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Glow discharge plasma as a surface preparation tool for microstructure investigations

Varvara Brackmann[a,*], Volker Hoffmann[a], Alexander Kauffmann[a,b], Arne Helth[a], Jürgen Thomas[a], Horst Wendrock[a], Jens Freudenberger[a], Thomas Gemming[a], Jürgen Eckert[a,b]

[a]IFW Dresden, P.O. Box 270116, D-01171 Dresden, Germany
[b]TU Dresden, Institute of Materials Science, D-01062 Dresden, Germany

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ABSTRACT

In order to reveal the true microstructure of a sample one must often go through tedious and time-consuming surface preparation steps. The most abundant ones are polishing and etching using hazardous chemicals. Sputtering in the glow discharge (GD) lends itself as a simple preparation tool, which supplies mild but fast removal of the deformation layer and exposure of the microstructure. However, the GD is not well studied in terms of its use for preparation of samples. In the present work the influence of GD sputtering on the sample surface is systematically studied. Some examples of the samples prepared with GD are shown. Polycrystalline Cu needs up to 8 h to be conventionally prepared, while with GD the same surface is reached after 10 s of sputtering. In the case of the Ti40Nb alloy, sputtering in GD is the best method for the preparation of this material only within 5 min. The successful EBSD-measurements of the considered samples approve the good quality of the prepared surfaces. Various artifacts of sputtering are found. On the one hand they disturb the measurement, but on the other hand, they may contain useful information about the sputtered material.

1. Introduction

Investigation of microstructure and composition is of great importance for the characterization of material properties. The most abundant methods used for microstructure investigations at surfaces are light optical microscopy (LM), scanning electron microscopy (SEM), and electron backscatter diffraction (EBSD). For elemental analysis energy-dispersive X-ray spectroscopy (EDXS), Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), and secondary ion mass spectrometry (SIMS) are most frequently applied. For all these methods it is crucial to treat the sample surface before analysis such that it most closely represents the bulk material. It is hugely important to avoid artifacts of the preparation, which may mask the true microstructure and lead to false interpretations. Therefore the preparation steps are laborious, time consuming and often frustrating. Usually the sample should be embedded, grinded and carefully polished. Grinding and polishing procedures lead to the formation of scratches and a plastically deformed layer on the sample surface. The depth of scratches, which determines the roughness and damage depth, shrinks with decreasing abrasive size. The deformation layer can reach up to several μm (e.g. 15 μm for steel and 20 μm for copper) [1]. Thus, during polishing it is important to remove not only the scratches from each prior step but also the deformed layer.

The microstructure of materials, whose components strongly differ in reflectivity or hardness, can be seen in LM or SEM in the as-polished condition (e.g. Pb–Sb–Sn or Bi–Mn alloys) [2].

* *Corresponding author*. Tel.: +49 3514659691; fax: +49 3514659452.
E-mail address: vefimova7@gmail.com (V. Brackmann).

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However, for many materials the microstructure is revealed only by etching. Etching is commonly done by exposure to appropriate chemical etchants (e.g. hydrofluoric acid, sulfuric acid) after which the sample surface gets a characteristic topography. One of the methods without application of hazardous chemicals is etching with accelerated ions. The ion etching methods may be classified into physical sputtering by an ion beam, physical cathode sputtering by ion bombardment from a gas discharge, and chemical removal of material through gas atoms activated in a gas discharge or ion beam (reactive ion etching (RIE) with CCl_3F_3, SF_6, CF_4, etc.). The RIE is intensively used in the semiconductor industry. The discharges with gas mixtures are usually applied for cleaning of the surfaces. For instance, in an $Ar–O_2$ plasma dissociated oxygen atoms chemically react with hydrocarbons and convert them to CO, CO_2, and H_2O that are removed by the vacuum system. For the exposure of the microstructure the existing plasma devices are not suitable due to low sputtering rates. In the case of an ion beam the directed ions (e.g. focused ion beam (FIB), ion energies in the keV region) bombard the surface at high vacuum conditions [3]. The ion beam techniques are often very expensive and time consuming. In this paper, a new sputtering approach for surface etching is proposed — physical sputtering in the glow discharge (GD) with pure Ar as discharge gas.

