. 2(b). The additional small shoulders at 1575 meV (for n=-2) and 1559 meV (for n=+2) cannot be assigned with absolute certainty to a particular resonance.<sup>15</sup>

At a temperature of 77 K, there is no drop in efficiency compared to the low-temperature measurement. At room temperature, the n=+2 sideband resonance broadens, as shown in Fig. 3, for a THz intensity of 18 kW/cm<sup>2</sup> (black line) and 41 kW/cm<sup>2</sup> (red dashed line). The broadening

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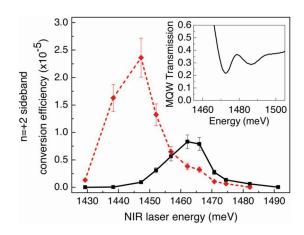


FIG. 3. (Color online) n=+2 sideband resonance taken above room temperature for an NIR intensity of 180 kW/cm<sup>2</sup>, an FEL intensity of 18 kW/cm<sup>2</sup> (black line) and 41 kW/cm<sup>2</sup> (red dashed line). The FEL energy is fixed at 8.9 meV. Due to pronounced lattice heating, the sideband spectra appear shifted to lower energies by around 7 meV (black line) and 24 meV (red dashed line), respectively, with respect to the MQW transmission without FEL (inset, taken at 290 K).

stems from broader excitonic resonances in the transmission spectrum (Fig. 3 inset). Nevertheless, at lower THz intensities of 18 kW/cm<sup>2</sup>, we still find an efficiency which is only reduced by a factor of 7 compared to the 10 K measurement. The FEL energy was kept at 8.9 meV, though the hh-lh splitting decreases from 17 to 14 meV. However, with the broader absorption lines, the need for matching the THz energy exactly with the energy levels is relaxed. Note that both spectra differ in the spectral position from the transmission measurement taken at 290 K. To overlap them with the transmission measurement, they have to be shifted to higher energies by 7 and 24 meV, respectively. This shift in bandgap is due to a pronounced FEL-induced lattice heating by roughly 17 and 52 K for 18 and 41 kW/cm<sup>2</sup>, respectively.

In summary, we have demonstrated efficient  $n = \pm 2$  sideband generation in a GaAs/AlGaAs MQW film with the THz beam under normal incidence. Relatively high conversion efficiencies of the 1.7  $\mu$ m thin quantum well film above 0.1% were measured despite somewhat broad linewidths. By tuning the THz energy in resonance with the hh(1*s*-2*p*) tran-

sition, we showed that this intraexcitonic transition dominates the sideband spectra compared to a THz energy of the interexcitonic transition between hh and lh states. Slightly above room temperature, the sideband efficiency drops only by a factor of 7. Together with the possibility of shifting the sample's exciton transitions via an applied bias,<sup>9</sup> one could think of efficient room temperature switches or modulators based on such systems.

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# Statistics

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Effect of the nature of anions of aluminum salts used to synthesize a precursor of the Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> ceramics on the stabilization of the tetragonal modification of zirconium dioxide *Zhurnal Prikladnoi Khimii* 82, 1268 (2009). *Russian Journal of Applied Chemistry* 82, 1364 (2009)

## Invited Conference Talks

 Abrasonis, G. Surface diffusion assisted phase separation during the growth of carbon:transition metal nanocomposite thin films

2<sup>nd</sup> Plasma-Nano International Workshop at Australia's Commonwealth Scientific and Industrial Research Organisation (CSIRO), 9.02.2009, Lindfield, Australia

 Brauer, G.; Anwand, W.; Grambole, D.; Egger, W.; Sperr, P.; Beynik, I.; Hofer, C.; Teichert, C.; Kuriplach, J.; Lang, J.; Zviagin, S.; Cizmar, E.; Ling, C. C.; Hsu, Y. F.; Xi, Y. Y.; Djurisic, A. B.; Skorupa, W. Characterization of ZnO nanostructures: A challenge to positron annihilation spectroscopy and other methods

15th International Conference on Positron Annihilation, 18.-23.01.2009, Kolkata, India

- Facsko, S.; Ranjan, M.; Keller, A.; Oates, T. W. H.; Rosen, J. Self-aligned metal nanoparticles and nanowires grown on ripple-templates MRS 2009 Fall Meeting, Symposium "Nanosclae Pattern Formation", 30.11.-4.12.2009, Boston MA, USA
- Faßbender, J.
   Engineering surfaces, interfaces and structural phases to tailor magnetic properties Gaede-Preis-Vortrag, Frühjahrstagung der Deutschen Physikalischen Gesellschaft, 24.03.2009, Dresden, Germany
- Faßbender, J.; Liedke, M. O.; Strache, T.; Marko, D.; Lenz, K.; Keller, A.; Facsko, S.; Tibus, S.; Springer, F.; Albrecht, M.; Rohrmann, H.
   Periodically modulated surfaces and interfaces: Introducing artificial length scales to tailor magnetic properties

MRS 2009 Spring Meeting, Symposium "Ion Beams and Nano-Engineering", 13.-17.04.2009, San Francisco, USA
2<sup>nd</sup> International Conference on Physics at Surfaces and Interfaces, 23.-27.02.2009, Puri, Orissa, India

- Faßbender, J. Nanomagnets - Created and tailored by ions International Seminar and Workshop on Magnonics: From Fundamentals to Applications, 2.-7.08.2009, Dresden, Germany
- 8. International Workshop "Nanoscale Modification of Surfaces and Thin Films", 30.08.-3.09.2009, Rathen, Germany
- Faßbender, J.; Liedke, M. O.; Körner, M.; Markó, D.; Lenz, K.; Facsko, S. Ion-erosion-induced pattern as template for layers with magnetic anisotropy and coupling MRS 2009 Fall Meeting, Symposium "Nanosclae Pattern Formation" 30.11.-4.12.2009, Boston MA, USA
- Gemming, S. First-principles investigation of metal/oxide contacts and related metal/group-IV interfaces CECAM-Workshop on Metal-Ceramic Interfaces, 20.-22.07.2009, Zürich, Switzerland
- 11. Heinig, K.-H. Fundamentals of ion beam synthesis of nanostructures
- 12. Formation of nanodots, nanowires, wormholes etc. driven by capillary forces-reaction pathways predicted by atomistic simulations
- 13. Spontaneous formation of regular nanopattern on surfaces during thin film deposion and ion beam erosion

3 Talks given at Winter School on Nanoelectronics and Nanophotonics, 19.-25.01.2009, Ankara, Turkey

- Heinig, K.-H.; Beyer, V.; Schmidt, B.; Stegemann, K.-H. Novel LEFET with two self-aligned Si nanocrystal layers Workshop on Nanoelectronics and Nanophotonics, 26.-28.01.2009, Ankara, Turkey
- Heinig, K.-H.; Liedke, B.; Numazawa, S.; Ranjan, M.; Möller, W.; Facsko, S. Ion-beam-assisted nanostructure formation – Atomistic reaction pathways International Conference on Ion-Surface Interactions 2009, 21.-25.08.2009, Zvenigorod, Russia
- Heinig, K.-H.; Schmidt, B.; Vredenberg, A.; Polman, A.; Toulemonde, M. Shaping of Au and Ge nanoparticles in SiO<sub>2</sub> with swift heavy ions International Workshop on Nanostructures in Silica, 6.-9.09.2009, Ivalo, Finland

- Kosmata, M.; Grötzschel, R.; Munnik, F.; Vieluf, M.; Möller, W.
   Quantitative ion beam analysis of ultra thin films with magnetic spectrometers using heavy MeV ions 5<sup>th</sup> International Workshop on High-Resolution Depth Profiling, 15.-19.11.2009, Kyoto, Japan
- Leiderer, P.; Erbe, A.
   Colloids: Mesoscopic model systems of matter on a nano scale Juelich Soft Matter Days 2009, 10.-13.11.2009, Bonn, Germany
- Lorenz, K.; Magalhães, S.; Franco, N.; Darakchieva, V.; Barradas, N. P.; Alves, E.; Pereira, S.; Correia, M. R.; Munnik, F.; Martin, R. W.; O'Donnell, K. P.; Watson, I. M. Al<sub>1-x</sub>In<sub>x</sub>N/GaN bilayers: Structure, morphology and optical properties International Conference on Nitride Semiconductors, 18.-23.10.2009, JeJu, South Korea
- 20. McCord, J.; Hamann, C.; Martin, N.; Mönch, I.; Kaltofen, R.; Schultz, L.; Strache, T.; Faßbender, J.; Mattheis, R.
  - Static and dynamic magnetization processes in magnetic property patterned thin films International Magnetics Conference, 4.-8.05.2009, Sacramento, USA
- McCord, J.; Faßbender, J.
   Designing soft magnetic materials by ion irradiation 18<sup>th</sup> International Materials Research Congress, 16.-20.08.2009, Cancun, Mexiko
- Posselt, M.; Wündisch, C.; Schmidt, B.; Schumann, T.; Mücklich, A.; Skorupa, W.; Clarysse, T.; Simoen, E.; Hortenbach, H.
   Millisecond flash lamp annealing of ultra-shallow implanted layers in Ge 216<sup>th</sup> Meeting of the Electrochemical Society, Symposium "ULSI Process Integration", 4.-9.10.2009, Vienna, Austria
- Schneider, H.; Liu, H. C.; Maier, T.; Walther, M. **Two-photon photocurrent spectroscopy of quantum well intersubband relaxation and dephasing** *SPIE Photonics West*, 24.-29.01.2009, San Jose CA, USA
- Schneider, H.; Liu, H. C.; Winnerl, S.; Drachenko, O.; Helm, M.; Walther, M.; Faist, J. Resonant two-photon transitions, quadratic detection, and photocurrent autocorrelation using multiple quantum wells SPIE Photonics West, 24.-29.01.2009, San Jose CA, USA
- Schneider, H. Quantum well infrared photodetectors for dual-band thermal imaging and two-photon detection 3<sup>rd</sup> International Symposium on Photoelectronic Detection and Imaging, 17.-19.06.2009, Beijing, China
- Schneider, H.; Winnerl, S.; Drachenko, O.; Helm, M.; Liu, H. C.; Song, C.; Maier, T.; Walther, M.; Faist, J. **Two-photon intersubband transition physics and detectors** 10<sup>th</sup> International Conference on Intersubband Transitions in Quantum Wells, 6.-11.09.2009, Montréal, Canada
- 27. Skorupa, W. Nanostructuring of semiconductors with ion beams LENS 2009 Summer School, 11.-14.06.2009, Reykjavik, Iceland
- Skorupa, W.
   Advances in Si & Ge millisecond processing: From silicon-on-insulator to superconducting Ge 17<sup>th</sup> IEEE International Conference on Advanced Thermal Processing of Semiconductors, 29.09.-2.10.2009, Albany NY, USA
- 29. Sort, J.; Menendez, E.; Varea, A.; Pi, F.; Surinach, S.; Baro, M. D.; Nogues, J. Nanoscale magnetic lithography on FeAl alloys by ion irradiation and nanoindentation International Conference on Advanced Materials, 20.-25.09.2009, Rio de Janeiro, Brazil
- Stehr, D.; Wagner, M.; Schneider, H.; Helm, M.; Andrews, A. M.; Roch, T.; Strasser, G. Two color pump-probe studies of intraminiband relaxation in doped GaAs/AlGaAs superlattices SPIE Photonics West, 24.-29.01.2009, San Jose CA, USA
- Vinnichenko, M.; Cornelius, S.; Rogozin, A.; Shevchenko, N.; Gago, R.; Kolitsch, A.; Möller, W. Properties of transparent conductive oxides deposited by magnetron sputtering 16<sup>th</sup> International Summer School on Vacuum, Electron and Ion Technologies, 28.09.-2.10.2009, Sunny Beach, Bulgaria
- 32. Zhou, S.

