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ELASTIC PROPERTY DISTRIBUTION OF Al_2O_3 FILM FABRICATED BY AEROSOL DEPOSITION METHOD WITH NANO-SCALE RESOLUTION

Hisato Ogiso, Mikiko Yoshida, Jun Akedo
National Institute of Advanced Industrial Science and Technology (AIST),
1-2-1 Namiki, Tsukuba, Ibaraki 305-8564 Japan
Fax:81-29-861-7091, e-mail: ogiso.h@aist.go.jp

We present a fine structure in elastic properties distribution of the α-Al_2O_3 film fabricated by the aerosol deposition (AD) method. The structure was observed by measuring the resonance characteristics of the cantilever in contact with the surface (ultrasonic atomic force microscopy (UAFM) method). The UAFM method successfully displayed the elastic property distribution of the α-Al_2O_3 film with nano-scale resolution, and revealed specific regions which have smaller resonance frequency and lower Q value in the surface vibration characteristics than the surrounding area. The specific regions in resonance characteristics corresponded to the protrusions, 20~ 40 nm in height and 300 ~ 500 nm in diameter, observed by atomic force microscopy (AFM). This result suggests that the bond under the protruded regions be incomplete.

1. INTRODUCTION

Aerosol deposition method (AD method) is a novel technique to deposit relatively thick films under room temperature[1,2]. The AD Method enables to form ceramic film without sintering treatment only by colliding fine particles, <1μm, to substrates with aerosol condition. This feature has a great advantage to apply the AD method to a Micro Electro Mechanical System (MEMS) fabrication. The mechanism of the bonding formation among the particles, however, has not been clarified. Although the deposition was conducted under room temperature, the mechanical properties of the α-Al_2O_3 film fabricated by the AD method was nevertheless excellent as a whole. This properties indicate that the film by the AD method is not simply an aggregate of the particles. To clarify the bonding state among the particles, a microscopic characterization of the mechanical property is, therefore, inevitable, however has not been conducted.

Mechanical property measurement of thin films is generally difficult. A nano-indentation method is frequently used to evaluate hardness and elasticity. The method, however, is not suitable for imaging the spatial distribution. On the other hand, it has been developed that methods to measure elastic properties with high spatial resolution using an atomic force microscope (AFM). Force modulation microscopy (FMM) [3], widely used, is one of the approaches that elastic deformation by the micro-cantilever is detected by AC force modulation, where the the modulation frequency is lower than the resonance frequency of the cantilever. The FMM, however, is not suitable to measure the elasticity of the α-Al_2O_3 film, because it has low sensitivity to evaluate relatively stiff materials. The low sensitivity is due to that the cantilevers for the topography measurement are so soft as not to deform the surface in the quasi-static condition. From these difficulty, it has not been achieved to measure the elastic property distribution of stiff almina with high spatial resolution.

In this research, we therefore attempted to observe an α-Al_2O_3 film on a glass substrate fabricated by the AD method using ultrasonic atomic force microscopy (UAFM) which is a novel technique to examine the elastic property of hard materials with high spatial resolution [4-6]. The UAFM is a method to evaluate a local elastic property by measuring the resonance characteristics of the cantilever in contact with the surface. Figure 1 shows the principle of the UAFM. Since a vibrating cantilever induces elastic deformation of sample surface dynamically, the cantilever and the surface build a coupled oscillation system. The resonance characteristics, therefore, depends on the local surface elastic property. The advantage is that the UAFM has high sensitivity to stiff materials by using at higher order resonance [3].

2. EXPERIMENTAL PROCEDURE

An α-Al_2O_3 film was deposited on glass substrates using AD method. Figure 2 shows the schematic viewgraph of the AD method system, and the deposition

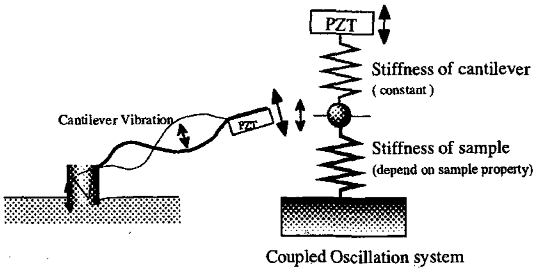

Fig. 1 Principle of UAFM

Table 1 Experimental conditions of a-Al_2O_3 deposition by AD method

Raw fine particles condition		Deposition Condition	
Average particle size	: 0.25 μm	Nozzle dimension	: 0.4 x 10 mm2
Average crystallite size	: 0.15 μm	Carrier Gas	: He
Purity	: 99.99 %	Gas flow rate	: 4 l/min
Crystal System	: α-Al_2O_3	Substrates temperature	: Room temperature
		Chamber pressure	: 0.8 torr
		Stage scanning velocity	: 1.25 mm/sec

