High-Fluence Ion Beam Irradiation of Semiconductor Nanowires

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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 shift in operating principle is found, for example, in data storage, which changed fundamentally when the effect of giant magneto-resistance (GMR) was discovered in 1988 [BBF$^+$88, BGSZ89]. This quickly formed the basis for the standard hard-drives (HDD) and HDDs soon dominated PC data storage. Nowadays, the older principle of flash memory is making a comeback 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 $^{bits}/_{cm^2}$ [MME13]. This shows, that it is not *a priori* 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 aimed at 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* [**?**], *Advanced Materials* [Adv15b], *Advanced Functional Materials* [Adv15a], *Nano Letters* [Nan15a], *Nature Nanotechnology* [Nat15], *Nano Today* [Nan15b], *Nanotoxicology* [Nan15c], *Small* [Sma15] and others, fundamental research and applications of nanoscale devices and effects from all natural sciences are published.

The specific class of nanomaterials investigated within 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 the surface-to-volume ratio is large. Because this ratio in general is proportional to $1/r$ for a body with a characteristic constraining length of r, it becomes 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 nanoscale wire than through a nanoscale dot.

The high surface to volume ration ensures that a semiconductor nanowire with two contacts at either end is already a very sensitive device. Such simple devices have been shown to be possible gas-sensors [SCRZ09], they could measure the pH inside a cell [CWPL01] or the impact of a single ion [JNGR11]. By functionalization of the surface, it is, as one example of many, possible to create a selective biological sensor that can even detect the attachment of a single viruses [PZH+04]. Additional functionality can be gained by adding impurities to semiconductor nanowires, because this dramatically changes their electronic properties [SN06]. For example, changing the doping from n to p-type within a nanowire creates a pn-junction that can be used as a solar cell [KTK+08, CZP+12], while the combination of n and p-type nanowires can be used to fabricate a thermo-electric generator [Sch14].

A particularity flexible method to add impurities to semiconductors is ion beam irradiation, because it can be used to 'mix' (i.e. dope) virtually any target material with a precisely controlled number of atoms of practically any element. Ion beam doping is a well established technology, and it was and is a key part in the processing and development of semiconductor technologies [HH12]. In general, ion beam doping has the advantage over doping during the synthesis of nanostructures, because 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 (crystalline) 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 [BHK+10]. The diamond is nanostructured to facilitate efficient extraction of light, while ion irradiation with nitrogen creates nitrogen-vacancy clusters very effectively. These are promising components in a future quantum information device. The precisely controlled ion irradiation, 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 fundamental investigations of high fluence irradiation presented in this dissertation. Firstly, there is the search for a nanostructured diluted magnetic semiconductor for which Mn doped $GaAs$-nanowires are a good candidate. Because $GaAs$ nanowires typically grow above $450°C$ but $MnAs$ segregates from $Ga_{(1-x)}Mn_xAs$ at around $350°C$ [DO06, SSK+11], there is no straightforward way to dope $GaAs$-nanowires with high concentrations of Mn during their growth. However, it can be achieved by implanting Mn in $GaAs$-nanowires. Best results are achieved when the irradiation is performed 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, through which this thesis was funded, 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 In^+ and As^+ and subsequently annealed with a flash-lamp, separated $InAs$ slices form within the Si nanowires [PGL+14, Gla15]. The supersaturation of Si with In and As by ion implantation can thus be utilized to create Si-$InAs$ 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 nanoscale devices 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, because their geometry is fully characterized by their height and radius. Spheres, which would have a degree of freedom less, are more difficult to handle, because 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 a general way explicitly, because the possible shapes of nanostructures are uncountable.

This dissertation will begin with an overview of the ion-solid interaction focused on the accuracy of simulations of this interaction. A detailed review of effects and literature relevant to the ion irradiation of nanostructured materials is also given (Chapter **??**). Next, the methods used for the experiments are briefly outlined (Chapter **??**). This dissertation adds to the growing field of nanostructure - ion beam interaction by discussing three effects which are especially important in high ion fluence irradiation. A separate chapter is dedicated to each high ion fluence experiment.

Chapter **??** - 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. 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 [Noa14] in his M.Sc. and also published in reference [JNW+15].

