Friedrich Schiller Universität Jena
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Dissertation

High-Fluence Ion Beam Irradiation of Semiconductor Nanowires

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Theses of the Dissertation

1. Semiconductor nanowires are a suitable model system to investigate the interaction between energetic ions and nanostructures.

2. The Sigmund sputtering model is a good approximation for the sputtering in nanostructures. It explains the qualitative structure-size dependence of the sputter yield.

3. Sputtering from nanostructures is comparable in experiments and simulations with the program *iradina*, a Monte-Carlo simulation tool based on the binary collision approximation.

4. The nanowire diameter and/or ion energy dependent sputter yield from nanowires is maximum where the nanowire diameter is equal to the projected range of the ion in the nanowire material.

5. The redeposition from the substrate onto upstanding nanowires is negligible compared to the sputtering from the nanowires. The fluence of atoms redeposited from the substrate onto the nanowire can be estimated to be around $0.1 \cdot SY \cdot \Phi$, with SY the sputter yield from the substrate surface.

6. Sputtering in nanostructures leads to a non-linear increase in the doping concentration with high irradiated ion fluences. This can be quantified by nano-XRF.

7. Static simulations are useful to predict doping concentrations up to an ion fluence where 20% of the total volume effected by the ion beam is sputtered. This fluence may be significantly lower in nanostructures than in bulk, because of the enhanced sputtering in nanostructures.

8. Si nanowires show plastic deformation when irradiated with 100 and $300\,keV\ Ar^+$ ions at room-temperature.

9. The deformation of Si nanowires by room temperature ion irradiation is not caused by point defects and is not oriented along the ion beam direction, the nanowires always become shorter.

10. A FIB system equipped with a micro-manipulator is a fun tool to manipulate and manufacture useful nanowire samples.

Contents

1 Introduction

Technological progress generally shows a competition between the optimization of the dominating technology and the development of fundamentally new operation principles. An example of this competition is recorded in the "International Technology Roadmap for Semiconductors", which aims to guide the scaling of digital devices to follow "Moore's Law" of improved performance, and the white paper "Towards a 'More-than-Moor' roadmap", which examines opportunities to include non-digital functionality where performance does not necessarily have to scale with size. Both are available at the ITRS website [map15]. A concrete example for a shift in operating principle for data storage was the fundamentally new effect of giant magneto-resistance (GMR) discovered in 1988. This quickly formed the basis for the standard hard-drives (HDD) and HDDs soon dominated PC data storage. Nowadays, the much older principle of flash memory is making a come-back in solid state drives (SSD), which begin to replace HDDs. They owe their viability (cost, speed and storage density) almost entirely to the advanced miniaturization, allowing the production of a floating gate for a transistor on a scale down to tens of nanometers per single *bit*, while producing *billions* of $^{bits}/_{cm^2}$. *A priori* it is not possible to discern with certainty which approach is going to produce the best results, so that much room is left for open minded fundamental research in general and on semiconductors in particular.

In the wake of the miniaturization used for the improvement of IT hardware technology, the new, multi-disciplinary field of nanotechnology has emerged. The scope of the field is illustrated by the high number of journals dedicated to research at the nanoscale. This includes semiconductor science, but in the leading journals *ACS Nano* [Nan15a], *Advanced Materials* [Adv15b], *Advanced Functional Materials* [Adv15a], *Nano Letters* [Nan15b], *Nature Nanotechnology* [Nat15], *Nano Today* [Nan15c], *Nanotoxicology* [Nan15d], *Small* [Sma15]

and others fundamental research and applications of nanoscaled devices and effects from all natural sciences are published.

Scaling down material to the nanoscale becomes interesting when the characteristic dimension of the effect governing the physical properties of interest become comparable to the structure size; then new, mesoscopic and quantum mechanical properties can emerge. As an example, the changed light-matter interaction in structures of the dimension of the wavelength of light is investigated in the field of nano-photonics and plasmonics [ST07, Mai07].

