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ScienceDirect
Surface & Coatings Technology 201 (2006) 965–970
SURFACE & COATINGS TECHNOLOGY
www.elsevier.com/locate/surfcoat

The hardness study of oxygen implanted aluminum thin films

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Received 7 November 2005; accepted in revised form 5 January 2006
Available online 21 February 2006

Abstract

Effects of mass analyzed low energy O_2^+ ion implantation in Al thin films on the hardening and microstrucure have been studied by nanoindenting atomic force microscopy (AFM). The fluence range was 1×10^{17}–1×10^{18} O atoms/cm^2. A maximum increase of hardness about 90% of the implanted samples is observed. Apparently there is no significant variation in the hardness values within the range of ion fluences investigated here. Finally, the surface roughness is found to decrease considerably by oxygen irradiation.

Keywords: Al thin film; O_2^+ ion implantation; Hardness; Nanoindentation; rms roughness; AFM

1. Introduction

Surface mechanical properties of metals is known to be dramatically modified by ion implantation [1]. Implantation changes the composition and chemical bond structure of the target surface region to a depth comparable to the projectile range. Hardness is one of the most important surface properties of materials, which is usually measured by Brinell, Vickers or Rockwell hardness tester [2]. In traditional hardness tests, the probe depth exceeds the thickness of the implanted layers ($\ll 1\,\mu$m), even at normal loads, resulting in a large contribution of the unimplanted layers to the apparent hardness value. The advent of atomic force microscopy-based nanoindentation technique has enabled to determine precisely the mechanical properties of surfaces of bulk materials and also that of thin films at the nanodepth scales. In nanoindentation, an indenter tip made of diamond with a known geometry is driven into the sample by applying an increasing normal load. The applied load varies from some tens of μN to several mN. When reaching a preset maximum value, the normal load is reduced until there is a partial or complete relaxation. For each loading/unloading cycle, the position of the indenter relative to the sample surface is recorded and a load–displacement curve is obtained. Another important advantage is that one can immediately take an image of the indented area by the same tip for residual area calculation.

Aluminum and its alloys have many engineering applications because of several advantages such as low density, high thermal and electrical conductivities and low cost. But the major disadvantage of aluminum is the poor performance in sliding and hardness. Recent publications [3,4] show that oxygen plasma source ion implantation can improve the tribological properties of bulk Al and its alloys. However, it is known that the mechanical properties of thin films may differ from those of the bulk due to size effect. In this paper, the hardness of Al thin films deposited on Si substrates followed by mass analyzed low energy O_2^+ ion bombardment has been investigated and the results are discussed.

2. Experimental

Thin films of Al were deposited on a clean Si(100) substrate by DC magnetron sputtering (Pfeiffer, PLS 500). The nominal thickness of the film was 200 nm as determined from the step height with a Dektak^3 ST surface profiler. The samples were implanted with mass analyzed 16.7 keV O_2^+ ions at normal incidence in a home built low energy ion beam (LEIB) set-up [5]. It is known that the molecular oxygen upon impact to the surface will be implanted as two O atoms of half the incident energy. The beam current density was 11 μA/cm^2. The projectile fluence was varied in the range 1×10^{17}

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0257-8972/$ - see front matter
doi:10.1016/j.surfcoat.2006.01.018

O atoms/cm^2 to 1×10^{18} O atoms/cm^2 measured by a current integrator (Danfysik, model 554) after suppression of the secondary electron emission. The base pressure in the target chamber was less than 8×10^{-8} mbar.

Nanoindenting is performed with a diamond-tipped cantilever mounted on a NanoScope IV MultiMode AFM (Veeco, USA) at ambient conditions [6]. The stainless steel cantilever has a high spring constant of 206.1 N/m with a resonant frequency of 63 kHz. The tip has a tetrahedric geometry (three-sided pyramid) with an apex angle of about 60° and the nominal tip radius of curvature is about 25 nm. The typical indentation force ranges from 1 to 100 μN with a resolution of ~0.5 μN. To provide more symmetric dents, the tip is given an *x*-rotation of 12° so that the vertical axis of the pyramid is approximately normal to the sample surface. For making series of indents we used Auto Indent option to produce the indentation arrays automatically setting the number of columns and rows in the array and the spacing between them. For each dent, a force–displacement curve was recorded. The applied force can be determined by Hooke's law, i.e., the product of the spring constant and the deflection of the cantilever. The geometrical dimensions and the relative positions of the indented areas can be imaged non-destructively with the same tip by operating the AFM in the tapping mode. However, the morphology of the surface at the non-indented area was measured separately by a silicon cantilever with the sharper silicon tip in order to quantify the surface roughness. An arithmetical average of the root-mean-square (rms) roughness, R_q is determined from three or more scans ($5\times5\,\mu m^2$) on different positions of the sample surface.