**Transition metal implanted ZnO: A correlation between structure and magnetism** *Thyssen Krupp Electrical Steel Dissertationspreis der AG Magnetismus, Frühjahrstagung der Deutschen Physikalischen Gesellschaft,* 22.-27.03.2009, *Dresden, Germany* 

### Conference Contributions

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- 161. SPIE Europe Microtechnologies for the New Millennium, 4.-6.05.2009, Dresden, Germany
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### Lectures / Talks

- Bischoff, L. Application of focused ion and electron beams in materials research Symposium at Imdea Nanociencia, 4.11.2009, Madrid, Spain
- 2. Kolloquiumsvortrag am Leibniz Institut für Oberflächenmodifizierung Leipzig, 10.12.2009, Leipzig, Germany
- Brauer, G. Positron annihilation spectroscopy in connection with activities towards p-type doping of ZnO Seminar at University of Orissa, Institute of Physics, 16.01.2009, Bhubaneswar, India
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   Characterisation of ZnO single crystals A challenge to positron annihilation spectroscopy Seminar am Institut f
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  üchtung Berlin, 17.08.2009, Berlin, Germany
- 5. Seminar at Charles University Prague, 2.11.2009, Prague, Czech Republic
- Eng, L. M.; Grafström, S.; Seifert, G.; Gemming, S.
   Polarisationsinduzierte Oberflächenfunktionalität an Ferroelektrika Abschlusskolloquium SPP 1157 "Integrierte elektrokeramische Funktionsstrukturen " 21.-22.09.2009, Bonn, Germany

7.	Erbe, A. <b>Capped colloidal particular: A model system for spin arrangements</b> Workshop SPP 1296 "Heterogene Keim- und Mikrostrukturbildung", 1314.10.2009, Bayreuth, Germany
8.	Faßbender, J. <b>Künstliche magnetische Materialien durch Nanostrukturierung</b> Kolloquiumsvortrag, Institut für Physik, Universität Mainz, 16.02.2009, Mainz, Germany
9.	Faßbender, J. Nanomagnets - Created and tailored by ions Forschungsseminar, Institut für Physik, HU Berlin, 13.05.2009, Berlin, Germany
	SFB 668 - Kolloquium "Magnetismus vom Einzelatom zur Nanostruktur", Institut für Angewandte Physik und Zentrum für Mikrostrukturforschung der Universität Hamburg, 16.06.2009, Hamburg, Germany Kolloquiumsvortrag Fachbereich Physik, Universität Paderborn, 2.07.2009, Paderborn, Germany
12.	Gemming, S. <b>Unusual transport phenomena in nanostructured materials</b> <i>NanoSeminar, 5.02.2009, Dresden, Germany</i>
13.	Gemming, S. <b>Modelling electronic and transport properties in ferroic devices</b> <i>Fraunhofer IWM - Seminarreihe zur Materialforschung,</i> 13.02.2009, <i>Freiburg, Brsg., Germany</i>
14.	Gemming, S. <b>Transport through nanostructures</b> Sächsisches Landesseminar Theoretische Chemie, 26.02.2009, Leipzig, Germany
15.	Gemming, S. <b>Simulation of bone replacement materials</b> Seminar "Topical Problems in Theoretical Physics", TU Chemnitz, 16.12.2009, Chemnitz, Germany
16.	Gemming, S. <b>Multi-scale approaches for structure and conductivity of complex materials</b> <i>Colloquium on Multi-Scale Modeling, 4.05.2009, Vienna, Austria</i>
17.	Gemming, S. <b>Density-functional theory in materials science</b> FZD Theory Seminar Series, 17.12.2009, Dresden-Rossendorf, Germany
18.	Grebing, J.; Dietsche, R.; Ganteför, G.; Kirchner, T.; Scheer, E. <b>Electronic transport measurements on Si<sub>4</sub> clusters</b> <i>Clustertreffen 2009, 49.10.2009, Herzogenhorn, Schwarzwald, Germany</i>
19.	Grenzer, J. <b>X-ray investigations off nanostructures manufactured by focused ion beam techniques</b> <i>Seminar, Charles University Prague, 6.01.2009, Prague, Czech Republic</i>
20.	Helm, M. <b>Nanometrologie für die Nanoelektronik</b> Talk at BMBF Cluster of Excellence Meeting "Cool Silicon", 6.05.2009, Dresden, Germany
21.	Helm, M. <b>Solid state spectroscopy using an infrared free-electron laser</b> Colloquium at Institute for Molecules and Materials, Radboud University, 10.11.2009, Nijmegen, The Netherlands
22.	Helm, M. <b>Infrared solid state spectroscopy using a free-electron laser</b> <i>Seminarvortrag bei ANKA</i> , 2.03.2009, <i>Karlsruhe, Germany</i>
23.	Helm, M. Silicon based light emitters: From rare-earth doped MOS devices to resonant-cavity LEDs Seminar at van der Waals - Zeeman Institute, University Amsterdam, 27.03.2009, Amsterdam, The Netherlands
24.	Hubrich, R. <b>Radiale Moden in THz-Feldern</b> Drittes THz-Frischlinge-Meeting 2009, 710.06.2009, Berlin, Germany
25.	Keller, A.; Facsko, S.; Möller, W. <b>Self-organized ripple patterns by ion erosion - Experiment, theory, and application</b> <i>Seminar, Institute of Solid State Electronics, TU Vienna, 16.06.2009, Vienna, Austria</i>

26.	Kosmata, M. <b>Ion beam analysis with sub nanometer depth resolution</b> 4 <sup>th</sup> Graduate Students Seminar, 1618.09.2009, Krögis, Germany
27.	Lenz, K. <b>Spin dynamics in ferromagnets: Gilbert damping vs. two-magnon scattering</b> <i>Talk at the Physics Seminar Series,</i> 12.10.2009, <i>Colorado Springs, USA</i>
28.	Talk at the Electromagnetics Group Seminar, 13.10.2009, Boulder, Colorado, USA
29.	Lenz, K. <b>Tailoring magnetic properties using ion beam irradiation</b> Division Seminar of the Condensed Matter Theory Group, 15.10.2009, Irvine, CA, USA
30.	Lenz, K.; Marko, D.; Strache, T.; Faßbender, J. <b>Spin wave excitations: coupling and damping effects in ultrathin films</b> 448. WE-Heraeus-Seminar: Excitement in Magnetism: Spin-dependent Scattering and Coupling of Excitations in Ferromagnets, 2225.11.2009, Ringberg, Germany
31.	Liedke, B.; Heinig, KH.; Möller, W. <b>Modelling of ion beam mixing with phase separation - Simulation of damage formation and thermally</b> <b>activated relaxation</b> <i>Seminar at Department of Physics, University of Bialystok, 21.11.2009, Bialystok, Poland</i>
32.	
33.	Merchel, S.; Bichler, M.; Sterba, J. H. <b>On the reliability of target element data for cosmogenic nuclide exposure dating</b> <i>Seminar Activation Analysis and Gamma-Spectroscopy</i> , 2527.02.2009, TU Vienna, Austria
34.	Merchel, S.; Grambole, D.; Grötzschel, R.; Munnik, F.; Neelmeijer, C. Accelerator mass spectrometry (AMS) and ion beam analysis (IBA) with the new 6 MV accelerator at FZ Dresden-Rossendorf Seminar Activation Analysis and Gamma-Spectroscopy, 2527.02.2009, TU Vienna, Austria
35.	Merchel, S. <b>Terrestrial cosmogenic radionuclides and the new AMS facility DREAMS</b> <i>Seminar über aktuelle Themen aus Kosmochemie und Astrophysik, 06.05.2009, Universität Mainz, Germany</i>
36.	Merchel, S.; Munnik, F. <b>Beschleunigermassenspektrometrie und Ionenstrahlanalytik</b> Seminarreihe "Moderne analytische Methoden der Physik", 14.07.2009, Berlin, Germany
37.	Merchel, S. <b>Cosmogenic nuclides: Endless opportunities?</b> GeoKolloquium - Geowissenschaftliches Kolloquium der Fakultät für Geowissenschaften, Geotechnik & Bergbau, TU Bergakademie Freiberg, 8.04.2009, TU Bergakademie Freiberg, Germany
38.	Möller, W. <b>Nanostructures by ion-driven self-organisation: Can ions induce order?</b> <i>Colloquium at Shanghai Institute of Microsystems and Information Technology, 5.05.2009, Shanghai, China</i>
39.	Möller, W. <b>Self-organized nanostructures by ion irradiation</b> Polymer-Physics-Seminar, Charles University Prague, 10.11.2009, Prague, Czech Republic
40.	Munnik, F.; Merchel, S. <b>Ion beam analysis (IBA) for thin film studies</b> <i>Seminarreihe "Moderne analytische Methoden der Physik",</i> 14.07.2009, <i>Berlin, Germany</i>
41.	Neelmeijer, C. <b>Zerstörungsfreie Materialanalyse an Kunstwerken</b> <i>Gastvorlesung, 24.02.2009, HfBK Dresden, Germany</i>
42.	Neidhardt, J. <b>Atomic billiard – Materials science using hyperthermal ions</b> <i>Lecture, 16.11.2009, University Linköping, Sweden</i>
43.	Odor, G.; Liedke, B.; Heinig, KH. Mapping of 2+1 dimensional KPZ growth onto driven lattice gas model of dimers

International Seminar on "Many-body systems far from equilibrium: Fluctuations, slow dynamics and long-range interactions", 16.-27.02.2009, Dresden, Germany

- Rebohle, L. Rare earth implanted MOS light emitting devices Seminar Talk, Department of Physics, University of Hong Kong 8.05.2009, Hong Kong, China
   Rebohle, L. Si-based light emitters: How bright is the future? Seminar Talk, Department of Electronics, University of Barcelona, 29.09.2009, Barcelona, Spain
   Schneider, H. Time-resolved infrared and terahertz spectroscopy of semiconductor quantum structures Seminar, Institute of Optics and Precision Mechanics, Xi'an University of Technology, 22.06.2009, Xi'an, China
- Schneider, H.
   Ultrafast infrared and terahertz spectroscopy of semiconductor quantum structures Physics Colloquium, Shanghai Jiao Tong University, 30.12.2009, Shanghai, China
- Schneider, H. Infrared and THz spectroscopy of semiconductor quantum structures with modelocked tabletop and freeelectron lasers Seminar, Rensslaer Polytechnic Institute, 14.09.2009, Troy NY, USA
- Skorupa, W.; Anwand, W.; Gebel, T. Energy pulse modification of electronic materials: From electronics via photonics to other advanced materials European Project Meeting "Teramagstor", 28.-29.05.2009, Santorini, Greece
- 50. Skorupa, W.; Anwand, W.; Gebel, T. Energy pulse modification of electronic materials: from electronics via photonics to other advanced materials Seminar at the University of Hong Kong, 8.05.2009, Hong Kong, China
- Zhou, J.; Facsko, S.; Keller, A.; Möller, W.
   Ion sputtering of Si surfaces at normal incidence: roughening versus smoothening Meeting DFG-Forschergruppe 845 "Selbstorganisierte Nanostrukturen durch niederenergetische Ionenstrahlerosion", 31.03.2009, Köln, Germany

### PhD Theses

- Berndt, M. Phase separation in carbon: transition metal nanocomposite thin films *TU Dresden*, 21.12.2009
- Beyer, V. Nanocrystals for nanodot memories - Ion beam synthesis and electrical studies Leibniz Universität Hannover, 11.12.2009
- 3. Güttler, D.

