

## Characterization of Atomically Smooth Al Films by Transmission Electron Microscopy and Atomic Force Microscopy

Morihide Higo,\* X. Lu,<sup>†</sup> U. Mazur,<sup>†</sup> and K. W. Hipps<sup>†</sup>

*Department of Applied Chemistry and Chemical Engineering, Faculty of Engineering, Kagoshima University, Korimoto, Kagoshima 890*

*<sup>†</sup>Department of Chemistry, Washington State University, Pullman, Washington 99164, U.S.A.*

(Received April 16, 1997; CL-970278)

Atomically smooth Al films were prepared by vacuum evaporation on heated mica substrates at 350°C. The films consist of single crystals 270 nm in diameter with (111) faces and the crystals are oriented randomly about the [111] direction which is perpendicular to the substrate. The roughness of the Al film surfaces is 0.6 nm over 1 × 1 μm areas.

Atomic force microscopy (AFM) is a new technique for imaging surfaces with a high spatial resolution and has been extensively used in many studies.<sup>1</sup> One of the most promising applications of AFM is the surface characterization of metal films, because metal films are hard and have the adequate size of surface structures.<sup>2,3</sup> The surface properties of thin Al and the oxide films influence such as wear, friction, coating, and light scattering. Lu et al.<sup>4</sup> have prepared atomically smooth gold films on mica at high temperatures and obtained the molecular images of metal phthalocyanine on the surfaces by scanning tunneling microscopy (STM). In the present letter, we report the preparation of atomically smooth Al films on mica and the characterization of the films by transmission electron microscopy and diffraction (TEM/TED). The morphology and roughness of the Al film surfaces were also studied by AFM.

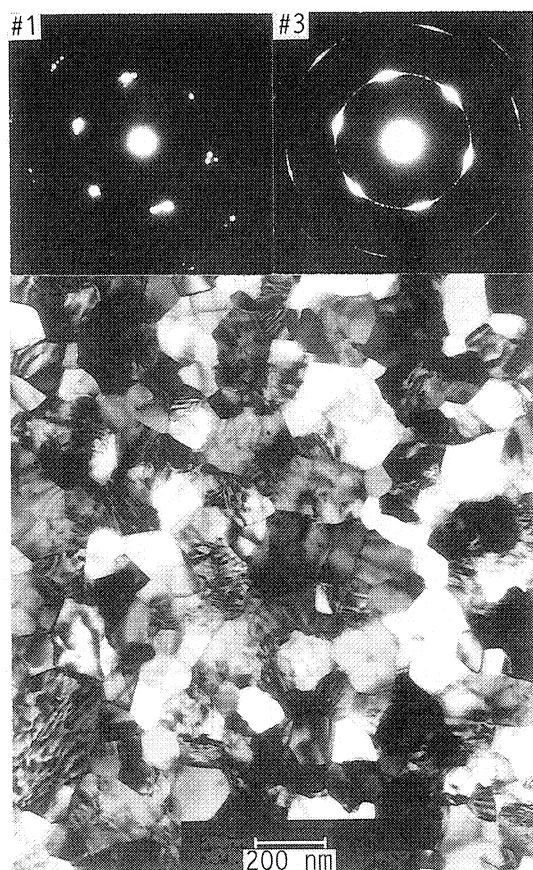
Aluminum (99.999%) was evaporated on heated mica sheets at a rate of 1.2 – 3.0 nm/s and the films were cooled (<90°C) in a vacuum (10<sup>-7</sup> Torr). The Al films were also prepared at 20°C on unheated mica. The TEM micrographs and TED patterns were taken with a Philips CM200. The Al films were peeled from the mica substrates and sandwiched between 2 grids. The TED patterns were taken with 3 different aperture settings and the actual diameters of the observation areas were 0.3, 1.2, and 6.0 μm. The AFM images were taken with a Digital Instruments NanoScope III.

The TEM micrograph and TED patterns of the Al film prepared at 20°C are shown in Figure 1. The TED patterns have 6-fold symmetry spots and typical patterns produced by the (111) face of a face centered cubic lattice.<sup>5</sup> The brightest spots in the TED pattern taken with the aperture #1 result from {220} reflection. As the aperture size increases, the spots become bigger, but still have 6-fold symmetry. The TEM micrograph and the TED patterns show that the film consists of small crystals with the (111) face and the grains have a preferential

