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Rapid and localized ion-beam etching of surfaces using initial notches

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ABSTRACT

Glancing-angle Ar^+ broad ion beam erosion is widely used for the preparation of high-quality transmission electron microscopy (TEM) samples. However, low erosion rates and lack of site specificity are major drawbacks of the method. Being inexpensive and easy to use – in particular when compared to widely used focused ion beam preparation methods – overcoming these drawbacks would significantly improve many existing preparation workflows. We present a novel method for rapid and localized surface erosion which combines laser-machining preprocessing with broad ion beam etching. In this article, preliminary studies of the method on bulk samples are reported. Furthermore, an electron-transparent lamella has been prepared as proof of concept.

Using an ultrashort-pulsed solid-state laser, notches were created on (100)-Si substrates. Due to the local change in surface inclination, preferential erosion took place behind the notches upon subsequent ion beam etching at glancing angles. As a consequence, a terrace structure possessing a well-defined jump in surface height was formed. The surface topography and its evolution dynamics were characterized and the findings compared to numerical simulations based on a deterministic, two-dimensional model. On this basis, a workflow utilizing these initial notches (iNotches™) for the preparation of an electron transparent lamella was realized and TEM micrographs of the prepared sample were taken.

1. Introduction

The ever-increasing demand for high-quality transmission electron microscopy (TEM) samples makes reliable and efficient preparation techniques a necessity. Samples are required to be electron-transparent, plane-parallel in large regions of interest and free of damage. Mechanical procedures such as cleaving (McCaffrey, 1993) and tripod or wedge polishing (Ayache and Albarède, 1995; Chen and Ivey, 2002) can satisfy these demands in some cases. However, most of the time ion beam techniques are used for either the required thinning itself or as a means of postprocessing for damage reduction.

Two different approaches to ion beam etching can be distinguished. Focused ion beams (FIB) have gained widespread use in target-preparation procedures, allowing for a highly accurate preparation with a resolution of a few nanometres (Cooper and Ben Assayag, 2013). The most commonly used type of FIB is based on Ga^+ liquid-metal ion sources and, albeit having excellent resolution, has considerable drawbacks due to low erosion speed and high implantation rates. A more recent approach employs Xe^+ plasma ion sources, which show a considerably higher erosion rate and low implantation at the cost of lower resolution (Smith et al., 2006). Furthermore, He^+ ions created by gas field ion sources have been used for ultra high precision material modification and removal (Fox et al., 2012). However, due to very low erosion rates as well as sample damage caused by the formation of helium bubbles within the sample, this approach is only suitable for final processing of samples. As an alternative to FIB, Ar^+ broad ion beam (BIB) erosion at glancing angles is an established method which has been successfully employed in the preparation of high-quality TEM samples for decades (Barna, 1991). Utilizing small incidence angles and low ion energies, damage to the material can be restricted to a layer of less than 1 nm thickness (Barna, Á. et al., 1999; Süess et al., 2011). However, due to low erosion rates at grazing incidence, pre-thinning is necessary. Furthermore, as typical beam diameters are in the order of a millimetre, there was no way of achieving site-specific erosion so far.

Recent research investigates the use of ultrashort-pulse laser micromachining in sample preparation (Höche et al., 2015). With typical tool sizes (i.e. beam spot diameters) in the order of 10 μm and erosion rates about 10^3 or 10^6 times higher than plasma FIB or liquid metal FIB, respectively, laser mircomachining fills a gap between mechanical preparation methods and FIB and BIB techniques (Martens et al., 2010). Sample cutting and pre-thinning to roughly 15 μm thickness is achievable within minutes. This does come at the cost of a laser-affected damage layer of some 100 nm though, which needs to be removed by final ion beam polishing.

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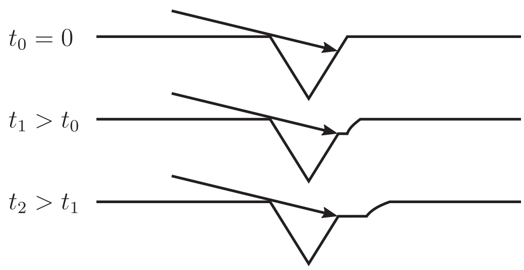

Fig. 1. Schematic drawing of the working principle of ion beam erosion utilizing initial notches, viewed as a surface cross-section perpendicular to a notch. Only the relative change of surface shape is shown by choosing a reference frame moving with the surface left of the notch. At the flank of the notch, preferential erosion due to locally changed surface inclination takes places, leading to the formation of a terrace. The terrace is bounded by a curved slope, which propagates forward with increasing erosion duration.

This contribution reports on a novel method combining the advantages of laser machining and BIB. The basic principle is shown in Fig. 1 in side view: surface modifications in the form of notches are prepared by laser micro-machining. Upon subsequent broad ion beam erosion, the sidewalls of these initial notches (iNotches™) are eroded preferentially due to the local change in surface inclination. Thus, a terrace structure with a well-defined jump height does form, leading to increased erosion speed and a laterally controlled erosion depth.