An investigation of target poisoning during reactive magnetron sputtering *TU Dresden*, 12.03.2009, *Wissenschaftlich-Technische Berichte FZD-515* (2009)

4. Heller, R.

**Wechselwirkung langsamer hochgeladener Ionen mit der Oberfläche von Ionenkristallen** *TU Dresden, 30.06.2009, Wissenschaftlich-Technische Berichte FZD-520 (2009)* 

- Keller, A.
   Evolution of ion-induced ripple patterns Anisotropy, nonlinearity, and scaling *TU Dresden*, 8.06 2009
- Ou, X. Defect engineering in SIMOX processing Shanghai Institute of Microsystems and Information Technology, Chinese Academy of Science, Shanghai, China, 1.07.2009
- Potfajova, J.
   Silicon based microcavity enhanced light emitting diodes TU Dresden, 15.12.2009, Wissenschaftlich-Technische Berichte FZD-526 (2009)

## Master & Diploma Theses

1. Buhl, M. Herstellung und magnetische Charakterisierung von Co-Nanopartikeln auf ionenstrahlerodierten Substraten

Westsächsische Hochschule Zwickau, 30.09.2009

- Hubrich, R. Untersuchung von Terahertz-Moden unterschiedlicher Polarisation TU Dresden, 17.11.2009
- 3. Lehmann, J. Correlation between electrical and optical properties of europium doped SiO<sub>2</sub>-layers *TU Dresden*, 7.10.2009
- Seeger, M. Charakterisierung von Si- MOS-Dioden mittels Kapazitätsspektroskopie TU Dresden, 28.08.2009

## Organization of Conferences / Workshops

- Borany, J. von, Jäger, C. Workshop "He-Ion Microscopy and its Applications" 9.12.2009, Forschungszentrum Dresden-Rossendorf, Germany
- Chason, E., Cuerno, R., Gray, J., Heinig, K.-H. MRS 2009 Fall Meeting, Symposium "Nanoscale Pattern Formation" 30.11.-3.12.2009, Boston MA, USA
- Facsko, S., Heinig, K.-H., Keller, A., Rauschenbach, B. International Workshop "Nanoscale Modification of Surfaces and Thin Films" 30.08.–3.09.2009, Rathen, Germany
- Liu, H. C., Gmachl, C., Helm, M.
   10<sup>th</sup> International Conference on Intersubband Transitions in Quantum Wells (ITQW 2009) 6.-11.09.2009, Montreal, Canada
- Rafaja, D., Bähtz, C.
   EUROMAT 2009, Symposium "X-ray Characterization of Microstructure Features", 7.-10.09.2009, Glasgow, United Kingdom

### Laboratory Visits

- 1. Abrasonis, G. ESRF Grenoble, France; 30.06.-5.07., 27.08.-5.09.2009
- 2. Bischoff, L. University of Madrid, Spain; 2.-6.11.2009
- 3. Borany, J. von ESRF Grenoble, France; 21.-28.01.2009
- 4. Bunce, C. Swiss Light Source, PSI Villigen, Switzerland; 13.-22.12.2009
- 5. Drachenko, O. Kurchatov Institute Moscow/Institute for Microstructures Nishni Novgorod, Russia; 7.-14.04.2009
- 6. Drachenko, O. Laboratoire Nationale des Champs Magnetiques Pulses, Toulouse, France; 12.-21.06.2009
- 7. Grenzer, J. ESRF Grenoble, France; 14.-21.04., 20.-26.05., 13.-17.07., 13.-15.10., 7.-14.12.2009
- 8. Grebing, J. Swiss Light Source, PSI Villigen, Switzerland; 7.-10.09.2009

- 9. Grötzschel, R. CMAM Madrid, Spain; 4.-7.05., 11.-14.05., 21.-23.05.2009 10. Hanf, D. MPI Halbleiterlabor, München, Germany; 5.-9.04.2009 11. Hanisch, A. ESRF Grenoble, France; 30.01.-3.02., 4.-10.07.2009 12. Hanisch, A. DESY Hamburg, Germany; 2.-8.12.2009 13. Heinig, K.-H. KFKI Budapest, Hungary; 14.-18.06.2009 14. Höwler, M. IPHT Jena, Germany; 23.-25.05.2009 15. Höwler, M. Universität Kaiserslautern, 23.-27.08.2009 16. Keller, A. TU Wien, Austria; 16.-27.06.2009 17. Kögler, R. SMIT Shanghai, Hangzhou University, China; 3.-10.05.2009 18. Körner, M. Swiss Light Source, PSI Villigen, Switzerland; 16.-25.02., 6.-10.09., 13.-22.12.2009 19. Kovacs, G. ESRF Grenoble, France; 16.-23.06., 30.06.-5.07., 27.08.-1.09.2009 20. Liedke, B. KFKI Budapest, Hungary; 14.-28.06.2009 21. Liedke, B. University Bialystok, Poland; 24.09.-4.10., 18.-22.11.2009 22. Liedke, M.O. Swiss Light Source, PSI Villigen, Switzerland; 26.04.-3.05.2009 23. Marko, D. Swiss Light Source, PSI Villigen, Switzerland; 16.-25.02., 13.-22.12.2009 24. Martinavisius, A. ESRF Grenoble, France; 27.08.-5.09.2009 25. Merchel, S. Aix-en-Provence, France; 2.-9.07.2009 26. Merchel, S. Ljubljana, Slovenien; 17.-22.07.2009 27. Möller, W. SMIT Shanghai, China; 3.-7.05.2009 28. Morawetz, K. ICCMP Brasilia, Brazil; 7.-30.01., 5.-19.03., 15.-29.04., 4.-19.06.2009 29. Numazawa, S. KFKI Budapest, Hungary; 6.-18.07., 25.11.-6.12.2009 30. Rebohle, L. SMIT Shanghai, City University Hong Kong, China; 3.-12.05.2009 31. Schneider, H. Xi'an University, Beijing, China; 15.-24.06.2009 Schumann, T. 32. Oslo University, Norway; 7.-20.06.2009 33. Shalimov, A. ESRF Grenoble, France; 16.-23.06., 17.-24.11.2009
- 34. Shevchenko, N. ESRF Grenoble, France; 13.-17.07.2009

35.	Skorupa, W.
	KFKI Budapest, Hungary; 2226.04.2009

- Skorupa, W. SMIT Shanghai, City University Hong Kong, China; 3.-12.05.2009
- 37. Skorupa, W. *Uppsala, Sweden; 5.-9.08.2009*
- Strache, T. Swiss Light Source, PSI Villigen, Switzerland; 16.-25.02., 26.04.-3.05., 6.-10.09., 5.-11.10., 13.-22.12.2009
- 39. Strache, T. Glasgow University, United Kingdom; 11.-15.05., 29.11.-4.12.2009
- 40. Voelskow, M. KFKI Budapest, Hungary; 22.-26.04.2009
- 41. Wintz, S. Swiss Light Source, PSI Villigen, Switzerland; 16.-25.02., 26.04.-3.05., 6.-10.09., 5.-11.10., 13.-18.12.2009
- 42. Wintz, S. Advanced Light Source, Berkeley, USA; 7.-27.05., 26.10.-9.11.2009
- 43. Wintz, S. Canadian Light Source, Saskatoon, Canada; 5.-12.12.2009
- 44. Zschornak, M. DESY Hamburg, Germany; 10.-15.09.2009

### Guests

- 1. Akhmerov, G. Izhevsk State Technical University, Russia; 9.03.-1.05.2009
- 2. Andermann, C. Universite de Rennes, France and TU Bergakademie Freiberg, Germany; 28.09.-17.10.2009
- 3. Arpaci, S. Ankara University, Turkey; 1.07.-31.08.2009
- 4. Avashti, D. K. Inter-University Accelerator Centre, New Delhi, India; 3.-6.06.2009
- 5. Bergmaier, A. Institut für Angewandte Physik, Universität der Bundeswehr, München, Germany; 8.-12.12.2009
- 6. Bhattacharyya, J. Tata Institute of Fundamental Research, Mumbai, India; 7.01.-28.02.2009
- 7. Bichler, M.; Eder, F.; Sterba, J.H. Atominstitut der Österreichischen Universitäten, TU Wien, Austria 15.-18.12.2009
- 8. Bregolin, F. Universidade Federal do Rio Grande do Sul, Departamento de Física, Porto Alegre, Brazil; 1.02.-24.04.2009
- 9. Dzenisevich, S. Yanka Kupala State University, Grodno, Belarus; 31.08.-1.11.2009
- 10. Enyashin, A. Institute of Solid State Chemistry, Ekaterinburg, Russia; 27.05.-1.06.2009
- 11. Gieniusz, R. University of Bialystok, Poland; 30.01.-28.02.2009
- 12. Ionescu, M. Austrian Nuclear Science and Technology Organization, Sydney, Australia; 3.-4.09.2009
- 13. Ivkova, T. Institute of Nuclear Fusion, Kurchatov Institute, Moscow; 5.-26.04.2009
- 14. John, V. Manipal Institute of Technology, Manipal , India; 9.06.-28.07.2009

Khan, S. Inter-University Accelerator Centre, New Delhi, India; 22.0314.05.2009
Kumar, H. Indian Institute of Technology, New Delhi, India; 26.0715.10.2009
Kutschera, W. Fakultät für Physik, Beschleunigerlabor (VERA), Universität Wien, Austria; 30.111.12.2009
Liechtenstein, V. Institute of Nuclear Fusion, Kurchatov Institute, Moscow; 526.04.2009
Li, L. Department of Nuclear Physics and Nuclear Technology, Beijing University , China; 1.1031.12.2009
Mackova, A. Laboratory of Nuclear Analytical Methods, Nuclear Institute of ASCZ, Rez, Czech Republic; 1920.03.2009
Markov, A. Institute of High Current Electronics, Tomsk, Russia; 5.1013.11.2009
Mazalski, P. University Bialystok, Poland; 16.0514.06., 11.0715.08., 18.0911.10.2009
Menendez Dalmau, E. Universidad Autonoma de Barcelona, Spain; 3.086.11.2009
Moiseev, K. Ioffe Physical-Technical Institute, St. Petersburg, Russia; 19.05.2009
Nazarov, A. Institute of Semiconductor Physics, Kiew, Ukraine; 15.067.08.2009
Oates, T. Linköping University, Sweden; 12.106.11.2009
Odor, G. KFKI Budapest, Hungary; 1520.02., 2430.03., 31.1013.11.2009
Om, P.S. Amity Institute of Nanotechnology, Noida, India; 25.0530.07.2009
Ou, X. Shanghai Institute of Microsystems and Information Technology, China; 1.0731.12.2009
Poljotow, J. Izhevsk State Technical University, Russia; 31.081.11.2009
Prucnal, S. University Marie Sklodowska Curie, Lublin, Poland; 1.0330.04.2009
Pryadko, E. Institute of High Current Electronics, Tomsk, Russia; 30.1018.12.2009
Sasaki, T. Tohoku University, Sendai, Japan; 1.0931.10.2009
Shoshan, N. Technion, Haifa, Israel; 1.011.02.2009
Song, J. Advanced Analysis Center, Korea Institute of Science and Technology, Seoul, South Korea; 1920.05.2009
Süle, P. KFKI Budapest, Hungary; 30.0820.09., 25.1013.11.2009
Sundaravel, B. Materials Science Division, Indira Gandhi Centre for Atomic Research, Kalpakkam, India; 1318.09.2009
Tyagulskiy, I. Institute of Semiconductor Physics, Kiew, Ukraine; 15.067.08.2009
Virsek, M. Jozef Stefan Institute, Ljubljana, Slovenia; 1931.01.2009

