Ultramicroscopy 186 (2018) 35–41

Contents lists available at ScienceDirect

Ultramicroscopy

journal homepage: www.elsevier.com/locate/ultramic

Preventing damage and redeposition during focused ion beam milling: The “umbrella” method

T. Vermeij[a,b], E. Plancher[a,*], C.C. Tasan[a,*]

[a] Massachusetts Institute of Technology, Department of Materials Science and Engineering, 77 Massachusetts Avenue, Cambridge, MA 02139, USA
[b] Eindhoven University of Technology, Department of Mechanical Engineering, Den Dolech 2, 5612 AZ Eindhoven, The Netherlands

ARTICLE INFO

Article history:
Received 20 September 2017
Revised 1 December 2017
Accepted 6 December 2017
Available online 7 December 2017

ABSTRACT

Focused ion beam (FIB) milling has enabled the development of key microstructure characterization techniques (*e.g.* 3D electron backscatter diffraction (EBSD), 3D scanning electron microscopy imaging, site-specific sample preparation for transmission electron microscopy, site-specific atom probe tomography), and micro-mechanical testing techniques (*e.g.* micro-pillar compression, micro-beam bending, in-situ TEM nanoindentation). Yet, in most milling conditions, some degree of FIB damage is introduced via material redeposition, Ga^+ ion implantation or another mechanism. The level of damage and its influence vary strongly with milling conditions and materials characteristics, and cannot always be minimized. Here, a masking technique is introduced, that employs standard FIB-SEM equipment to protect specific surfaces from redeposition and ion implantation. To investigate the efficiency of this technique, high angular resolution EBSD (HR-EBSD) has been used to monitor the quality of the top surface of several micro-pillars, as they were created by milling a ringcore hole in a stress-free silicon wafer, with or without protection due to an “umbrella”. HR-EBSD provides a high-sensitivity estimation of the amount of FIB damage on the surface. Without the umbrella, EBSD patterns are severely influenced, especially within 5 µm of the milled region. With an optimized umbrella, sharp diffraction patterns are obtained near the hole, as revealed by average cross correlation factors greater than 0.9 and equivalent phantom strains of the order 2×10^{-4}. Thus, the umbrella method is an efficient and versatile tool to support a variety of FIB based techniques.

1. Introduction

In the last couple of decades, focused ion beam (FIB) milling has been established as a key tool to manufacture, modify or polish specimens at the micrometer and nanometer scales [1]. A wide variety of tools to analyze the microstructure (*e.g.* transmission electron microscopy (TEM) [2], 3D tomographic imaging [3], 3D electron backscatter diffraction (EBSD) [4], 3D electron channeling contrast imaging (ECCI) [5] and atom probe tomography [6]) and micromechanical testing techniques (*e.g.* micro-pillar compression [7], micro-beam bending [8] and *in situ* tensile tests [9–11]) strongly rely on FIB to quantitatively characterize microstructural features or mechanical fields down to atomistic scale [12,13]. Yet, measurements in the vicinity of FIB-milled areas are often disturbed by undesired features stemming from ion implantation [14–18] or material redeposition [19].

Ion implantation is caused by the capture of Ga^+ ions within the crystal [18]. The implantation process leads to the creation of crystallographic defects such as vacancies and dislocation loops [17], and even leads to amorphization [20]. The TEM bright field image shown in Fig. 1(a) illustrates the difference in defect density observed in a copper film between the undamaged crystal and an area exposed to the Ga^+ ion beam [16]. Similar observations have been made with ECCI [21]. Increasing defect density (i) blurs channeling and diffraction contrasts in ECCI images and Kikuchi patterns used for EBSD, creating significant challenges for several microstructure characterization techniques in the scanning electron microscope (SEM) [22]; and (ii) has significant influence on the mechanical testing of plasticity mechanisms [14].

Redeposition takes place since some of the atoms removed by the ion beam (which cannot all be evacuated by the vacuum system) reattach elsewhere on the surface [23]. Fig. 1(b) shows how redeposition builds up at the edges of a trench created in steel by FIB milling [19]. As seen in the atomic force microscopy (AFM) measurement, redeposition pile-ups are formed at the edges of the trench. The amount of redeposition gradually decreases with the distance to the trench. When the thickness of the amorphous

* Corresponding authors.
E-mail addresses: emeric.plancher@mines-saint-etienne.org (E. Plancher), tasan@mit.edu (C.C. Tasan).

https://doi.org/10.1016/j.ultramic.2017.12.012

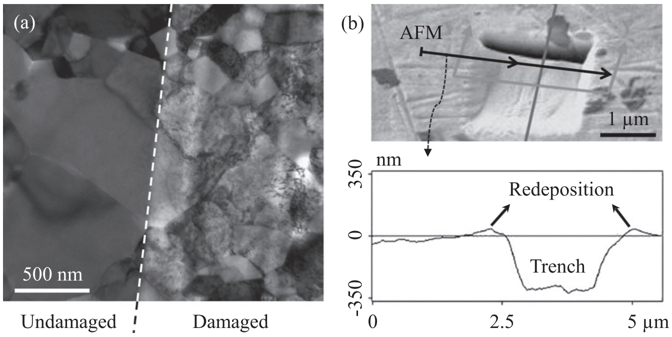

Fig. 1. Ion implantation and redeposition induced by FIB milling. (a) Defects introduced in a copper film partially exposed to FIB. The TEM bright-field image shows the difference in defect density between the undamaged crystal and a damaged area (reproduced with permission from [16]). (b) Surface topography changes due to redeposition close to a trench. The top part shows a secondary electron image of the FIB-milled trench. The bottom part reports the AFM height profile measured over the above line, showing redeposition outside the trench (reproduced with permission from [19]).

redeposition layer approaches the size of the backscatter electron interaction volume, channeling and diffraction signals are diffused, leading to a diminution of the contrast in ECCI images and Kikuchi patterns. In conventional ringcore experiments, redeposition also tends to cover the patterns used for digital image correlation of secondary electron images [12]. The contrast distribution is disrupted in the SE images as the milling proceeds, potentially leading to erroneous measurements of the initial strain state.