The specific class of nanomaterials investigated in this thesis are semiconductor nanowires, which have gained a significant amount of interest [HMF$^+$01, CWPL01, DHC$^+$01, XYS$^+$03, LW07]. 'Nanowire' is a term used for many morphologies, but it seems a reasonable name for structures with a cross-section that is between 1×1 and $1000 \times 1000\,nm^2$ and a large length to form a high aspect ratio. One of the general aspects of this shape and also of nanostructured materials in general, is that the surface properties play a dominating role. This is simply caused by the fact, that there is a lot of surface per volume of material. As the surface to volume ratio in general is proportional to $1/r$ for a body with a characteristic constraining length of r, it gets very large for small structure sizes.

The wire shape has an inherent advantage over three dimensionally constrained particles (nanoclusters, quantum dots etc.), in that it is easier to define contacts and drive a current through a nanoscaled wire than through a nanoscaled dot. Incidentally, the idea to combine this specific advantage of nanowires with new properties obtained by the stronger three dimensional confinement of quantum dots is the main idea behind the 'Deutsche Forschungsgemeinschaft' (DFG) project "wiring quantum dots", which funded this work.

The defining property of semiconductors is the ability to dramatically change their electronic properties by adding impurities [SN06]. As ion beam irradiation can be used to 'mix' (i.e. dope) virtually any target material with a precisely controlled number of atoms of practically any element, it was and is a key part in the processing and development of semiconductor technologies.

In general, ion beam doping has the advantage over doping during the synthesis of nanostructures, since it is not inherently limited by the chemical potentials and thermodynamics which typically have to be carefully controlled for the synthesis of nanostructures. It is a non-equilibrium physical process

by which different elements can forcefully be introduced into a target matrix with much higher energies than those involved in chemical bonding. The extent of disorder created in the target during this bombardment, whether the intermixing is thermodynamically stable, and whether a desired (crystal) order can be reestablished by thermal annealing is in the focus of ion-beam physics. A good background on this can be gained from dedicated literature [ZLB85, Eck91, Nas08, Sch12].

Typical ion ranges for the doping of semiconductors lie in the range of 10-100 nm. Therefore, ion beam irradiation of nanostructures of the same dimension will show some interplay between the irradiated structures' dimensions and the ion range. The many practical applications of the combination of ion beams and nanostructures warrants general investigations of the nanostructure - ion beam interaction and the topic has therefore gained increased interest very recently [Bor12, GHB$^+$13, NSUM14, JHMR15, UBNM15].

A specific example in which the combination of nanostructures and ion beams is advantageous is the ion irradiation of diamond to create nitrogen-vacancy clusters. The diamond is nanostructured to facilitate efficient extraction of light, while ion irradiation with N creates nitrogen-vacancy clusters very effectively. These are interesting as promising components in a future quantum information device [BHK$^+$10]. The precise control ion irradiation gives, makes it possible to implant a well defined number of ions with reasonable spacial accuracy. This control is extravagantly demonstrated by the possibility of single ion irradiation, shown in references [MVB$^+$06, Ohd08].

In addition to this extremely low ion fluence example of ion irradiation, the next two examples of the concurrence of nanotechnology and ion-irradiation led more or less directly into the investigations into high fluence irradiation presented in this dissertation. Firstly there is the search for a diluted magnetic semiconductor for which Mn doped $GaAs$-nanowires are a good candidate. As $GaAs$ nanowires typically grow above $450°C$ but $MnAs$ segregates from $Ga_{(1-x)}Mn_xAs$ at $350°C$ [DO06, SSK$^+$11], there is no straightforward way to dope $GaAs$ with high concentrations of Mn during nanowire growth. However, it can be achieved by implanting Mn in $GaAs$-nanowires. The key to this problem is to do the irradiation at elevated temperatures, hot enough to minimize disorder introduced by the ion beam, but cold enough to prevent segregation of $MnAs$ [BMB$^+$11, PKB$^+$12, Bor12, KPJ$^+$13, PKJ$^+$14].

Conversely, the "wiring quantum dots" project aimed to utilize the segregation of ion-implanted material in a nanowire to form nanowires decorated with nanoclusters. When Si nanowires are irradiated with high fluences of Ga^+ and As^+ and subsequently annealed with a flash-lamp, separated $GaAs$ slices form within the Si nanowires [PGL$^+$14, Gla15]. The supersaturation of Si with Ga and As by ion implantation can thus be utilized to create $GaAs$-Si nanowire hetero-structures from a Si nanowire template in a relatively straightforward manner.

