Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 54 (2006) 5393–5399
Acta MATERIALIA
www.actamat-journals.com

Converting polycrystals into single crystals – Selective grain growth by high-energy ion bombardment

Sven Olliges [a], Patric Gruber [b], Anita Bardill [a], Daniel Ehrler [a], Heinz Dieter Carstanjen [c], Ralph Spolenak [a,*]

[a] *Laboratory for Nanometallurgy, Department of Materials, ETH Zurich, Wolfgang-Pauli-Strasse 10, 8093 Zurich, Switzerland*
[b] *Universität Stuttgart, Institut für Metallkunde, Heisenbergstrasse 3, 70569 Stuttgart, Germany*
[c] *Max Planck Institute for Metals Research, Heisenbergstrasse 3, 70569 Stuttgart, Germany*

Received 24 March 2006; received in revised form 6 July 2006; accepted 7 July 2006
Available online 27 September 2006

Abstract

Common failure mechanisms in microelectronics such as electromigration, creep and fatigue can be positively influenced by microstructure optimization. In this paper a new mechanism of microstructure optimization in thin metal films is proposed. Post-deposition ion bombardment can produce an in-plane texture in originally highly fiber textured thin metal films by a selective grain growth process. In extreme cases the in-plane texture becomes as sharp as the out-of-plane fiber texture. A subset of grains oriented for ion channeling was found to grow significantly at the expense of the remaining grain fraction. We studied the selective grain growth as a function of ion species (N^+, Ne^+, Ar^+), ion energy (1–3.5 MeV) and target temperature (liquid nitrogen to 400 °C). In a textured thin film the degree of preferred in-plane orientation can be strongly influenced by ion bombardment, and therefore this technique has the potential to become a powerful tool for the enhancement of reliability in micro- and nanosystems.

Keywords: Ion-beam processing; Electron backscattering diffraction; Thin films; Crystal growth

1. Introduction

The texture of thin metal films can be controlled by ion bombardment assisted deposition [1–3]. Dong and Srolovitz [1,2] report in their theoretical work two main effects that influence film texture during deposition: (a) the anisotropy of the sputter rate, and (b) the anisotropic generation of defects due to ion bombardment; the latter effect is the stronger. Due to anisotropic defect generation, less damaged grains grow at the expense of strongly damaged grains. Minimization of the free volume energy is the driving force.

Changes to thin film texture caused by post-deposition ion bombardment were observed by Spolenak et al. [4,5] in a focused ion beam (FIB) microscope. They observed selective ion-induced grain growth in near-surface regions of thin gold and copper films by 30 keV Ga^+ bombardment, where the grains showing channeling grow at the expense of the grains showing no channeling. Since the cross-section for defect generation is reduced under channeling when compared to non-channeling conditions, the results are qualitatively consistent with the simulations of Dong and Srolovitz [1,2] and the experimental observations of Park et al. [3], respectively.

To date, no selective grain growth by ion bombardment in the 1.0–4.0 MeV range has been observed. In the present work, experiments with a variety of ion species, ion bombardment temperatures (T_{irr}) and ion energies were performed to control the texture in existing thin gold films. Because of the high ion energy the effect was observable also in non-near-surface regions in the 500–1000 nm depth range. Since the influence of ion bombardment on the

* Corresponding author.
E-mail address: ralph.spolenak@mat.ethz.ch (R. Spolenak).

1359-6454/$30.00
doi:10.1016/j.actamat.2006.07.005

average grain size is well known [6,7], in the present paper we focus on the development of in-plane textures.

2. Experimental

Polycrystalline gold thin films were deposited under ultrahigh vacuum conditions (10^{-9} mbar) on a silicon substrate coated with 50 nm SiO_2 and 50 nm Si_3N_4 by magnetron sputtering at room temperature (RT). Before deposition, the substrate surface was cleaned by 1 keV Ar^+ bombardment for 1 min. The thin gold films show a strong (1 1 1) fiber texture parallel to the surface normal. The grains have a mean diameter of approximately 80 nm and a columnar structure; the thicknesses of the films are 500 and 1000 nm.