- 40. Yao, S. School of Microelectronics and Solid-State Electronics, University of Electronic Science and Technology of China , Chengdu, Sichuan, China; 20.09.-31.12.2009
- 41. Ye, S. Universität Duisburg-Essen, Germany; 10.-17.02.2009
- 42. Yilgin, R. Gebze Institute of Technology, Department of Physics, Gebze-Kocaeli, Turkey; 16.06.-25.11.2009
- 43. Zhou, J. University Shanghai, China; 1.01.-9.10.2009

### AIM Visitors

- 1. Abrasheva, M. *University of Sofia, Bulgaria;* 22.02.-4.03.2009
- Barlak, M. Soltan Institute for Nuclear Studies, Otwock, Poland; 22.-27.11.2009
- 3. Barquinha, P. CENIMAT Lisboa, Portugal; 26.-31.07.2009
- 4. Bugoi, R. Institute for Nuclear Physics, Bucharest, Romania; 6.-13.12.2009
- 5. Caciolli, A. INFN Padova, Italy; 1.-14.03., 18.-22.10.2009
- 6. Capogrosso, V. INFN Padova, Italy; 8.-13.03.2009
- 7. Constantinescu, B. Institute for Nuclear Physics, Bucharest, Romania; 6.-13.12.2009
- 8. Dekov, V. University of Sofia, Bulgaria; 22.02.-4.03.2009
- 9. Erhard, M. INFN Padova, Italy; 22.02.-7.03.2009
- 10. Franz, R. University Leoben, Austria; 31.03.-3.04.2009
- 11. Gomez, P.R. University Sevilla, Spain; 30.10.-6.11.2009
- 12. Gösselsberger, C. TU Vienna, Austria; 25.01.-7.02., 26.04.-1.05.2009
- 13. Gonzalez Arrabal, R. Universidad Politécnica de Madrid, Spain; 13.-19.12.2009
- 14. Igreja, R. CENIMAT Lisboa, Portugal; 26.-31.07.2009
- 15. Kotb, H. IEMT Warsaw, Poland; 23.-30.05.2009
- 16. Martins, R. INT Sacavem, Portugal; 3.-19.12.2009
- 17. Meissl, W. *TU Vienna, Austria;* 25.01.-7.02.2009
- 18. Nasdala, L. University Vienna, Austria; 6.-9.04.2009
- 19. Pagowska, K. IEMT Warsaw, Poland; 6.-15.01.2009
- 20. Preuschl, F. University Vienna, Austria; 6.-9.04.2009

- 21. Sartowska, N. Soltan Institute for Nuclear Studies, Otwock, Poland; 22.-27.11.2009
- 22. Sicinski, M. Unversity Lodz, Poland; 12.-15.10.2009
- 23. Stonert, A. IEMT Warsaw, Poland; 23.-30.05.2009
- 24. Turos, A. IEMT Warsaw, Poland; 6.-15.01., 2.-4.03.2009
- 25. Vasko, C. TU Vienna, Austria; 26.04.-1.05.2009
- 26. Winklehner, D. *TU Vienna, Austria; 6.-17.07.2009*

### FEL Visitors

- 1. Aleksa, V. Vilnius University, Lithuania; 5.-11.04., 6.-10.05.2009
- 2. Balocco, C. Manchester University, United Kingdom; 6.-9.02., 28.02.-1.03.2009
- 3. Kasjoo, S.R. Manchester University, United Kingdom; 6.-9.02., 28.02.-5.03., 23.-27.07., 11.-14.08.2009
- 4. Lu, X. Manchester University, United Kingdom; 28.02.-1.03.2009
- 5. Orlita, M. High Field Laboratory Grenoble, France; 20.-25.09.2009
- 6. Porter, N. University of Sheffield, United Kingdom; 27.-31.01., 25.02.-1.03.2009
- 7. Sablinskas, V. Vilnius University, Lithuania; 5.-11.04., 6.-10.05.2009
- 8. Sandall, I. University of Sheffield, United Kingdom; 27.-31.01., 25.02.- 1.03.2009
- 9. Song, A. M. Manchester University, United Kingdom; 6.-8.02., 23.-25.07.2009
- 10. Vaskevicius, K. Vilnius University, Lithuania; 5.-11.04.2009

### **ROBL-MRH** Visitors

- 1. Bartels, M. Institut für Röntgenphysik, Georg-August Universität Göttingen, Germany; 21.-24.02.2009
- Bayer, B. Department of Engineering, University of Cambridge, United Kingdom; 22.-28.04.2009
- 3. Biermanns, A. Fachbereich Physik, Universität Siegen, Germany; 31.01.- 3.02., 5.-10.07.2009
- 4. Birch, J. Department of Physics, Chemistry and Biology, Linkoeping University, Sweden; 6.-12.05.2009
- 5. Braz Fernades, F. CENIMAT - Materials Science Department, Caparica, Portugal; 23.-27.01.2009
- 6. Buljan, M. Department of Condensed Matter Physics, Charles University Praha, Czech Republic; 15.–21.04.; 9.–15.12.2009

7.	Dopita, M. Institute of Materials Science, Technische Universität Bergakademie Freiberg, Germany; 2831.01., 2127.10.2009
8.	Gaca, J. Institute of Electronic Materials Technology, Warsaw, Poland; 10.–14.07.2009
9.	Hoefer, S. Fachbereich Röntgenoptik, Friedrich-Schiller Universität Jena, Germany; 18.–21.02.2009
10.	Höglund, C. Department of Physics, Chemistry and Biology, Linkoeping University, Sweden; 612.05., 1621.12.2009
11.	Holy, V. Department of Condensed Matter Physics, Charles University Praha, Czech Republic; 15.–21.04.; 9.–15.12.2009
12.	Karimbi Koosappa, M. CENIMAT - Materials Science Department, Caparica, Portugal; 23 27.01.2009
13.	Kalinichenka, S. Fraunhofer Institut IFAM, Dresden, Germany; 18.–22.09.2009
14.	Kräußlich, J. Fachbereich Röntgenoptik, Friedrich Schiller Universität Jena, Germany; 18.–21.02.2009
15.	Krüger, S Institut für Röntgenphysik, Georg-August Universität Göttingen, Germany; 21 -24.02.2009
16.	Lechner, R. Institute of Physics, University of Leoben, Austria, 27.02.–3.03.; 23.–29.09.2009
17.	Mazur, K. Institute of Electronic Materials Technology, Warsaw, Poland; 10.–14.07.2010
18.	Meduna, M. Institute of Condensed Matter Physics, Masaryk University Brno, Czech Republic; 27.02.–3.03.2009
19.	Motylenko, M. Institute of Materials Science, Technische Universität Bergakademie Freiberg, Germany; 2831.01., 2127.10.2009
20.	Navarro Quezada, A. Institut für Halbleiterphysik, Johannes Kepler Universität Linz, Austria; 27.02.–3.03.; 23.– 29.09.2009
21.	Neubauer, H. Institut für Röntgenphysik, Georg-August Universität Göttingen, Germany; 2124.02.2009
22.	Radic, N. Materials Physics, Rudjer Boskovic Institute, Zagreb, Croatia; 9.–15.12.2009
23.	Röntzsch, L. Fraunhofer Institut IFAM, Dresden, Germany; 18.–22.09.2009
24.	Rovezzi, M. Institute of Semiconductor Physics, Johannes Kepler University Linz, Austria; 27.02.– 3.03.; 23.–29.09.2009
25.	Silva Cordeiro, R. CENIMAT - Materials Science Department, Caparica, Portugal; 2327.01.2009
26.	Vales, V. Department of Condensed Matter Physics, Charles University Praha, Czech Republic; 15.–21.04.2009
27.	Wirth, C. Department of Engineering, University of Cambridge, United Kingdom; 2228.04.2009
28.	Wojcik, M. Institute of Electronic Materials Technology, Warsaw, Poland; 10.–14.07.2009
29.	Wüstefeld, C. Institute of Materials Science, Technische Universität Bergakademie Freiberg, Germany; 2831.01., 2127.10.2009
30.	Zastrau, U.

## Colloquium

- Aumayr, F. Institut f
  ür Allgemeine Physik, TU Wien, Austria Interaction of slow highly-charged ions with solid surfaces 29.06.2009
- 2. van den Berg, J. Joule Physics Laboratory and Institute for Materials Research, University of Salford, United Kingdom

Medium energy ion scattering (MEIS) for the high depth resolution characterisation of ultra shallow implant effects and high k-dielectric gate oxide layers 10.12.2009

- Golser, R. Fakultät für Physik Isotopenforschung, TU Wien, Austria Beschleunigermassenspektrometrie und Ionenstrahlanalyse bei VERA 4.06.2009
- Grützmacher, D. Institut für Bio- und Nanosysteme (IBN-1), Forschungszentrum Jülich, Germany Semiconductor nanostructures: On the search for ultimately scaled devices 5.03.2009
- Jörg, G. Institut für Radiochemie, TU München, Germany Determination of long-lived radionuclides, which are relevant for final disposal, by radiometric and sophisticated mass-spectrometic methods 16.04.2009
- Lerch, W. Centrotherm thermal solutions GmbH, Blaubeuren, Germany Advanced activation strategies for boron and arsenic by combinations of single, multiple flash anneals and spike rapid thermal annealing 24.09.2009
- Masselink, T. Institut f
  ür Physik, Humboldt Universit
  ät Berlin, Germany Strained epitaxy for high-performance mid-infrared quantum-cascade lasers 9.07.2009
- McCord, J. Leibniz Institut f
  ür Festk
  örper- und Werkstoffforschung Dresden, Germany Aspects of magnetic heterostructures - statics and dynamics 19.11.2009
- Michel, R. Zentrum f
  ür Strahlenschutz und Radioökologie, Leibnitz Universit
  ät Hannover, Germany Untersuchung langlebiger Radionuklide in der Umwelt mittels AMS: Aktuelle Entwicklungen und Anwendungen in den Umweltwissenschaften 11.06.2009
- Schneider, C. M. Institut f
  ür Festk
  örperforschung, Forschungszentrum J
  ülich, Germany Magnetism and sprintronics: Research activities at the IFF in J
  ülich 2.04.2009
- Schreyer, A. Institute for Materials Research, GKSS Research Center, Germany Material science with photons and neutrons at GKSS 5.11.2009
- Schroer, C. Institut f
  ür Strukturphysik, TU Dresden, Germany Microscopy and tomography using hard X-rays 8.10.2009
- Schubert, M. Department of Electrical Engineering and Center for Materials Research and Analysis, University of Nebraska-Lincoln, USA
   Generalized terahertz to ultraviolet ellipsometry in nanoscience: Optical, electrical and structrural properties of glancing angle deposited hybrid nanostructures 13.08.2009
- Tautz, S. Institut f
  ür Bio-und Nanosysteme (IBN-3), Forschungszentrum J
  ülich, Germany Organic molecules on metals: From surface science to molecular electronics 17.12.2009
- Timm, C. Institut f
  ür Theoretische Physik, TU Dresden, Germany Magnetism and transport: From molecules to semiconductors 14.05.2009