The technique was investigated on (100)-Si bulk samples. The evolution of surfaces with initial notches exposed to glancing-angle erosion using several ion-etching parameters was characterized by scanning electron microscopy and optical profilometry and the results were compared to numerical simulations. Finally, as proof of concept, an electron-transparent lamella was prepared and TEM micrographs were taken.

2. Materials and methods

2.1. Simulation algorithm

The evolution of a surface under ion bombardment was simulated using a two-dimensional model. The following, simplifying assumptions were made:

- sputtering is considered as a deterministic process,
- the sputter yield Y only depends on the local ion-impact angle,
- ion flux is uniform,
- the sample medium is homogeneous and isotropic and the edge of the sample is far away from the notches, so that it does not influence the surface evolution at the notches.

An algorithm described by Dieterle et al., (2011) was adopted, which will be outlined below. The employed geometry is sketched in Fig. 2. The surface consists of a string of points (x_i, h_i) with related local surface angles α_i and projected coordinates l_i. The projected coordinate is used to formulate a shadowing condition: a point i is shadowed if there is some point $j < i$ with $l_j \geq l_i$. All points not shadowed are eroded and the corresponding height change is given by

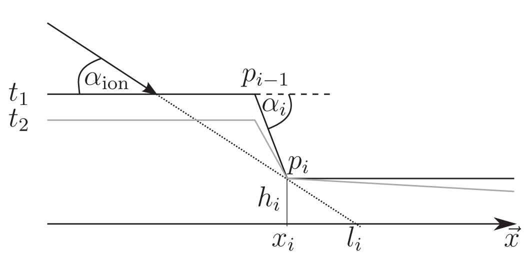

Fig. 2. Schematic drawing of the geometry used in the numerical model.

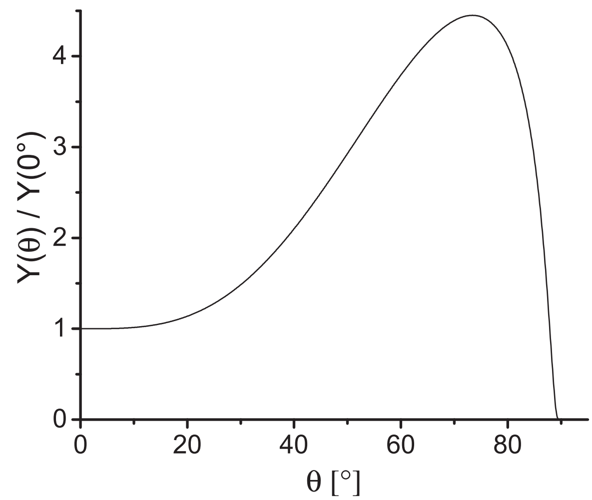

Fig. 3. Angle dependence of the sputter yield as a function of incidence angle to the surface normal (cf. Eq. (2)), with $a = 4.92$, $b = 3.30$ and $c = 0.068$.

$$\Delta h_i = \mathrm{K}Y(\theta_{\mathrm{imp},i})\frac{\cos(\theta_{\mathrm{imp},i})}{\cos\alpha_i}. \tag{1}$$

The local ion impact angle $\theta_{\mathrm{imp},i}$, measured towards the surface normal, is given by $\theta_{\mathrm{imp},i} = |90° - (\alpha_i + \alpha_{\mathrm{ion}})|$. The factor K is a time scale factor that controls the amount of material removed within one simulation cycle. In this study, $K = 0.0004$ was used, which provided satisfactory results in terms of numerical stability and computation speed. The cosine term in the denominator of Eq. (1) projects the erosion along the local surface normal onto the h-axis, while the cosine term in the enumerator projects the ion flux onto the local surface element. The latter factor is not present in Dieterle et al. (2011), since a stochastic Monte-Carlo scheme was used in that work, in contrast to the deterministic algorithm outlined above. This adaptation significantly reduces computation time and algorithmic complexity. In this study, the main roughening process was due to irregularities of the laser-cut notches (see Section 3.2), therefore, neglecting sputter noise is a reasonable simplification.[1]

The angle dependence of the sputter yield was modeled by an analytic function proposed by Dieterle et al. (2011),

$$Y(\theta) = (1 + a\sin^b(\theta))\exp\left(-c\left(\frac{1}{\cos(\theta)} - 1\right)\right), \tag{2}$$

with $a = 4.92$, $b = 3.30$ and $c = 0.068$. In Fig. 3, this dependence is shown as a function of ion incidence angle relative to the surface normal. The code was implemented using GNU Octave (Eaton et al., 2015).

2.2. Experimental methods

The experimental approach followed a three-step scheme, namely:

1. sample preparation and laser cutting of initial notches,
2. broad ion beam erosion, and
3. surface metrology.

As a model system, (100)-Si was used. Discs with three milimeter diameter and roughly 200 µm thickness were prepared using ultrasonic drilling and mechanical grinding. The initial notches were cut using a microPREP™ (3D-Micromac AG) laser machining tool with a picosecond diode-pumped solid-state laser. The system operates at a laser

[1] Other secondary processes like sputter redeposition and ion reflection, while certainly being relevant, have been excluded from this model as well.