Chapter **??** - 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 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, because the sputtering is enhanced when compared to bulk samples, as demonstrated in chapter **??**, but also because there is simply less material. Hence, the effect of removing material by sputtering already becomes significant at lower fluencies in nanostructures than in bulk. The presented results were acquired by compositional analysis using nano-XRF performed on 175 keV Mn^+ ion irradiated ZnO nanowires and are partially published in reference [JNP+14]. They are discussed in comparison to a pseudo-dynamic simulation performed using results from *iradina*.

Chapter **??** - Plastic Flow in Silicon Nanowires

In high ion fluence irradiated Si nanowires an unexpected tendency of the nanowires to become shorter was observed. 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]. These results were also obtained in part within the M.Sc of Stefan Noack [Noa14] and are published in reference [JNW+15]. A probable mechanism for the deformation can be presented by comparing the experimentally observed deformation with dedicated *iradina* simulations and literature on MD simulations of similar conditions.

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 ion irradiation. This is especially true in the irradiation of nanostructures, where sputter yields can be much larger than in bulk. The diameter dependent enhancement of the sputter yield was shown for Si-nanowires in this thesis. These results show that a good qualitative estimation, or intuition, of how any given nanostructure will be sputtered can be obtained by using the Sigmund model for sputtering. The relative size of the overlap of the ions' nuclear energy loss 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 nanoscale geometry the MC BCA simulation program *iradina* [BR11] can be used to make a more detailed analysis. The diameter dependence of the sputter yield simulated with *iradina* is qualitatively reproduced by the experiments reported 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. Because 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 [ND08]. Because 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, while the available theoretical models differ. An example of interesting sputtering behavior is found in Ni

and Cu, which are very similar in mass and density, yet show a factor of two difference in their sputter yield due to differences in surface binding, electronic energy loss and nuclear interaction [Bie87].

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. This approach is not limited to semiconductor nanowires, only by the availability of the nanowires of the desired material. This is not a significant constraint, because in addition to the bottom-up growth methods, template driven nanowire synthesis methods are very versatile [Mar96]. The systematical deviation, such as the one caused in the experiments reported here by oxidation of the Si-nanowires can certainly be controlled by an optimized experimental protocol. Overall, a quantitative evaluation of the sputter yield should be possible. Such experiments should be combined and compared with ion impact-angle dependent measurements of the sputtering [HZM14] and the angle resolved emission of the sputtered atoms [WM08, VWMS08]. The combination of such experiments can not produce the correct interaction potential, however, it can be a powerful test for results from simulations with different interaction potentials to determine which describes the inter-atomic interaction 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, which can be caused by shadowing of the ion beam in a nanostructure or amongst separate nanostructures. This was illustrated with the nano-XRF investigation of Mn-doped ZnO nanowires. The nano-XRF investigation shows 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 nanostructure's volume. Similar approximations can be made in bulk [ME84, And86, MEB88, SO93, ZS95], even if the given references don't explicitly state a limiting ion fluence or sputtered depth. For ion irradiation with higher ion fluences, dynamic simulations are needed to predict the correct dopant concentration and profile. The limiting ion fluence can be much lower for nanostructures than in bulk, because there is less material to be sputtered and sputtering is enhanced.

For high fluence ion irradiation, where more than 20% of the material is expected to be sputtered, dynamic simulations are recommended. 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. Because nano-XRF is highly sensitive to elemental concentrations, it can widen the scope of the proposed studies into sputtering by investigating compositional changes in 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. A comparison to simulation results would further increase the understanding of the parameters influencing the preferential sputtering, which has practical meaning in secondary ion mass spectroscopy (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, irradiated and investigated in parallel, and thus a larger parameter space becomes accessible to simultaneous investigation.

The sputtering of nanostructures is enhanced relative to bulk, not only because of the high surface to volume ratio, but also by thermal effects. These can be very pronounced if the energy deposited by the impinging ion is confined to a small volume [GHB+13, IKN+14, NSUM14, ABU15, UBNM15]. This can lead to explosive ejection of large clusters of 1000*s* of atoms. Such extreme thermal sputtering effects were not observed in the experiments presented in this thesis; firstly, because the nanowires are relatively large compared to the ion energy deposited in them, leading, on average, to only little energy deposited per atom; secondly, the ion mass and target atom mass are both relatively low, leading to a low stopping power and a low density of the energy deposition. The simulation of sputtering with the BCA and the Sigmund theory would break down in experiments where this is not the case.