 Toulemonde, M. - Center of Research on Ions Materials and Photonics at GANIL, Caen, France Surface material modifications by slow and high charge ions: Role of the electron-phonon coupling 29.01.2009

### FZD Materials Science Seminar

- Erbe, A. Colloids: Mesoscopic model systems for matter on the nanoscale 16.12.2009
  - Heera, V. Superconductivity in Ga doped Ge 20.10.2009
- Heinig, K.-H. Nanodots, nanowires, surface pattern –Atomistic simulations in materials science 23.02.2009
- Kanjilal, A. Interplay between the microstructure and the optical properties of Er-doped Ge-rich SiO<sub>2</sub> layers 17.11.2009
- Krause-Rehberg, R. Materials research with positrons - The EPOS Project at FZD 21.04.2009
- Rebohle, L. Rare earth implanted MOS light emitting devices 17.03.2009
- Kögler, R. Silicon-on-insulator (SOI) materials: Intention, production, prospects 8.09.2009

### **IIM Seminars**

- Giraud, R. Laboratoire de Photonique et de Nanostructures, Marcoussis, France Quantum spin transport in GaMnAs nanomagnets 8.05.2009
- Grebing, J. Universität Konstanz, Germany Elektronische Transportmessungen durch Si4-Cluster 1.04.2009
- 3. Heine, T. School of Engineering and Science, Jacobs University, Bremen, Germany Computational science on the nanoscale: Nanoelectromechanics and nanopores 5.10.2009
- Kelling, J. TU Dresden, Germany NVIDIA-Grafikkarten zum parallelen Rechnen von Monte-Carlo-Simulationen 29.09.2009
- Kirchner, T. Universität Konstanz, Germany Electrical conduction through single molecule junctions 8.07.2009
- Ling, F. C. C. Department of Physics, The University of Hong Kong, China p-type doping of ZnO by ion implantation and radio frequency magnetron sputtering 11.08.2009
- Löser, M. Institut für Angewandte Physik, Universität Münster, Germany Fasergestützte Repetitionsratenstabilisierung eines diodengepumpten Ultrakurzpuls-Erbium-Faserlasers 9.01.2009
- Meier, G. Universität Hamburg, Germany Current-induced magnetization dynamics: x-ray microscopy and rf-transport experiments 26.10.2009

2.

- Moiseev, K. Ioffe Physical-Technical Institute of the Russion Academy of Science, St. Petersburg, Russia Low-dimensional nanoobjects in narrow-gap type II antymonide-based heterostructures 6.05.2009
- Nazarov, A.N. Lashkaryov Institute of Semiconductor Physics, National Academy of Sciences of Ukraine, Kiev, Ukraine
   Study of amorphous Si<sub>x</sub>C<sub>1-x</sub>:H and Si<sub>x</sub>C<sub>y</sub>O<sub>z</sub>:H films on crystalline silicon for light-emission 7.07.2009
- Oesterlin, P. INNOVAVENT GmbH, Göttingen, Germany Laser-Annealing mit frequenzverdoppelten Festkörperlasern: Anwendungen und Technologie 26.2.2009
- Przybylski, M. MPI für Mikrostrukturphysik Halle, Germany Oscillatory magnetic anisotropy originating from quantum well states in Fe films 5.10.2010
- Reichel, D. TU Dresden, Germany T-Messung bei Kurzzeittemperung: Eine Übersicht 17.02.2009
- Sadewasser, S. Humboldtzentrum Berlin, Germany Kelvin probe force microscopy on semiconductors 2.11.2009
- Sharma, R. MPI for the Science of Light, University Erlangen-Nürnberg, Germany Electrodynamically confined microscale lasers 21.09.2009
- Villis, B.J. School of Physics, University of Melbourne, Australia The electrical and optical properties of defects in ion implanted silicon 26.11.2009
- Vogel, A. Universität Hamburg, Germany Enhanced spin injection and detection in spin valves with integrated tunnel barriers 1.07.2009
- Wiesenhütter, U. IFW Dresden, Germany Hochauflösende Transmissionselektronenmikroskopie an Übergangsmetall-Partikeln 30.06.2009
- Windl, W. Materials Science and Engineering Department, Ohio State University, USA Ab-initio calculations for silicon as spintronics material 29.07.2009
- Zeh, C. TU Dresden, Germany Interferometrie mit polarisationserhaltenden Glasfasern und Anwendungen auf die streuende optische Nahfeldmikroskopie 4.02.2009

### Projects

1.	03/2004 - 02/2009 <b>IA-SFS - Integrating activ</b> Prof. M. Helm	European Union vity on synchrotron and free ex Tel.: 0351 260-2260	ectron laser science m.helm@fzd.de	EU
2.	11/2004 – 12/2009 <b>Hochenergie-Ionenimpla</b> Dr. J. von Borany	Silicon Sensor Berlin GmbH <b>intation für optische Sensoren</b> Tel.: 0351 260 3378	j.v.borany@fzd.de	Industry
3.		European Union action of massively parallel in malysis and synthesis (PRON Tel.: 0351 260 2726	<b>e</b>	EU
4.	06/2005 – 03/2010 <b>Mössbauerspektroskopie</b> Dr. H. Reuther	Deutsche Forschungsgemeins <b>an ionenimplantierten magn</b> Tel.: 0351 260 2898		DFG
5.	09/2005 – 02/2010 Fullerene-based opportu	European Union nities for robust engineering:		EU

	<b>Making optimised surfa</b> Prof. A. Kolitsch	<b>ces for tribology (FOREMOS</b> Tel.: 0351 260 3348	Г) a.kolitsch@fzd.de	
6.	01/2006 – 06/2010 <b>Ion technology and spec</b> Dr. S. Facsko	European Union <b>troscopy at low energy ion be</b> Tel.: 0351 260 2987	am facilities (ITS-LEIF) s.facsko@fzd.de	EU
7.	04/2006 – 03/2010 <b>Center for application of</b> Prof. A. Kolitsch	European Union <b>ion beams to materials resea</b> Tel.: 0351 260 3348	<b>rch (AIM)</b> a.kolitsch@fzd.de	EU
8.		Deutsche Forschungsgemeir metallschichten auf Piezosub -Forschergruppe 520: Ferroiso Tel.: 0351 260 2470	straten	DFG
9.	12/2006 – 12/2009 Durchführung von REM Untersuchungen an Met Dr. H. Reuther	VKTA e.V. Dresden - <b>bzw. EDX- sowie Mössbaue</b> allproben Tel.: 0351 260 2898	r <b>-spektroskopischen</b> h.reuther@fzd.de	Bilateral
10.	04/2007 – 03/2009 <b>Unterdrückung der Saue</b> Prof. A. Kolitsch	Arbeitsgemeinschaft industr r <b>stoffversprödung von Titan</b> Tel.: 0351 260 3348	ieller Forschungsvereinigungen l <b>egierungen</b> a.kolitsch@fzd.de	AiF
11.	04/2007 – 03/2009 <b>Ion-beam induced rippli</b> Dr. J. Grenzer	Deutsche Forschungsgemeir <b>ng at the amorphous-crystall</b> Tel.: 0351 260 3389		DFG
12.	05/2007 – 06/2009 <b>Strukturübergänge eing</b> Dr. K. Potzger	Deutsche Forschungsgemeir Ebetteter magnetischer Nanop Tel.: 0351 260 3148		DFG
13.	07/2007 – 01/2013 <b>Hybride Magnetische M</b> Prof. J. Faßbender	Deutsche Forschungsgemeir <b>aterialien</b> Tel.: 0351 260 3096	ischaft j.faßbender@fzd.de	DFG
14.	08/2007 - 07/2009 Grenzen des Halogeneff industriellen Bedingung Prof. A. Kolitsch	ektes für TiAl-Hochtemperat	ieller Forschungsvereinigungen <b>urleichtbaulegierungen unter</b> a.kolitsch@fzd.de	AiF
15.	08/2007 – 11/2010 <b>Nanostrukturierung von</b> Dr. S. Facsko	Deutsche Forschungsgemeir <b>Oberflächen mit direkter Ex</b> Tel.: 0351 260 2987	ischaft t <b>raktion der Ionen aus Plasmaquellen</b> s.facsko@fzd.de	DFG
16.		Deutsche Forschungsgemeir strukturen durch niederenerg Tel.: 0351 260 3288		DFG
17.	08/2007 - 08/2010 <b>Ferromagnetism in trans</b> Dr. H. Schmidt	Deutsche Forschungsgemeir <b>ition metal doped ZnO</b> Tel.: 0351 260 2724	schaft heidemarie.schmidt@fzd.de	DFG
18.	09/2007 – 08/2009 <b>TEMPUS courses of mat</b> Prof. W. Möller	European Union <b>erials science</b> Tel.: 0351 260 2245	w.moeller@fzd.de	EU
19.	09/2007 – 10/2010 Infrared scattering near- resonances using a free- Prof. M. Helm	Deutsche Forschungsgemeir field optical microscopy near electron laser Tel.: 0351 260 2260		DFG
20.	10/2007 – 08/2009 Gastaufenthalt Herr Dr. Prof. M. Helm	Alexander-von-Humboldt-S <b>A. Kanjilal</b> Tel.: 0351 260 2260	tiftung m.helm@fzd.de	AvH
21.	10/2007 - 09/2009 <b>Haifischhaut</b> Prof. A. Kolitsch	Dechema Tel.: 0351 260 3348	a.kolitsch@fzd.de	Bilateral