Sputtering by means of GD plasma has a number of features, which make it attractive for sample preparation:

- The sputtered area is comparably large, typically some mm^2.
- Due to the comparably high gas pressure (several mbar), atoms and ions in the GD undergo numerous collisions transferring their kinetic energy and changing their trajectories. Hence, in contrast to high vacuum ion beam sputtering, the atoms and ions in the GD strike the target with a wide angular distribution [4]. This favors homogeneous sputtering over the sample surface.
- Due to the frequent collisions the particles in the GD lose their energy and bombard the sample with significantly lower energies than at high vacuum sputtering (some 100 eV vs >1 keV, commonly) [5,6]. The trajectories of low energy ions in the target are confined to a shallower depth of some Angstroms. Also the number of collisions made by a projectile in the solid is much smaller at low energies [7,8]. Thus the sputtering induced surface changes like ion implantation, atomic mixing and surface topography formation [9] are less pronounced in the case of GD.
- As compared to high vacuum sputtering, GD has much bigger current densities (100 mA/cm^2 vs 1 $\mu A/cm^2$) because of the higher pressure. Therefore, for GD sputtering the sample erosion rates are much higher (100 nm/s vs 1 nm/s) but with far less lattice damage [4].
- The sputtering yield in the GD plasma depends on the material properties, such as composition, crystallographic orientation, and density. Hence, after sputtering the characteristic topography is elaborated.

The above-listed properties of GD sputtering could render it a useful surface preparation tool, which can both mild but fast remove the deformation layer induced by polishing and expose the microstructure.

So far, GD is usually applied for the elemental analysis of solids where the material under investigation is sputtered. For elemental analysis, the GD plasma, where the sputtered material is introduced, is usually under focus, whereas very few authors pay attention to the sputtered sample surface [10–17]. Bruhn and Harrison studied the influence of the sputtering parameters on the surface sputtered in GD, but for another source configuration, which is not suitable for the preparation purposes [11]. Recently, Shimizu and Mitani published a pioneering work about the application of GD sputtering for sample preparation [18]. Several examples of application in this book show that GD is an advantageous sample preparation tool, which leads to significant improvement of the SEM image quality. However, the practical use of GD sputtering requires operator experience. In the following parts, a systematic study of the influence of the sputtering parameters on the sample surface as well as some preparation examples is shown. The presented results describe the various effects and artifacts, which can be expected for GD sputtering.

2. Experimental

For the preparation purposes in the present study, a Grimm-type plasma source integrated into the GD OES (glow discharge optical emission spectrometer, GDA 650, Spectruma) was applied. The anode diameter and hence the diameter of the sputtered area were 2.5 mm. The plasma gas was 99.999% pure Ar.

Microstructural characterization was carried out by a scanning electron microscope (SEM) (Zeiss Gemini 1530) with attached electron backscatter diffraction (EBSD) system (Channel5, Oxford instruments, with Nordlys2 detector). SEM images were taken at an acceleration voltage of 20 kV, SE detection, and nontilted sample.

The Cu samples for the systematic study were prepared by the following procedure: firstly commercially available oxygen-free Cu was melted in an induction furnace and cast into a graphite mold (Ø = 25.5 mm) in Ar atmosphere. Afterwards the samples were deformed from Ø 25.5 mm down to 10.5 mm into a rod shape. The rods were annealed in air for 4 min at 500 °C (the Cu oxide formed only on the sample surface due to the short annealing time). This way samples with a crystallite size of some μm were reproducibly obtained. From the rods disks were cut, embedded in graphite-grafted epoxy and grinded up to 4000 paper grade.

3. Results and Discussion

3.1. Systematic Study

When looking for the optimal sputtering conditions for the preparation, the question arises how the sputtering parameters impact on the sputtered surface? In the case of GD, up to now there is no literature, where this aspect is comprehensively explored. Therefore, the discussion in the following part is dedicated to the sputtering conditions and their influence on the sample.