The [1 1 0] axes are the main channeling axes in the face-centered cubic (fcc) lattice. Since one [1 1 1] axis of any grain is parallel to the surface normal, ion channeling along [1 1 0] corresponds to an incident angle of 35.24° to the surface normal (given by the angle between the [1 1 1] and [1 1 0] directions, Fig. 1). Small variations in the incident angle are permitted; their size should just be smaller than the critical angle for axial ion channeling in crystals as derived by Lindhard [8]

$$\psi_c = \sqrt{\frac{2Z_1Z_2e^2}{d \cdot E}}, \tag{1}$$

where $Z_{1,2}$ are the atomic numbers of the incident ions and target atoms, respectively, d the distance of adjacent atoms in an atomic chain, E the ion energy in eV and $e^2 = 14.4$ eVÅ the square of the elementary charge. For 1 MeV N^+, as used here in one of the irradiation experiments, the critical angle for the [1 1 0] main channel axis in gold is calculated to be 4.3°.

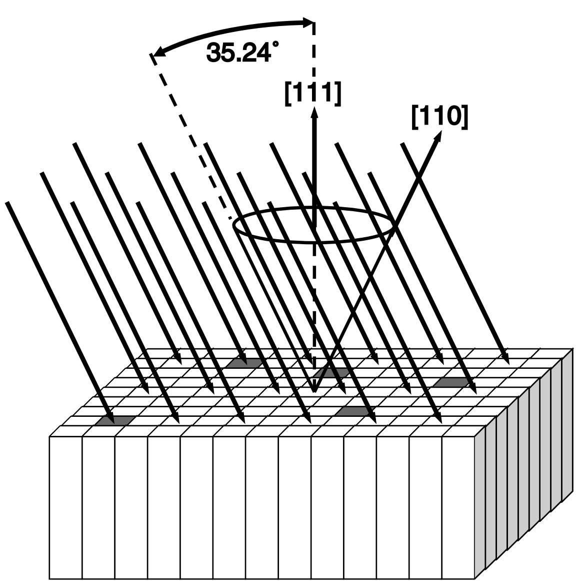

Fig. 1. Schematic drawing of the experimental situation in a (1 1 1) textured film. One [1 1 1] axis of any grain is parallel to the surface normal while the other [1 1 1] axes are distributed randomly due to one degree of freedom with respect to the surface normal. The [1 1 0] axes form a cone with an angle of 35.24° to the surface normal.

The thin films were irradiated with 1.0–3.5 MeV N^+, Ne^+ and Ar^+ ions with the ion beam direction at an angle of 35.24° to the surface normal using the 6.5 MV Pelletron accelerator of the Max Planck Institute (MPI) for Metals Research in Stuttgart. The ion fluences were in the region of 10^{17} ions/cm^2 and the target currents in the 10–100 nA range, depending on the ion species. The temperature during ion irradiation was kept at either liquid nitrogen (LN_2, approximately −180 °C) temperature, RT or 400 °C, respectively. After ion bombardment, the beam spots on the target were measured using a dark-field optical microscope to calculate the respective ion fluence from the total ion charge collected on the target. Normalized pole figures, obtained by electron backscatter diffraction (EBSD) using an HKL Channel 5 EBSD detector in a LEO 1530 VP scanning electron microscope at the MPI for Metals Research, Stuttgart, display the thin film texture before and after ion bombardment.

The area of the beam spot exceeded the grain size by eight orders of magnitudes. Thus, only a few irradiated grains showed channeling while the rest showed non-channeling behavior. For a polycrystalline thin film with (1 1 1) texture normal to the surface, but otherwise random orientation, the percentage of grains showing channeling is given by

$$p = \frac{\psi_c \cdot n}{4 \cdot \sin\alpha} = \frac{A_i}{A_0} \tag{2}$$

for $\alpha > \psi_c$, where ψ_c is the critical angle of the investigated system, n the symmetry group C_n of the channeling system with respect to the surface normal, and α the angle between the beam and the surface normal. In the present case of the [1 1 0] direction, $n = 3$, $\alpha = 35.24°$ and $\psi_c = \psi_c([1\,1\,0], E, Z_1, Z_2)$. This expression is equivalent to the normalized subset A_i showing channeling of an area A_0. In the current investigations the critical angles are in the regime of 4° which corresponds to approximately 9% of the grains (or area) showing channeling.

In comparison to non-channeling, the interaction between ions and lattice-atoms is strongly reduced due to the lattice-steering effect during ion channeling [9], which results in a non-uniform generation of defects in the thin film. According to previous theoretical and experimental investigations [1,2,4], we expect changes in the in-plane texture due to ion-induced selective grain growth, where the fully rotational degree of freedom with respect to the [1 1 1] axis (parallel to the surface normal) is reduced to a cone of the size of the critical angle ψ_c along the direction of the incident ion beam.

3. Results

Fig. 2(a) shows the initial pole figure of an untreated thin film (1000 nm thick). It exhibits a sharp peak in the centre of the {1 1 1} pole figure corresponding to the