22.	11/2007 – 10/2010 Technologietransfer c-Bl			Industry
	Prof. A. Kolitsch	Tel.: 0351 260 3348	a.kolitsch@fzd.de	
23.	12/2007 – 01/2009 NanoAnalytik	Qimonda Dresden GmbH		Industry
	Dr. J. von Borany	Tel.: 0351 260 3378	j.v.borany@fzd.de	
24.	01/2008 - 01/2009 Laserannealing	IXYS Semiconductor GmbH	· 1	Industry
	Dr. J. von Borany	Tel.: 0351 260 3378	j.v.borany@fzd.de	
25.	01/2008 – 12/2009 <b>Projektbezogener Persor</b> Dr. W. Skorupa	Deutscher Akademischer Au nenaustausch mit Hong Kong Tel.: 0351 260 3612	stauschdienst w.skorupa@fzd.de	DAAD
26.	1 1	Deutscher Akademischer Au	stauschdienst	DAAD
	<b>Projektbezogener Persor</b> Dr. KH. Heinig	nenaustausch mit Ungarn Tel.: 0351 260 3288	k.h.heinig@fzd.de	
27.	Bildung, Stabilität und S	Deutsche Forschungsgemein Struktur nanoskaliger Phasen f die Materialwissenschaft (C Tel.: 0351 260 2470	in funktionellen Oxiden	DFG
28.	01/2008 – 12/2009 <b>Polarisationsinduzierte (</b> Dr. S. Gemming	Deutsche Forschungsgemein: Dberflächenfunktionalität an Tel.: 0351 260 2470		DFG
29.	02/2008 - 02/2010	Deutsche Forschungsgemein l <b>en an strukturierten Fluid-Fe</b> Tel.: 0351 260 2470	schaft	DFG
30.	05/2008 – 04/2010 Blitztemperung 200	FHR Anlagenbau Ottendorf-	Okrilla/IHP Frankfurt/O.	Industry
	Dr. W. Skorupa	Tel.: 0351 260 3612	w.skorupa@fzd.de	
31.	07/2008 – 12/2010 WTZ Russland: Duplexb Prof. A. Kolitsch	Bundesministerium für Bildu ehandlung rostfreier Stähle Tel.: 0351 260 3348	ng und Forschung a.kolitsch@fzd.de	BMBF
32.	07/2008 - 06/2011	Deutsche Forschungsgemein magnetische Anisotropie- un Tel.: 0351 260 3096		DFG
33.	07/2008 - 07/2011 Multifunktionale Speich			BMBF
	Prof. J. Faßbender	Tel.: 0351 260 3096	j.faßbender@fzd.de	
34.	International Conference	Deutsche Forschungsgemeine on Nonequilibrium Nanostr	uctures	DFG
0.5	Dr. K. Morawetz	Tel.: 0351 260 2470	k.morawetz@fzd.de	T 1 4
35.	09/2008 – 06/2009 <b>TEM-Untersuchungen</b> Dr. J. von Borany	AMD Tel.: 0351 260 3378	j.v.borany@fzd.de	Industry
36	09/2008 - 12/2010	TU Dresden	j.v.boluly sizu.de	
50.			mit Wachstumsproteinen (BIOMIN) s.gemming@fzd.de	BMBF
37.	11/2008 – 02/2009 Ionenimplantation BASI	BASF Ludwigshafen		Industry
	Prof. A. Kolitsch	Tel.: 0351 260 3348	a.kolitsch@fzd.de	
38.	11/2009 – 03/2010 <b>Abscheidung hochhafter</b> Prof. A. Kolitsch	Burgmann Industries Wolfra nder TiN-Hartstoffschichten Tel.: 0351 260 3348	a.kolitsch@fzd.de	Industry
39.		KETEK GmbH München		Industry
~ ~ *	<b>Wafer-Prozessierung</b> Dr. B. Schmidt	Tel.: 0351 260 2726	bernd.schmidt@fzd.de	y

40.	01/2009 - 12/2010 <b>Projektbezogener Person</b> Prof. J. Faßbender	Deutscher Akademischer Aus enaustausch mit Polen Tel.: 0351 260 3096	itauschdienst j.faßbender@fzd.de	DAAD
41.	02/2009 – 12/2009 Magnetismus von nanosl eingeschränkten Dimens Prof. J. Faßbender		chaft <b>n templatbedingt geometrisch</b> j.faßbender@fzd.de	DFG
42.	02/2009 – 12/2009 <b>Speicherprogrammierte</b> S Dr. J. von Borany	Johannes Kepler Universität I <b>Steuerung</b> Tel.: 0351 260 3378	.inz j.v.borany@fzd.de	Bilateral
43.	, ,	Sächsisches Staatsministeriun ungsinfrastruktur mit Mitteln		SMWK
44.	Prof. A. Kolitsch 03/2009 – 02/2013 <b>Support of Public and In</b> Prof. W. Möller	Tel.: 0351 260 3348 European Union <b>dustrial Research using Ion B</b> Tel.: 0351 260 2245	a.kolitsch@fzd.de eam Technology (SPIRIT) w.moeller@fzd.de	EU
45.	03/2009 – 08/2011 <b>European Light Sources</b> A Prof. M. Helm	European Union <b>Activities: Synchrotrons and F</b> Tel. 0351 260 2260	<b>ree Electron Lasers (ELISA)</b> m.helm@fzd.de	EU
46.	05/2009 - 04/2010 <b>Gastaufenthalt Frau Dr. J</b> Dr. H. Schneider	Alexander-von-Humboldt-Sti J. <b>Bhattacharyya</b> Tel.: 0351 260 2880	ftung h.schneider@fzd.de	AvH
47.	05/2009 - 04/2011 <b>FZD-Applikationslabor I</b> Prof. A. Kolitsch		Verkehr und Stadtentwicklung a.kolitsch@fzd.de	BMBVS
48.	Rezeptor-selektive Anrei		ng und Forschung <b>ik zur qualitativen und quantitativen</b> i <b>rkenden Substanzen (Bio-LED-Sensorik)</b> l.rebohle@fzd.de	BMBF
49.	07/2009 - 12/2009 <b>TEM-Untersuchungen</b> Dr. J. von Borany	GLOBALFOUNDRIES Dresder Tel.: 0351 260 3378	en j.v.borany@fzd.de	Industry
50.	07/2009 - 06/2011	Deutscher Akademischer Aus enaustausch mit Großbritann Tel.: 0351 260 3096	stauschdienst ien	DAAD
51.	07/2009 - 06/2011 <b>Eigenschaften nano- und</b> Dr. W. Skorupa	TU Bergakademie Freiberg <b>mikrokristalliner Si-Dünnfil</b> Tel.: 0351 260 3612	<b>me</b> w.skorupa@fzd.de	Bilateral
52.	09/2009 - 02/2011 <b>Gastaufenthalt Herr Dr.</b> A Dr. S. Facsko	Alexander-von-Humboldt-Sti <b>A.S. El-Said</b> Tel.: 0351 260 2987	ftung s.facsko@fzd.de	AvH
53.	09/2009 – 08/2012 Bestimmung der lagespe	Deutsche Forschungsgemeins zifischen und räumlich aufge		DFG
54.	11/2009 - 04/2010	Wilhelm- und Else-Heraeus-S <b>:per-DFT an der RWTH Aache</b> Tel.: 0351 260 2470	tiftung	WEH

# **Experimental Equipment**

### 1. Accelerators, Ion Implanters and Ion-Assisted-Deposition

$\Rightarrow$	Van de Graaff Accelerator	(VdG)	1.8 MV	TuR Dresden, DE
$\Rightarrow$	Tandem Accelerator	(Td)	5 MV	NIIEFA, RU
$\Rightarrow$	Tandetron Accelerator	(Tdt)	3 MV	HVEE, NL
$\Rightarrow$	Tandetron Accelerator (commissionin	lg)	6 MV	HVEE, NL
$\Rightarrow$	Low-Energy Ion Implanter	0,	0.5 - 50 kV	Danfysik, DK
$\Rightarrow$	High-Current Ion Implanter		20 - 200 kV	Danfysik, DK
$\Rightarrow$	High-Energy Ion Implanter		40 - 500 kV	HVEE, NL
$\Rightarrow$	Plasma Immersion Ion Implantation		5 - 60 keV	GBR, DE/Home-built
$\Rightarrow$			30 keV, 10 A/cm <sup>2</sup>	Orsay Physics, FR
$\Rightarrow$	Highly-Charged Ion Facility	,	25 eV – 25 keV × Q	Home-built
			Q = 140 (Xe)	
$\Rightarrow$	Dual-Beam Magnetron Sputter Depos	sition		Roth & Rau, DE
$\Rightarrow$				Danfysik, DK
$\Rightarrow$	Ion-Beam Sputtering		200 - 2000 V	Home-built
$\Rightarrow$	UHV Ion Irradiation (Ar, He, etc.)		0 - 5 keV	VG, USA
			Scan 10×10 mm <sup>2</sup>	

### 2. Ion Beam Analysis (IBA)

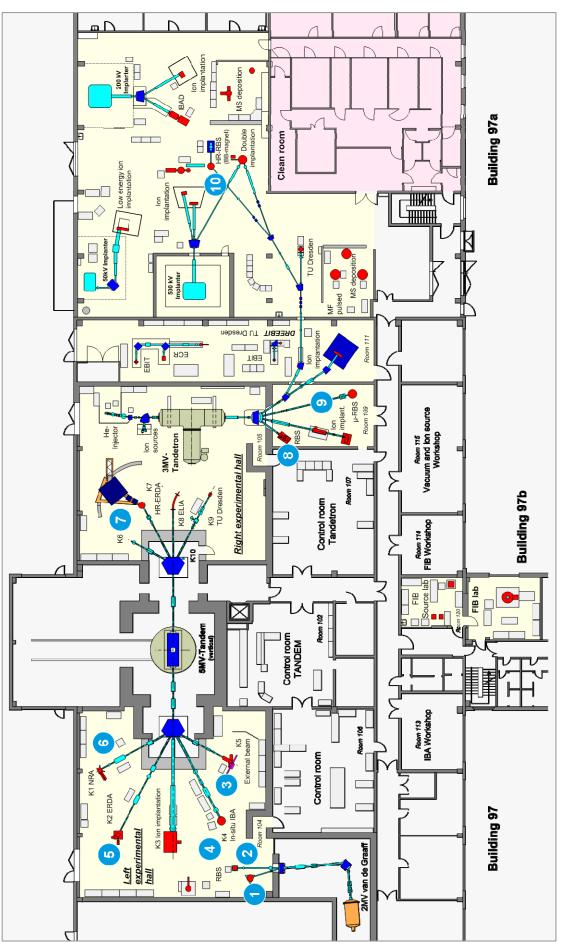
A wide variety of advanced IBA techniques are available at the MeV accelerators (see figure).

$\Rightarrow$	RBS	Rutherford Backscattering	(1), (2), (3), (8)	VdG, Td, Tdtr
$\Rightarrow$	RBS/C	RBS + Channelling	(1), (2), (3), (8)	VdG, Td, Tdtr
		High-Resolution RBS/C	(7), (10)	Tdtr
$\Rightarrow$	ERDA	Elastic Recoil Detection Analysis	(2), (4), (5)	VdG, Td
		High-resolution ERDA	(7)	Td
$\Rightarrow$	PIXE	Proton-Induced x-ray Emission	(3)	Td
$\Rightarrow$	PIGE	Proton-Induced γ Emission	(3)	Td
$\Rightarrow$	NRA	Nuclear Reaction Analysis	(4)	Td
$\Rightarrow$	NRRA	Nuclear Resonance Reaction Analysis	(6)	Td
$\Rightarrow$	ΝμΡ	Nuclear Microprobe	(9)	Tdtr
$\Rightarrow$	AMS	Accelerator Mass Spectrometry	(commissioning)	
		(focused to cosmogenic radionuclides:	<sup>10</sup> Be, <sup>26</sup> Al, <sup>36</sup> Cl, <sup>41</sup> C	a, <sup>129</sup> J

Some stations are equipped with additional process facilities which enable *in-situ* IBA investigations during ion irradiation, sputtering, deposition, annealing etc.