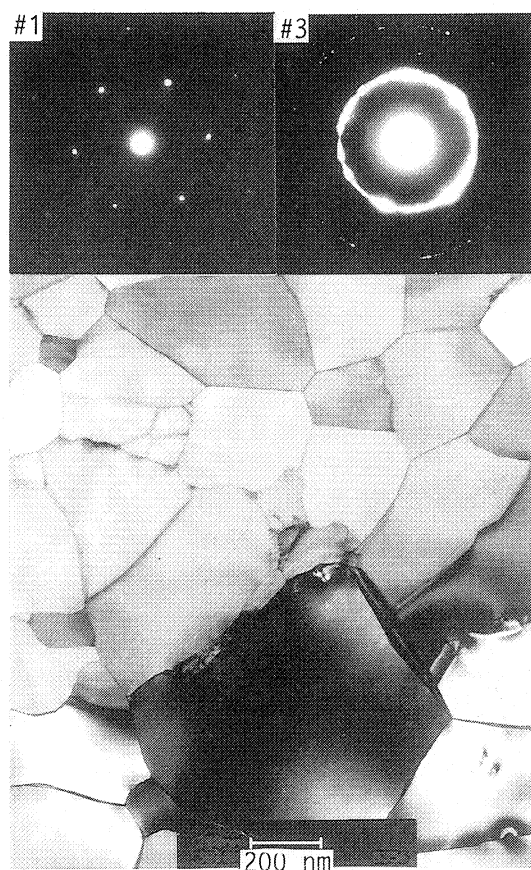
orientation on the mica substrate. The AFM surface plot of the Al film prepared on mica at 20°C is shown in Figure 2. The picture size is 1 × 1 μm wide and 20 nm height. The surface plot shows that the surface is very rough. The roughness analysis over the whole surface area indicates that the root-mean-square roughness (Rms) is 1.6 nm and the maximum height between the highest point and lowest point (Rmax) is 12.2 nm. There are small crystals about 100 nm in diameter and their tops are rounded. The film is typical polycrystalline and the structure is characterized by columns with domed tops. The surface morphology of the film is caused by self shadowing during the deposition, because the surface diffusion of adatoms is too small to diffuse into the shadowed regions at this temperature.<sup>2</sup>

The TEM micrograph and the TED patterns of the Al film prepared at 350°C is shown in Figure 3. The TED pattern (#1) has clear 6-fold symmetry spots and is a typical pattern produced by the (111) face of a single crystal. As the aperture size increases, the pattern becomes a ring. The TED patterns show that the film consists of relatively big crystals with (111) faces, but each crystal is oriented randomly about the [111] direction. The dark-field TEM micrograph shows almost the same contrast of all the crystals and indicates that the crystals are exactly oriented about the [111] direction which is perpendicular to the substrate. The Al film prepared on mica at 350°C is not epitaxial. The AFM surface plot of the Al film at 350°C is shown in Figure 4. It is clear that the crystals become bigger and the surfaces are very smooth. The Rms and Rmax values are 0.6 and 5.4 nm, respectively. These values are due to steps of a few atoms high rather than to roughness in the terraces. The crystal size is about 300 nm and their terraces are atomically smooth. Apparently, surface diffusion of the adatoms is sufficient to overcome the self-shadowing effect and it leads to the surface recrystallization and the atomically smooth surface at this temperature. This film will enable us to observe the morphology of adsorbed species on the surface. AFM provides valuable information on the surface morphology and the roughness of the films.

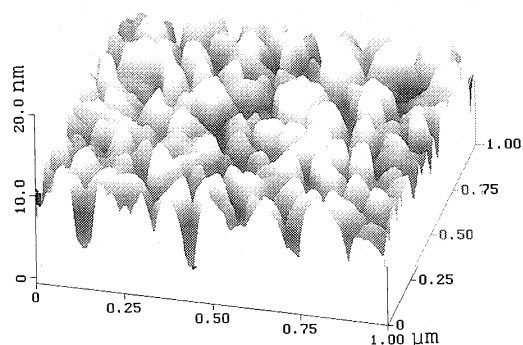
The present study was supported by the fellowship from the Ministry of Education of Japan. Instrumentation was provided through NSF grant 9205197.



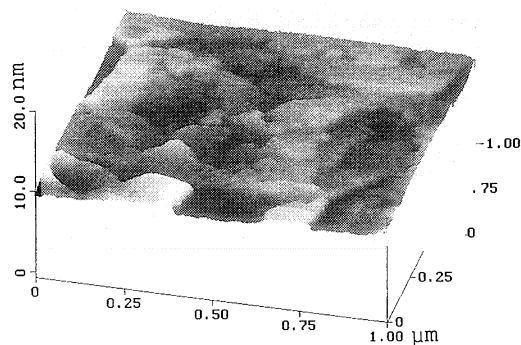
**Figure 1.** TEM micrograph and TED patterns (apertures #1 and 3) of an Al film (thickness: 180 nm) prepared on a mica substrate at 20°C.



**Figure 3.** TEM micrograph and TED patterns (apertures #1 and 3) of an Al film (thickness: 180 nm) prepared on a mica substrate at 350°C.



**Figure 2.** AFM surface plot of an Al film (180 nm) prepared on a mica substrate at 20°C.



**Figure 4.** AFM surface plot of an Al film (180 nm) prepared on a mica substrate at 350°C.

**Table 1.** Grain size (D) calculated from the TEM micrographs and Rms and Rmax obtained from the roughness analysis of the AFM images (1 × 1 μm) of the Al films deposited on mica substrates at the indicated temperatures (T)

T/°C	D/nm	Rms/nm	Rmax/nm
20	130 ± 50	1.6 ± 0.5	12.2 ± 2.8
350	270 ± 160	0.6 ± 0.2	5.4 ± 1.6

#### References

- 1 Y. Martin, "Selected Papers on Scanning Probe Microscopes," SPIE Opt. Eng. Press (1995).
- 2 B. A. Movchan and A. V. Demchishin, *Phys. Met. Metallogr.*, **28**, 83 (1969).
- 3 K. L. Westra, A. W. Mitchell, and D. J. Thomson, *J. Appl. Phys.*, **74**, 3608 (1993).
- 4 X. Lu, K. W. Hipps, X. D. Wang, and U. Mazur, *J. Am. Chem. Soc.*, **118**, 7197 (1996).
- 5 L. Reimer, "Transmission Electron Microscopy," Springer-Verlag (1993).