A further important example of the intersection of nanotechnology and ion beams is found in the ubiquitous focused-ion-beam (FIB) systems. The production and development of many of the novel applications of nanostructures on the horizon often requires the precise ion-beam milling that FIBs provide with a resolution of few nanometers [KFM$^+$01, GTC$^+$10, CCJ$^+$13]. In all the examples given so far, and virtually per definition in the last one, the typical structure sizes irradiated are in the same order of magnitude at the range of the impinging ions. Understanding how this affects the ion-matter interaction can be crucial to the successful outcome of the respective experiments.

In the effort to understand principles and fundamental interactions on the nanometer length scales, nanowires are a very good model system to investigate, as their geometry is fully characterized by their height and radius. Spheres, which would have a degree of freedom less, are more difficult to handle, as the unavoidable proximity of a substrate may influence their behavior [HCA02, KZB$^+$09, Mö14, JHMR15]. The understanding of the ion-nanostructure interaction gained by investigating irradiated nanowires is principally transferable to any nanostructure. However, this can hardly be done in any general way explicitly, as the possible shapes of nanostructures are uncountable. This dissertation adds to the growing field of nanostructure - ion beam interaction the discussion of three effects which are especially important in high fluence irradiation and dedicates a separate chapter to each.

Chapter 3 - Sputtering of Nanowires

In the dissertation of Dr. C. Borschel [Bor12] the program *iradina* [BR11] was developed and used to simulate the ion irradiation of nanostructures. It predicts an enhanced, diameter-dependent sputter yield in nanostructures. This chapter discusses the simulation and compares

its predictions with experimentally obtained diameter-dependent sputtering in nanowires. Some first results on the sputtering during Mn irradiation of $GaAs$-nanowires are published elsewhere [JNP+14]. The results presented here are on Ar irradiated Si-nanowires. They were obtained in close cooperation with Stefan Noack in his M.Sc. and also published in reference [JNW+15].

Chapter 4 - High Doping Concentrations in Nanowires

The concentration of dopants does not follow a linear increase with the fluence of ions implanted for high fluences. It has already been observed in the early days of investigations into ion implantation that sputtering of the target will dynamically change its composition during the ion irradiation in addition to the intended change by incorporation of the ions within the target material [ME84, MEB88, MIS+91, SO93, Eck00]. This effect is enhanced in nanostructures, first, since the sputtering is enhanced when compared to bulk samples as shown in the preceding chapter, but also since there is simply less material in the structure. Hence, the effect of removing material by sputtering already becomes significant at lower fluencies in nanostructures than in bulk. The presented results are acquired by compositional analysis using nano-XRF performed on 175 keV Mn^+ ion irradiated ZnO nanowires [JNP+14]. They are discussed in comparison to a pseudo-dynamic simulation performed using results from *iradina*.

Chapter 5 - Plastic Flow in Silicon Nanowires

In the high ion fluence irradiated Si nanowires an unexpected tendency of the nanowires to become shorter was observed. This chapter presents a dedicated investigation into this plastic deformation of Si under ion irradiation which has been previously seen only in high energy ($\geq MeV$) ion irradiations [Vol91, TR95, HKW04, HKW05].

2 Conclusions and Outlook

The first conclusion, although it is actually almost a premise to this dissertation, is that sputtering is indeed an important effect that needs considering in high-fluence irradiation. As was shown in this dissertation for the example of Si-nanowires, this is especially true in the case of nanostructures, where sputter yields can be greatly enhanced. Qualitatively, a good estimation, or intuition, of how any given nanostructure will be sputtered can be obtained using the Sigmund model for sputtering. The relative size of overlap of the nuclear energy loss of the ion and the surface of the target, even if it is nanostructured, is a reasonable estimation for the relative sputter yield. Thus, a feeling for which part of a complicated nanostructure will be most affected by sputtering during the irradiation with ions of a certain species and energy can be gained. For a specific nanoscaled geometry the MC BCA simulation program *iradina* [BR11] can be used to make a more detailed analysis. The diameter dependence of the simulated sputter yield is confirmed by the experiments performed in this thesis.