### 3. Other Particle Based Analytical Techniques

$\Rightarrow$	SEM	Scanning Electron Microscope	1 - 30 keV	Hitachi, JP
			+ EDX	
$\Rightarrow$	TEM	Transmission Electron Microscope	80 - 300 keV	FEI, NL
		(Titan 80-300 with Image Corrector)	+ EDX, +GIF	
$\Rightarrow$	FIB/SEM	Focused Ion / Electron Cross Beam	0.5 <b>-</b> 30 keV	Zeiss-NTS, DE
		(NVision 40 with Elphy Plus Litho)	+ IL, + EDX	Raith, Bruker, DE
$\Rightarrow$	AES	Auger Electron Spectroscopy	+ XPS	Fisions, UK
$\Rightarrow$	CEMS	Mössbauer Spectroscopy	<sup>57</sup> Fe source	Home-built
$\Rightarrow$	PAS	Positron Annihilation Spectroscopy	<sup>22</sup> Na source	Home-built
			30 V - 36 kV	



### 4. Photon Based Analytical Techniques

		- –	
$\Rightarrow$	XRD/XRR	X-Ray Diffraction and Reflection	Cu-K <sub>a</sub>
	HR-XRD	High-Resolution XRD	Cu-K <sub>a</sub>
$\Rightarrow$	XRD/XRR	with Synchrotron Radiation	5 – 35 keV
$\Rightarrow$	SE	Spectroscopic Ellipsometry	250 - 1700 nm
$\Rightarrow$	FTIR	Fourier-Transform Infrared Spectr.	600 - 7000 cm <sup>-1</sup>
$\Rightarrow$	FTIR	Fourier-Transform Infrared Spectr.	50 - 15000 cm <sup>-1</sup>
$\Rightarrow$		Ti:Sapphire Femtosecond Laser	
$\Rightarrow$		Femtosecond Optical Parametric Osci.	
$\Rightarrow$		Ti:Sapphire Femtosecond Amplifier	
$\Rightarrow$		Femtosecond Optical Parametric Amp	lifier
$\Rightarrow$	THz-TDS	Terahertz Time-Domain Spectroscopy	0.1 - 4 THz
$\Rightarrow$	Raman	Raman Spectroscopy	45 cm <sup>-1</sup> shift
$\Rightarrow$	PL	Photoluminescence	300 - 1500 nm
$\Rightarrow$	TRPL	Time-Resolved PL	$\tau = 3 \text{ ps} - 2 \text{ ns}$
			$\tau > 5 \text{ ns}$
$\Rightarrow$	EL	Electroluminescence (10-300 K)	300 - 1500 nm
		Optical Split-Coil Supercond. Magnet	7 T
$\Rightarrow$	PR	Photomodulated Reflectivity	300 - 1500 nm
$\Rightarrow$	PLE	Photoluminescence Excitation	300 - 1500 nm

### 5. Magnetic Thin Film Deposition and Analysis

$\Rightarrow$	MBE	Molecular Beam Epitaxy with in-situ F	ΤB
$\Rightarrow$	MBE	Molecular Beam Epitaxy	
$\Rightarrow$	PLD	Pulsed Laser Deposition	
$\Rightarrow$	MFM	Magnetic Force Microscope	$\sim$ 50 nm resol.
$\Rightarrow$	SQUID	Supercond. Quantum Interf. Device	±7 T
$\Rightarrow$	MOKE	Magneto-Optic Kerr Effect (in-plane)	$\pm 0.35 \text{ T}$
$\Rightarrow$	MOKE	Magneto-Optic Kerr Effect (perpend.)	± 2 T
$\Rightarrow$	SKM	Scanning Kerr Microscope	
$\Rightarrow$		Kerr Microscope	
$\Rightarrow$	TR-MOKE	Time-Resolved MOKE (Pump-Probe)	
$\Rightarrow$	VNA-FMR	Vector Network Analyzer Ferromagne	etic Resonance
$\Rightarrow$	ME	Magnetoellipsometer	

### 6. Other Analytical and Measuring Techniques

$\Rightarrow$	STM	Scanning Tunneling Microscope (with	DME, DK	
$\Rightarrow$	STM	In-situ Scanning Tunneling Microscop	e (T variable)	Omicron, DE
$\Rightarrow$	AFM	Atomic Force Microscope (Tapping M	lode)	SIS, DE
$\Rightarrow$	AFM	Atomic Force Microscope (with c-AFM	M, SCM-Module)	Veeco Instruments, UK
$\Rightarrow$	KFM	Kelvin Probe Force Microscopy		Anfatec, DE
$\Rightarrow$		Dektak Surface Profilometer		Veeco, US
$\Rightarrow$		Micro Indenter / Scratch Tester		Shimatsu, JP
$\Rightarrow$		Wear Tester (pin-on disc)		Home-built
$\Rightarrow$	HE	Hall Effect Equipment	(2 <b>-</b> 400 K, ≤ 9 T)	LakeShore, US
$\Rightarrow$	RS	Sheet-Rho-Scanner		AIT, South Korea
$\Rightarrow$	DLTS	Deep Level Transient Spectroscopy	(+ I-U / C-V)	PhysTech, DE
			(10 - 300 K, 1 MHz	2)
$\Rightarrow$		Photocapacitance (+I-U/G-V)	(250 - 2500 nm)	Home-built
$\Rightarrow$		I-V and C-V Analyzer		Keithley, US

Bruker axs, DE GE Inspection, DE ROBL at ESRF, FR Woolam, US Nicolet, US Bruker, DE

Spectra Physics, US

Jobin-Yvon-Horiba, FR Jobin-Yvon-Horiba, FR Hamamatsu Phot., JP Stanford Research, US Jobin-Yvon-Horiba, FR Jobin-Yvon-Horiba, FR Jobin-Yvon-Horiba, FR

Femtolasers, AT Light Conversion, LI

APE, DE

Home-built

CreaTec, DE Home-built SURFACE, DE VEECO / DI, US Quantum Design, US

Home-built Home-built Home-built

Home-built

Evico Magnetics, DE

Agilent / Home-built LOT, DE; AMAC, US

112			E	xperimental Equipment
$\stackrel{\Rightarrow}{\Rightarrow}$	I-V and C-V Semi-A I-V Prober	utomatic Prober	(-60 – 300°C) (4.2 – 600 K)	Süss, DE; Keithley, US LakeShore, Agilent, US
7.	Processing and Preparation	n Techniques		
$\Rightarrow$	Physical Deposition	Sputtering DC / R Electron Beam Eva Thermal Evaporati	poration System	Nordiko, UK Leybold Optics, DE Bal-Tec, LI
$\Rightarrow$	Chemical Deposition	Plasma Enhanced (for a-Si, SiO <sub>2</sub> , SiOl	CVD	Oxford Instruments, UK
$\Rightarrow$	Dry Etching	Plasma and RIE M	ode	Sentech, DE
$\Rightarrow$	Reactive Ion Beam Etching	Ø 6", Ar, CF <sub>4</sub>		Roth & Rau, DE
$\Rightarrow$	Etching / Cleaning	incl. Anisotropic Se	elective KOH Etchir	ıg
$\Rightarrow$	Photolithography	Mask-Aligner, 2 µr	n-level	Süss, DE
$\Rightarrow$	Electron Beam Lithography	Raith 150-TWO: Ø	06", 10 nm res.	Raith, DE
$\Rightarrow$	Thermal Treatment	Room Temperatur	e - 2000°C	
	• Furnace			InnoTherm, DE
	<ul> <li>Rapid Thermal Annealing</li> </ul>			ADDAX, FR
	• Flash-Lamp Units (0.5 – 20 ms)			Home-built; FHR, DE
	• RF Heating (Vacuum)			JIP.ELEC, FR
$\Rightarrow$	Bonding Techniques	Ultrasonic Wire Bo	onding	Kulicke & Soffa, US
$\Rightarrow$	Cutting, Grinding, Polishing		-	Bühler, DE
$\Rightarrow$	TEM Sample Preparation	Plan View and Cro incl. Ion Milling Ec		Gatan, US

## **SPIRIT**

The Institute of Ion Beam Physics and Materials Research is coordinating the Integrated Infrastructure SPIRIT under the FP7 Capacities Programme of the European Union. The duration of the project is from March 2009 to February 2013, the total funding is  $6.991.000 \in$ .

SUPPORT OF PUBLIC AND INDUSTRIAL RESEARCH USING ION BEAM TECHNOLOGY

> Integrated Activity, EU Project No. 227012 www.spirit-ion.eu



SPIRIT integrates 11 leading ion beam facilities from six European Member States and two Associated States. Seven of the partners provide Transnational Access to European researchers and industry at their infrastructures. Ion beams in an energy range from ~10 keV to ~100 MeV are supplied for modification and analysis of solid surfaces, interfaces, thin films and nanostructured systems, being mainly applied in research related to materials, biomedicine, environment, and cultural heritage. SPIRIT aims at increasing user access and the quality of research by sharing best practice, balancing supply and demand, harmonizing procedures and extending the services into new emerging fields and to new users especially from the New European Member States and industry. The project comprises a management section (4% of the total funding) and three interlinked activities, which are Transnational Access (45%), Networking (19%) and Joint Research (32%).

Analysis Techniques		Materials Modification Techniques	
Rutherford Backscattering Spectrometry	RBS	Ion Implantation	II
Elastic Recoil Detection	ERD	High-Energy Ion Irradiation	HEIR
High-Resolution Ion Beam Analysis	HRIBA	Plasma-Immersion-Ion-Implantation	PIII
Proton-Induced X-ray (γ-ray) Emission	PIXE/	Ion Beam Lithography	IBL
	PIGE		

Potential users from public of industrial research are invited for free use of the ion beam facilities at the SPIRIT partners laboratories, either by conducting experiments personally or by sending samples in case of standard ion implantation or ion beam materials analysis. Regularly, access is given on a transnational basis, i.e. the user has to be employed in a European Member or Associate State in which the specific infrastructure is not situated. However, there are exceptions in particular in case of industrial use and/or non-conventional use of ion technologies.

Proposals may be turned in continuously through the SPIRIT website and will be evaluated promptly by an international User Selection Panel. Any successful proposer will be granted free access to the infrastructure, with the associated travel costs and daily expenses being covered by the European Commission.

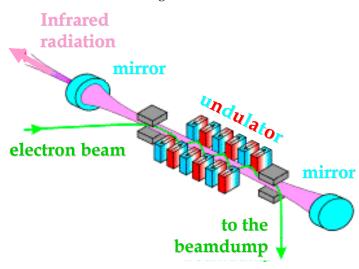
Until 31-March-2010, the institute is still providing Transnational User Access under the FP6 project AIM, which then will be followed up by access under SPIRIT.