To overcome these drastic effects, various strategies have been developed to prevent FIB damage [10,24–27]. Most common approaches are based on optimizing FIB parameters or the milling geometry. Examples include using multiple FIB currents in a single machining process, milling the surface to be analyzed from the backside [10], avoiding the use of FIB images by more careful SEM imaging [24]. Such approaches successfully reduce ion implantation or redeposition, however, it is difficult to ensure for all cases that a fully damage-free microstructure is obtained. The success thereof often depends on how focused the beam is at low accelerating voltages, a parameter difficult to assess experimentally. A preliminary ECCI investigation carried out by the authors (not reported here) has shown that even at low accelerating voltages (~1 kV) and under grazing incidence conditions, FIB damage could not be avoided in a dual-phase steel. Other more intrusive approaches focus on processing the sample either prior to the milling step (*e.g.* by adding sacrificial layers [25,26]) or after it (*e.g.* by carrying out annealing treatments [27]). These methods are also successful but introduce other challenges: heat treatments have unwanted effects on the microstructure and sacrificial layers limit the access to the surface. It might be that other strategies to decrease FIB damage have been investigated and even employed, but the literature is currently limited on this topic despite a steady increase in the popularity of FIB. The task is especially challenging because assessing damage *in situ* (without removing the sample from the FIB instrument) is hardly possible.

This brief overview reveals an urgent need for a generic method to efficiently protect the sample surface against all FIB damage. This method should: (i) enable the use of damage-sensitive techniques such as ECCI and high angular resolution EBSD [28] (HR-EBSD) after FIB milling; (ii) require only standard equipment and accessories found in dual-beam FIB-SEM instruments; and (iii) enable site-specific protection that can be applied to a wide range of specimen shapes and material types. In what follows, we present the “umbrella” method that involves temporarily masking a given surface area of interest with a soft polymer block to fulfil the three conditions set above. This is a simple idea at first glance, however, fully shielding the surface against FIB-induced damage with such an approach is not possible without a thorough optimization of the umbrella material and shape. Additionally, we propose HR-EBSD as a powerful *in situ* tool to assess the amount of FIB damage. To evaluate the efficiency of the umbrella method, it is used here to monitor the difference in quality of the top surface of several micro-pillars, as they are being created by milling a ringcore hole in a stress-free silicon wafer, with or without the protection provided by the umbrella.

2. Methods

A schematic overview of the umbrella method is shown in Fig. 2. The specific goal here is to protect the top surface of micro-pillars obtained by ringcore drilling experiments [29]. In this work, several approaches have been investigated. They are illustrated in Fig. 2(a). Without any protection (red path) extensive FIB damage, due to both ion implantation and redeposition, is observed on the sample surface. Some improvement is observed when an unoptimized umbrella is temporarily placed on the top of the pillar during the milling step, using a micro-manipulator (MM) (orange path). In this scenario, the umbrella shape and material are not optimized so the amount of damage observed is reduced but not minimized. The surface is mostly shielded from direct ion implantation but redeposition occurs under the umbrella, as atoms ejected from the hole penetrates in the gap between the surface and the umbrella. Most of our trials have fallen into this category. To achieve an optimal protection, the gap should be closed, by enabling an excellent contact between the umbrella and the surface of the material to be milled (green path). Additionally, the um-

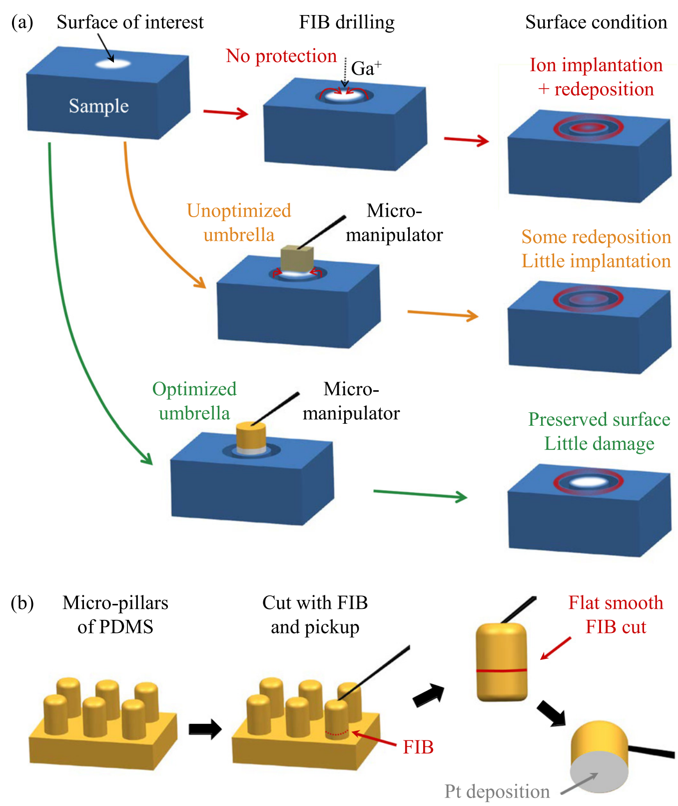

Fig. 2. Schematic overview of the umbrella method. (a) Damage obtained in a FIB drilling experiment of a ringcore hole, without protection (red), with protection of an unoptimized umbrella (orange) and the optimized umbrella (green). Damage is shown as red glow on the surface. (b) Schematic representation of the umbrella manufacturing process. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)