Nevertheless, the maximum sputter yield is observed at lower nanowire diameters in the experiment than in the simulation. This could be caused by the correlation of thermal sputtering and the number of atoms in the volume effected by the ion, because a comparable amount of energy is distributed amongst fewer atoms in thinner nanowires than in thicker ones.

That thermal effects certainly play a significant role is illustrated by the fact that Si-nanowires show plastic deformation when irradiated with medium weight Ar^+ ions at energies of $100\,keV$ and room-temperature and not at elevated temperatures. It could be shown that this deformation is not mediated by point defects and is not directed along the ion beam. Where the lower ion energy threshold for the deformation is and whether there is an upper threshold ion energy above which the deformation ceases is not clear. The deformation is observed in amorphous Si-nanowires, but not in crystalline Si, both irradiated at elevated temperatures. In the crystalline case, the efficient recrystallization of the ion damaged nanowire volume recreates the long range order of the crystal lattice after every ion impact, while the amorphous material is free to remain incrementally deformed. This has also been observed in the plastic deformation or viscous flow of bulk samples by swift-heavy ion irradiation, which can be explained very well by ion track induced plastic deformation as proposed by Trinkaus et al. in reference [TR95]. It is clear, however, that the observed deformation in the experiments reported in this thesis is not another example of the mechanism reported by Trinkaus, because the energy loss of the ion is too low to form any sort of ion track. Also, it is not certain how the locally increased pressure required by an adaptation of this model can be confined to the limited volume of a nanowire. Therefore, a surface tension driven model, which relies on a locally reduced viscosity, was presented in this dissertation. This model is further supported by the fact that the deformed nanowires show pronounced smoothing of their surface and of edges, especially visible by a rounding of the top facet, which the undeformed nanowires irradiated at elevated temperatures do not show.

Regardless of the underlying mechanism, the plastic deformation of Si has great potential for nanostructuring applications, because it is highly localized at the point of the ions impact. In combination with the relatively low ion energy required it is predestined to be used in FIB assisted fabrication of nanostructures. It may be relevant to the formation of nanopores [GTC+10] and certainly to the bending and manipulation of nanowires [CFL+13] and freestanding films [KCA+06]. With suitable templates that may have to be thinned at the bending points, it could be possible to go as far as building '*Si*-nano-origami' similar to the metal 'origami' fabricated in reference [CCJ+13]. Such folding of *Si*-nanoshapes in particular may be a versatile fabrication tool in the growing field of *Si* MEMS devices. Furthermore, plastic deformation may have to be considered in the formulation of the mechanism for the formation of ripples on ion irradiated *Si* surfaces. The dated model by Bradley and Harper [BH88] considers only curvature and angle dependent sputtering as a roughening mechanism and is contested by models including ion induced strain and mass-transport [Nor12, KRG14]. The latter shows some similarity with the results presented here, indicating that an atomistic investigation may be necessary to resolve this issue.

All three chapters of this thesis have compared MC BCA simulations performed with *iradina* to experimental results on nanowires irradiated with high ion fluences. One limit to the applicability of the BCA is found were thermal effects have to be considered. This is not quite the case for the presented diameter-dependent sputter yields, where the simulation overlaps qualitatively with the experimental results. However, the plastic deformation found in amorphous *Si*-nanowires can not be explained with this simulation technique. Furthermore, the obtainable accuracy of *iradina* simulations of the prediction of the doping concentration in nanostructures determined with is satisfactory, but limited to low ion fluences. When the desired doping concentration is high and high ion fluences have to be implanted, dynamic simulations become neccessary. A rule of thumb lower limit to what constitutes a 'high' ion fluence is given by the fluence at which 20% of the volume effected by the ion beam is sputtered. These limitations do not formulate a criticism of the program *iradina* or the MC BCA simulation technique in general, but only serve to warn against their naive use in situations in which they are not applicable.

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