	The SPIRIT Consortium		
	Forschungszentrum Dresden-Rossendorf	Germany	
	CNRS – CENBG Bordeaux	France	
CAPACITIES	Katholieke Universiteit Leuven	Belgium	
	Jozef Stefan Institut Ljubljana	Slovenia	
	Universität der Bundeswehr München	Germany	
	CEA – JANNUS Saclay and CIMAP Caen	France	
	University of Surrey	U.K.	
	Institute Tecnologico e Nuclear Lisboa	Portugal	
	University de Pierre et Marie Curie Paris	France	
	Ruder Boskovic Institute Zagreb	Croatia	
	Swiss Federal Institute of Technology Zurich Switzerland		
	(The top seven partners provide Transnation	nal Access)	

## Free Electron Laser FELBE

ELBE is an acronym for the free-electron laser (FEL) at the Electron Linear accelerator with high Brilliance and Low Emittance (ELBE) located at the Forschungszentrum Dresden-Rossendorf,

Germany. The heart of ELBE is a superconducting linear accelerator operating in cw mode with a pulse repetition rate of 13 MHz. The electron beam (40 MeV, 1 mA max.) is guided to several laboratories where secondary beams (particle and electromagnetic) are generated. Two free-electron lasers (U27-FEL and U100-FEL) produce intense, coherent electromagnetic radiation in the mid and far infrared, which is tunable over a wide wavelength range  $(4 - 250 \,\mu\text{m})$  by changing the electron energy or the undulator magnetic field. Main parameters of the infrared radiation produced by FELBE are as follows:



Parameter	Range / Value	Remarks		
Wavelength	4 - 22 μm 18 – 250 μm	FEL with undulator U27 FEL with undulator U100		
Pulse energy	0.01 – 2 µJ	depends on wavelength		
Pulse length	1 – 25 ps	depends on wavelength		
Repetition rate	13 MHz	3 modes: cw / macropulsed (> 100 μs, < 25 Hz) / single pulsed (HzkHz)		

The free electron laser is a user facility. Applications for beam time can be submitted twice a year, typically by April, 15 and October, 15. User from EU countries can receive support through the FP7 Integrated Infrastructure Initiative (I3) ELISA (European LIght Sources Activities). Typical applications are picesecond nump probe spectroscopy (also in combination with several other



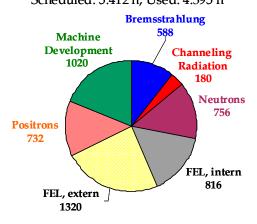
picosecond pump-probe spectroscopy (also in combination with several other femtosecond lasers, which are synchronized to the FEL), near-field microscopy and nonlinear optics. The FELBE facility also serves as a far-infrared source for experiments at the

High-Field Laboratory Dresden (HLD) involving pulsed magnetic fields up to 70 Tesla.

The statistics shows that the FEL used 2136 hours beamtime of the ELBE accelerator, which corresponds to 40% of total beamtime. These 40% were again distributed among internal and external users as shown in the graph.

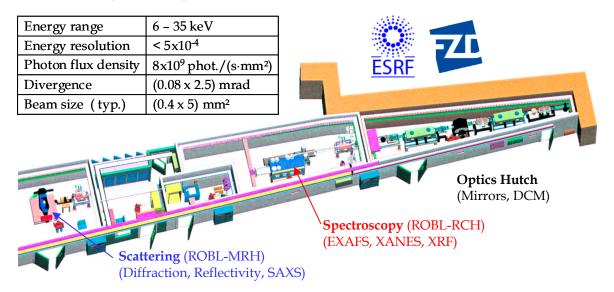
For further information please contact Prof. Manfred Helm (<u>m.helm@fzd.de</u>) or visit the FELBE webpage <u>www.fzd.de/FELBE</u>.

#### **Beamtime Distribution at ELBE 2009** Scheduled: 5.412 h; Used: 4.395 h



# Robl

The **RO**ssendorf **B**eamLine (ROBL), operated by the FZD since 1998, is a bending magnet synchrotron beam line located at the European Synchrotron Radiation Facility (ESRF) in Genoble, France. The set-up and main parameters are sketched as follows:



The activeness of ROBL is based upon the high specialization of its two end-stations for Radiochemistry (RCH) and Materials Research (MRH). ROBL-RCH is one of only two stations in Europe dedicated to X-ray absorption spectroscopy of actinides and other radionuclides. The core competence of ROBL-MRH is the analysis of thin films, multilayers and (ion-beam-synthesized) nanostructures using X-ray scattering techniques. A continuously increasing number of experiments are performed as *in-situ* X-ray studies using process chambers for magnetron sputter deposition, ion irradiation or annealing under vacuum or various (including reactive) atmospheres, respectively. Non-destructive X-ray analysis during materials processing is applied to characterize the formation or transformation of crystalline phases, their texture, the evolution of nanostructures, surfaces and interfaces properties or strain/stress development at thin films. These data are the key to understand the underlying physical or chemical processes and to interpret the corresponding macroscopic materials properties with the general aim to design materials with well defined properties.

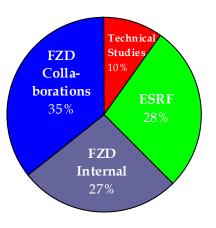
ROBL has the status of an user facility. Applications for ROBL beam time can be submitted twice a year, typically by March, 1 and September, 1 via the official ESRF proposal submission portal. Successfully reviewed proposals will be financially supported by the ESRF by covering the travel and accommodation costs. In addition, there is the possibility to use in-house research beam time for collaborative experiments between external users and FZD scientists for studies of common interest.

With more than 5000 beam time hours per year ROBL belongs to the most reliable and booked beam lines at the ESRF. The diagram at the right plots the beam time distribution of ROBL which reveal an availability of about 90% for experimental research studies. In 2009 visitors from more than 20 European and Overseas Universities and Research Institutions were involved in ROBL experiments.

For further information please contact

Dr. A. Scheinost	(ROBL-RCH):	scheinost@esrf.fr
Dr. C. Bähtz	(ROBL-MRH):	<u>baehtz@esrf.fr</u>

or visit the ROBL webpage www.fzd.de/ROBL.



# Services

### Main Areas of Competence:

- Ion implantation in a broad range of ion energy (~ 200 eV to ~ 50 MeV) and substrate temperature
- Advanced ion beam technologies (high energy ion implantation, plasma immersion ion implantation, focused ion beam) for (micro)electronic applications
- Deposition of functional coatings using ion-assisted physical vapor deposition
- Development and fabrication of sensors and detectors for charged particle spectroscopy
- High energy ion implantation service for power devices and laser structures
- Doping of semiconductors, in particular wide bandgap semiconductors
- Surface analysis of solid materials with high energy ion beams
- Optical characterization of materials (luminescence, FTIR, Raman)

### **Offers:**

- Consultation and problem evaluation for ion beam applications
- Process development for ion beam processing of metals, ceramics, semiconductors, thin films
- Preparation and treatment of material samples, tools or complex parts of devices
- Ion implantation and ion beam analysis services
- Electron beam lithography and focused ion beam patterning
- Fabrication of silicon radiation sensors under clean room conditions
- Structural diagnostics of materials including e-beam and X-ray techniques

### Contact:

Please direct your inquiry about the application of ion beams for modification and analysis of materials to one of the following experts:

Field of application	Name	Phone / Fax	E-mail
Ion implantation (metals, cera- mics, polymers, biomaterials)	Prof. Andreas Kolitsch	3348 / 2703	a.kolitsch@fzd.de
Ion implantation (semiconduc- tors, in particular high energy)	Dr. Johannes von Borany	3378 / 3438	j.v.borany@fzd.de
Thin film deposition	Prof. Andreas Kolitsch	3348 / 2703	a.kolitsch@fzd.de
High energy ion beam analysis	Dr. Silke Merchel	2802 / 3701	s.merchel@fzd.de
Semiconductor preparation Detector / Sensor fabrication	Dr. Bernd Schmidt	2726 / 3285	bernd.schmidt@fzd.de
Electron beam lithography	Dr. Artur Erbe	2366	a.erbe@fzd.de
Focused ion beams	Dr. Lothar Bischoff	2963 / 3285	l.bischoff@fzd.de
Structural diagnostics	Dr. Johannes von Borany	3378 / 3438	j.v.borany@fzd.de
Materials research with Synchro- tron radiation at ROBL (ESRF)	Dr. Carsten Bähtz	2367	baehtz@esrf.fr
Optical materials characterization	Dr. Harald Schneider	2880 / 3285	h.schneider@fzd.de

For all phone/ fax-numbers choose the country / local code:

+49 351 260 - xxxx (for FZD) +33 47 688 - xxxx (for ROBL)

The institute also recommends the homepages of its spin-off companies

- "GeSiM mbH" Si- Mikrosysteme
  - "APT Dresden" Angewandte Pulstechnik
  - "DFT GmbH" Dünnschichttechnologie

www.gesim.de www.apt-dresden.de www.dtf-technology.de

## Forschungszentrum Dresden - Rossendorf e.V.

## Institute of Ion Beam Physics and Materials Research (IIM)

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ION TECHNOLOGY (FWII) Prof. Dr. Andreas Kolitsch / 3326	SEMICONDUCTOR MATERIALS (FWIM) Dr. Wolfgang Skorupa / 3612		
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NANOFUNCTIONAL FILMS (FWIN) Prof. Dr. Jürgen Faßbender / 3096	SEMICONDUCTOR SPECTROSCOPY (FWIH) Dr. Harald Schneider / 2880		
<ul> <li>Modification of Magnetic Materials</li> <li>High Anisotropy Nanoparticles</li> <li>Magnetic Semiconductors / Spintronics</li> <li>Magnetization Dynamics</li> <li>Fullerene-like Materials</li> </ul>	<ul> <li>Semiconductor Quantum Structures</li> <li>Terahertz Spectroscopy</li> <li>Femtosecond Spectroscopy</li> <li>Free Electron Laser at ELBE</li> <li>Optical Characterization (PL, FTIR, Raman)</li> </ul>		
ION BEAM ANALYSIS (FWIA) Dr. Silke Merchel / 2802	STRUCTURAL DIAGNOSTICS (FWIS) Dr. Johannes von Borany / 3378		
<ul> <li>Ion-Solid-Interaction</li> <li>High-Energy Ion Beam Analysis</li> <li>Accelerator Mass Spectrometry</li> <li>Non-destructive Analysis of Art Objects</li> <li>Composition / Modification of Materials</li> </ul>	<ul> <li>Electron Microscopy (TEM, SEM)</li> <li>Electron Spectroscopy (AES, XPS)</li> <li>Mössbauer Spectroscopy</li> <li>X-ray Analysis</li> <li>Materials Research with Synchr. Radiation</li> </ul>		
THEORY (FWIT) Dr. Matthias Posselt / 3279	PROCESS TECHNOLOGY (FWIP) Dr. Bernd Schmidt / 2726		
<ul> <li>Ion-Beam Synthesis of Nanostructures</li> <li>Formation and Evolution of Defects</li> <li>Atomistic Simulation of Ion implantation and Ion-Assisted Deposition</li> <li>Interatomic Potentials for Solids Reaction-Diffusion-Models</li> </ul>	<ul> <li>Semiconductor Technology</li> <li>Focused Ion Beam Technology</li> <li>Thin Film Deposition</li> <li>Computer Aided Structure Design</li> <li>Electrical Characterization</li> <li>Clean Room Operation</li> </ul>		

# List of Personnel 2009

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Dr. H. Schneider	C. Baumgart	Diploma Students:	A. Scholz
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Dr. D. Stehr	K. Bernert	S. Germer	M. Steinert
Dr. M. Voelskow	M. Buhl	R. Hubrich	U. Strauch
Dr. S. Winnerl	D. Bürger	J. König	K. Thiemig
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	C. Cherkouk	J. Osten	J. Wagner
Dr. R. Heller	S. Cornelius	P. Philipp	R. Weidauer
Dr. N. Jeutter	M. Fritsche	E. Ritter	A. Weise
Dr. G. Kovacs	A. Hanisch	T. Schönherr	J. Winkelmann
Dr. K. Lenz	M. Höwler	R. Wiemann	U. Strauch
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	A. Keller	C. Wilde	I. Winkler
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Dr. C. Bunce	M. Kosmata	Technical Staff:	J. Zscharschuch
Dr. W. Bürger	T. Kunze	- certificar Otari,	Projects:
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Dr. M. Friedrich	A. Martinavicius	Ry. Aniol	I. Skorupa
Dr. D. Grambole	K. M. Mok	E. Christalle	A. Weißig
Dr. R. Grötzschel	S. Numazawa	H. Felsmann B. Gebauer	
Dr. J. Grebing	X. Ou	D. Gebauer	