The quantitative values of sputtering are, in general, not accessible through the naive use of MC BCA simulations. However, very good agreement can be found for certain material and ion combinations [Bie87, HZM14], so the situation is not at all hopeless. The main difficulty is to find correct low energy interaction potential for the colliding atoms and ions for the given situation. As the secondary ion mass spectrometry (SIMS) technique is highly reliant on sputter yields and MD simulations also require the correct interaction potential at low particle energies, there is some interest in solving this problem. As sputtering is dominated by low energy collisions, it is very sensitive to the interaction potential precisely at the energy range where it is not easily accessible to other experiments. Therefore, experiments on sputtering of defined nanostructures, such as the ones performed on nanowires within this thesis, may be a useful approach to test theoretical predictions based on different

interaction potentials. Such experiments should be combined and compared with angle dependent measurements of the sputtering [HZM14] and the angle resolved emission of the sputtered atoms [WM08, VWMS08]. Unfortunately, this aproach will not produce the correct interaction potential, however, it can be used to test results from simulations with different potentials to determine which describes sputtering best.

The main goal of ion irradiation is typically not sputtering, but the incorporation of dopants in the target. For nanostructured targets, care has to be taken to avoid an inhomogeneous irradiation and doping profile due to shadowing of the ion beam. This was illustrated with the nano-XRF investigation of Mn-doped ZnO nanowires. The first new result from this investigation is that the BC MCA simulation is adequate for the prediction of the doping concentration for low ion fluences in nanowires. The limit of this applicability is given by the point where around 20% of the material affected by the ion beam is sputtered, which in nanostructures is typically 20% of the whole nanostructures' volume. Similar approximations can be made in bulk [ME84, And86, MEB88, SO93, ZS95], even if the given references don't explicitly state a limiting fluence or sputtered depth. For irradiations with higher fluences, dynamic simulations are needed to predict the correct dopant concentration and profile. For nanostructures this ion fluence can be much lower than in bulk as there is less material to be sputtered and sputtering is enhanced. Software, which can dynamically change the structure and composition of the ion irradiated, nanostructured target, has been revealed recently in reference [Mö14]. A comparison of the experimental results presented in this thesis with the results from such a simulation is a logical next step.

Down these lines, the application of the nano-XRF quantification technique to ion irradiated nanostructures can produce further interesting results. As nano-XRF is highly sensitive to elemental concentrations, it can widen the scope of the proposed studies into sputtering by investigating compositional changes ion irradiated nanostructures of compound materials. In compound materials preferential sputtering of one of the materials' components may become relevant even before a high dopant concentration has been reached. The interplay of nano-structuring, compositional changes and preferential sputtering could thus be investigated for a vast array of materials, by no means limited to semiconductors. Comparison to simulation results would further

the understanding of the parameters influencing the preferential sputtering, which has practical meaning in SIMS, but also in the development of materials for fusion reactor components [Kel78, Rot90, KOW11]. Using nanowires for such an experiment has the advantage that samples with multiple diameters can be fabricated in parallel and thus a larger parameter space becomes accessible.

The sputtering of nanostructures is not only enhanced relatice to bulk because of the high surface to volume ratio, but may also be influenced by thermal effects, which are pronounced because energy deposited by the impinging ion is confined to a small volume [GHB$^+$13, IKN$^+$14, NSUM14, ABU15, UBNM15]. In the experiments presented in this thesis only minor thermal effects are observed. Two approximations show why this is the case: firstly, the nanostructures are relatively large compared to the ion energy deposited in them, leading to only a little energy deposited per atom; secondly, the ion mass and target atom mass are both relatively low, leading a low energy loss and a low density of the energy deposition.

Nevertheless, Si nanowires show plastic deformation when irradiated with medium weight (Ar^+) ions at room-temperature and energies of 100-300 keV. Where the lower threshold for the deformation is and whether there is an upper threshold energy above which the deformation ceases is not clear. It is clear that the observed deformation is not in line with the ion track induced plastic deformation proposed by Trinkaus et al. [TR95], as the energy loss of the ion is to low.

For high ion energies ($> 1\,MeV$), the deformation mechanism may transition from the proposed

Origami deformation of layers

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