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Full Length Article

On the mechanism of ion-induced bending of nanostructures

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ABSTRACT

This contribution concentrates on ion-induced bending phenomena which may serve as a versatile tool to manufacture nanostructured devices. In particular bending was studied in free standing Au cantilevers. The preparation and irradiation of the cantilevers were performed using a TESCAN LYRA dual beam system. Cantilevers with thicknesses ranging between 90 and 200 nm were irradiated with 30 keV Ga ions normal to the sample surface up to a maximum fluence of $\sim 3 \times 10^{20}$ Ga/m^2. The bending of the cantilevers towards the incident beam is discussed in terms of local volume change due to accumulation of radiation-induced vacancies and substitutional Ga atoms in the Ga implantation layer, as well as due to accumulation of interstitial type clusters in the region beyond the Ga penetration range. A model is proposed to explain the observations, based on a set of rate equations for concentrations of point defects, i.e. vacancies, self-interstitials and implanted Ga atoms. The influence of preexisting defects is also discussed. The work shows that an in-depth understanding the ion-beam bending can play a predictive role in a quantitative control in for the micro- and nanofabrication of small-sized products.

1. Introduction

Focused ion beam (FIB) technology came a long way from its advent in the seventies, when their utility for maskless doping of silicon was first proposed. As the feasibility of different liquid metal sources was proved and the beam spot sizes were reduced to the range of tenths of nanometers, ion beam technology became an advantageous method for sub-micron fabrication and maskless processes such as imaging, implantation, etching and deposition [1]. Today it plays an important role in applications which range from micro- and nanofabrication to sample preparation and materials characterization. Assessment of the properties of submicron- and nanometer-sized samples is challenging, however, because of the difficulties in fabricating specimens which are ideally free of preparation artifacts. FIB milling techniques for nanosample preparation and characterization can result in undesirable effects such as bending and microstructural modification due to ion implantation. In particular, dual beam systems combining focused ion beam and scanning electron microscope (FIB-SEM) are frequently used for sample preparation and characterization. Typically, the FIB gun operates with 30 keV Ga$^+$ ions and is used for cutting away or building up structures on a material surface with a resolution of about 50 nm.

A rather recent development has been the ability to bend small-nanosized structures by ion irradiation in a reproducible manner, with a good potential for the manufacturing of 3D nanodevices in photonics and metamaterials. It has been demonstrated e.g. by the fabrication of free-standing plasmonic nanograter structures with the FIB [2]. Ion induced bending (IIB) was observed in multi-wall carbon nanotubes [3], thin film cantilevers of silicon nitride [4], amorphous-metal bilayers [5], a series of metals and nitrides irradiated with different ion species [6], as well as nanoporous metallic pillars [7], proving the versatility of the technique.

The phenomenon was first reported by Yoshida et al. [8] who were able to bend a W_2S thin film cantilever towards or away from the incident Ga$^+$ ion beam by changing its energy. Borschel et al. [9] observed the opposite relation between ion energy and bending direction using GaAs nanowires. In both cases, the bending momentum was argued to be related to the ion implanted range in relation to the sample thickness – due to relaxation of internal stresses in the former and generation of crystallographic defects in the latter. A study on ZnO nanowires reported no recovery of bending after annealing up to 800 °C, suggesting that bending has been made permanent by the nucleation and motion of dislocations [10]. Attempts to explain ion beam bending mechanism were based on the stress generated by ion implantation and gas/vacancy complexes, thermal gradients induced by ion irradiation or the movement of knock-on atoms along the film thickness [4–6]. The observation of 'no bending' in the case of electron beam

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irradiation shows that the thermal expansion due to irradiation-induced heating is not significant in the case of metallic nano-objects [5]. Ion-induced amorphization has also been suggested as a main factor in ion-induced bending of semiconductors [11]. Romano et al. [12] investigated ion bending of Ge nanowires and suggested a mechanism of ion-induced amorphization and subsequent densification. Pecora et al. [13] observed recovery of bending in Si nanowires after annealing and attributed bending to the generation of a crystalline/amorphous interface. Ishii et al. [14] confirmed that most of the stress lies in the amorphous phase for the case of Si and developed a model of relaxation of ion-induced defects to account for the flux dependence of stress evolution. In metals, the bending process can be thought to result from volumetric expansion caused by ion implantation. From an atomistic point of view, stresses generated in the free standing sample of thin film are generally attributed to the accumulation of defects during ion irradiation. The Bragg Coherent X-ray Diffraction Imaging (BCDI) has been recently used to resolve lattice distortion in gold nanoparticles with high precision [17]. The results suggest that the use of FIB causes large lattice distortions, which are consistent with a damage microstructure dominated by vacancies. At larger ion fluences, typical for FIB milling, formation of a dislocation network was reported. However, in general the underlying physics of the bending effect is not clear. Understanding of this phenomenon is crucial for attaining high accuracy and predictability, which will enable the use of IIB as a reliable method for manipulating the shape and geometry of functional nanostructures in nanodevices.