3. Results and discussion

In Fig. 1, the AFM images of the pristine surface and bombarded surfaces present a sequence of the evolution of the surface topography with increasing ion fluences. Fig. 2 shows the corresponding rms roughness versus the ion fluence. One can see that there is an exponential decrease of roughness with increasing ion fluence. The initial rms roughness is about 7.4 nm and the final surface roughness after a total fluence of 1×10^{18} O atoms/cm^2 becomes ~2 nm. The smoothing of rough surfaces by reactive ion beam etching has also been demonstrated by Frost et al. [7]. For 16.7 keV O_2^+ implantation in Al, SRIM simulation [8] yields values for the projected range, R_p and straggling σ as 20 nm and 10 nm, respectively. However, SRIM does not take into account sputtering which affects the implantation profile. We have calculated the expected implantation depth profiles following the method of Krimmel and Pfleiderer [9], where the simultaneous action of ion implantation and the erosion of the surface due to sputtering have been considered. The

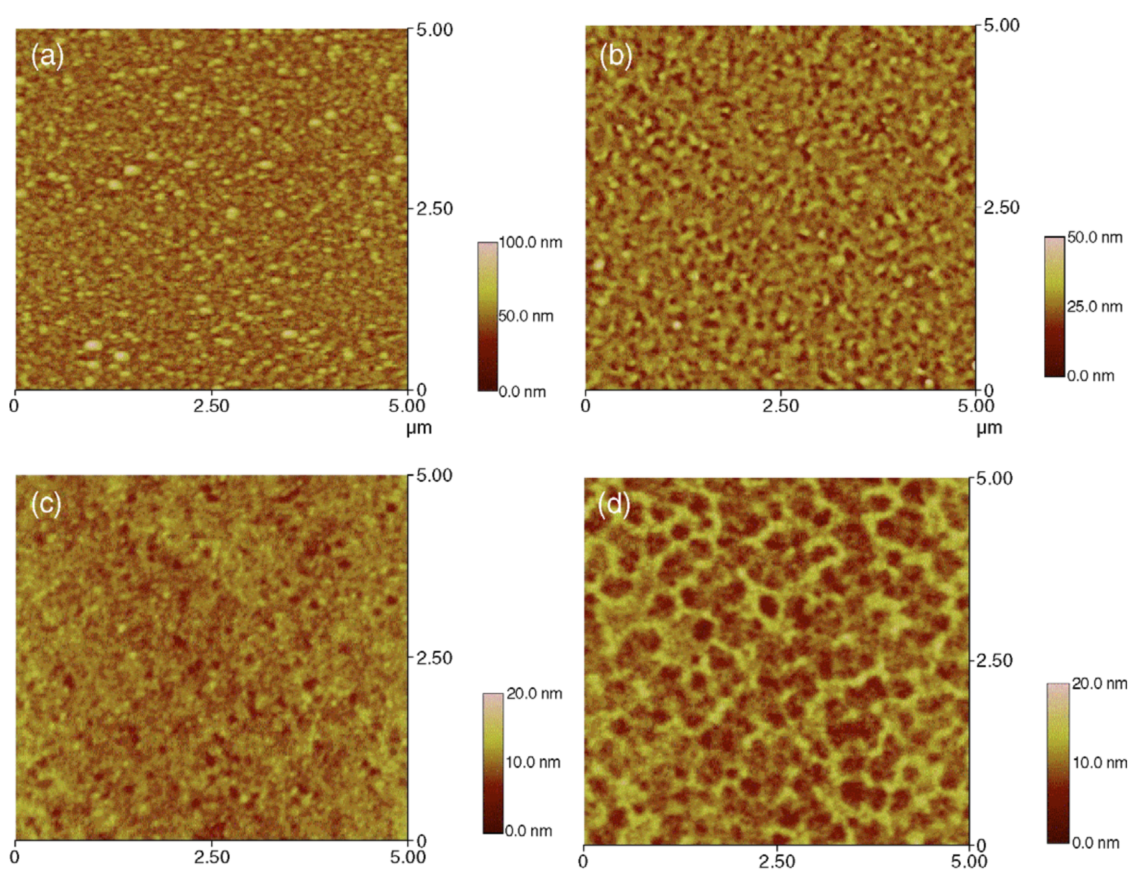

Fig. 1. Some representative AFM images showing surface topography of Al thin films: (a) as-deposited; after irradiation with 16.7 keV O_2^+ ion beam at (b) 1×10^{17} O atoms/cm^2, (c) 5×10^{17} O atoms/cm^2, and (d) 1×10^{18} O atoms/cm^2. Note the different height scales of the AFM images.