In this paper we present detailed investigations of IIB on metallic cantilevers taking Au as a typical example of an fcc structured material. The next section presents experimental details on sample preparation and characterization. Section 3 reports experimental results. In Section 4 we discuss possible bending mechanisms and present a new kinetic model based on a set of rate equation for concentrations of implanted Ga atoms and point defects (PD). The influences of preexisting dislocations and PD clusters created in displacement cascades are also discussed.

Modeling results discussed in Section 5 show that the bending under ion beam irradiation cannot be explained just by the generation of isolated PDs and deposition of Ga ions since their effects are too small. We argue that IIB can be explained by a mass transfer from irradiated cantilever layer to the deeper layers due to gliding/diffusion of interstitial clusters which are formed in displacement cascades. Mobile clusters collide with each other and form sessile clusters and interstitial dislocation loops. The amount of material transferred to the unirradiated zone grows with the fluence and leads to local volume increase resulting in bending.

2. Experiments

Thin gold films were deposited by evaporation (Temescal FC-2000 electron-beam evaporator) on mechanically polished sodium chloride substrates. These were posteriorly dissolved in water, allowing the films to float and to be collected with a TEM grid so that the films were free-standing. The grids containing the films were annealed at 340 °C for 30 min in argon atmosphere to minimize the influence of defects, such as grain boundaries and dislocations which could have been introduced during the films deposition and manipulation. They were then placed in an in-house developed holder that is able to position the samples surface perpendicular to the ion beam in a Tescan Lyra dual-beam FIB instrument.

To prepare the cantilevers and to perform the bending experiments (Fig. 1) the film specimens were irradiated with 30 keV Ga^+ ions at normal incidence.

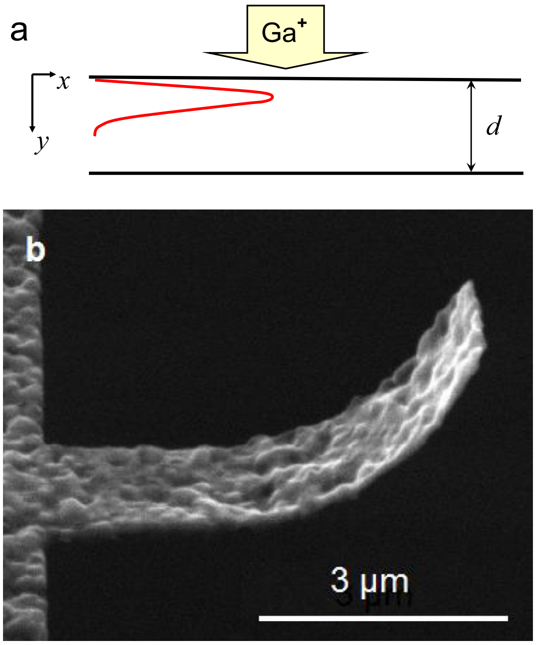

Fig. 1. Ion-beam irradiation of the cantilever (a); the implantation profile of Ga^+ ions is shown schematically. Deflection of Au cantilever ($d_0 = 144$ nm) after irradiation to a fluence of $1.4 \times 10^{20}\ m^{-2}$ (b).

The bending cantilevers were fabricated with FIB as illustrated in Fig. 2. The procedure was chosen to avoid bending during the cutting steps and to provide a clear view to the cantilever during the experiments. Two areas were cut on both sides of the interest region and bent up by irradiating the lines that connect them to the rest of the film. Finally the remaining "bridge" was cut forming the cantilever which will be irradiated. A current of 200 pA was used for cutting and 40 pA for all other steps. Care was taken to reduce as much as possible the ion exposure of the regions of interest prior to the bending experiments.

The irradiation was performed over an area slightly larger than the cantilevers to assure that all parts of it were equally processed. A parallel scanning strategy was used, meaning that each scanning pixel in the selected area would be irradiated sequentially for a dwell time of 1 µs. A minimum of 300 scans (equivalent to a fluence of $\sim 3.0 \times 10^{19}$ Ga/m^2 at 40 pA) was used for each measurement step, before the cantilever would be imaged by scanning electron microscopy. The irradiation/imaging steps continued until bending reached an advanced stage. The cantilever deflection was measured from the SE images using ImageJ [15] and Engauge [16], considering the perspective correction required by the 55° tilt of the electron beam in relation to the ion beam.

The Ga concentration in the irradiated films was measured by energy-dispersive X-ray spectroscopy (EDS) in an FEI-Philips FEG XL30S operating at an acceleration voltage of 5 kV. A sample with initial thickness of 144 nm was used and irradiation with progressive fluences was performed without additional preparation over areas of 8 × 8 µm. Element quantification was obtained using the semi-quantitative, standardless eZAF method; comparative analysis is nevertheless possible as all measurements were made using identical parameters in a single sample.

3. Experimental results

Fig. 3 presents the deflection and curvature plots for several irradiation fluences. Intermediate lines regarding smaller steps were omitted for clarity. The curvature along the cantilevers

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