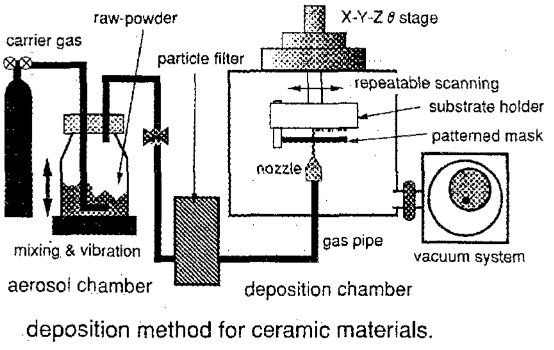

Fig. 2 Schematic view of AD method system.

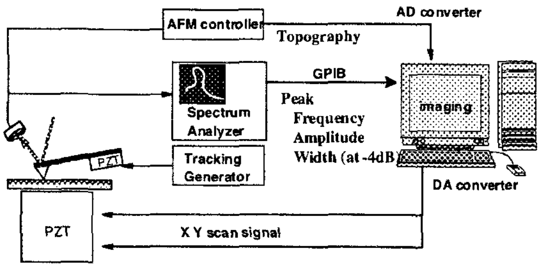

Fig. 3 Schematic view of ultrasonic acoustic force microscopy

conditions are shown in table 1. The area of the fabricated α-Al_2O_3 film was 10 x 10 mm^2, and the thickness was 6 μm.

The elastic property distribution was measured by UAFM system. Figure 3 shows the schematic view of UAFM measurement system. A commercial AFM (JEOL, JAFM-4210) was remodeled into the system. The used cantilever was a commercial product (Nanosensors Ltd. product), which is normally used for AC-mode detections, the tip was diamond coated, the spring constant was nominally 31-70 N/m. The sample position was X-Y scanned for imaging, the resonance frequency and the resonance peak width (at -3 dB) at every position (64 x 64 pixels^2) were measured, where the applied frequency was swept ranging from 860 kHz to 910 kHz. Besides the contact mode AFM image was simultaneously observed. It took about 1.5 s to measure the values at each position.

3. RESULT AND DISCUSSION

Figure 4 shows the images of α-Al_2O_3 fabricated by AD method using the UAFM. Figure 4a is the topographical image. The surface is flat as a whole, the root mean square of the surface was 16 nm. Two noticeable protrusions are found in the image, 300 - 500 nanometer in diameter and 20 - 40 nanometer in height. On the other hand, the regions in which the resonance frequency decreases and the width of resonance are broadened are found in Fig. 4b and 4c. Note that these regions coincident with the protrusions in Fig 4a. The low resonance frequency means that the sample surface has low stiffness at the contacted area, since the spring constant of the cantilever is constant during the measurement, indicating that the decrease of the resonance frequency can be attributed to the decrease of the stiffness of the sample surface. Similarly, the broadening of the resonance width, namely low Q value, indicates that the sample surface has a greater factor of energy dissipation in vibration. Therefore, the protrusions has lower stiffness and higher energy dissipation than the surroundings.

From the result, we propose a model which explains the surface condition of α-Al_2O_3 fabricated by AD method (Fig.5). Single Al_2O_3 crystal has very high Q value in vibration. Therefore, the decrease of Q value is caused by the bond imperfection at the grain boundary. Especially, in case of the Al_2O_3 films fabricated by AD method, it is not necessary that the bonds between particles should be perfect. Therefore, the protrusions are possibly identified with the particles which bond to the film imperfectly. It is noticeable that the findings of imperfection, however, did not mean that the film fabricated by AD method was very weak. The α-Al_2O_3 film actually achieved the vickers hardness of 14.7 GPa.

We would like to append a note about the spatial resolution of the UAFM observation. The resolution of the images in Fig 4 is actually 31 nm, which is attributed to the distance between the pixels, not to the limitation of UAFM method itself [6]. At present, the number of pixels, 64x64, are practically maximum, since it takes long time to observe one image. A faster data-acquisition system is then required.

4. CONCLUSION

To examine the microstructure of the α-Al_2O_3

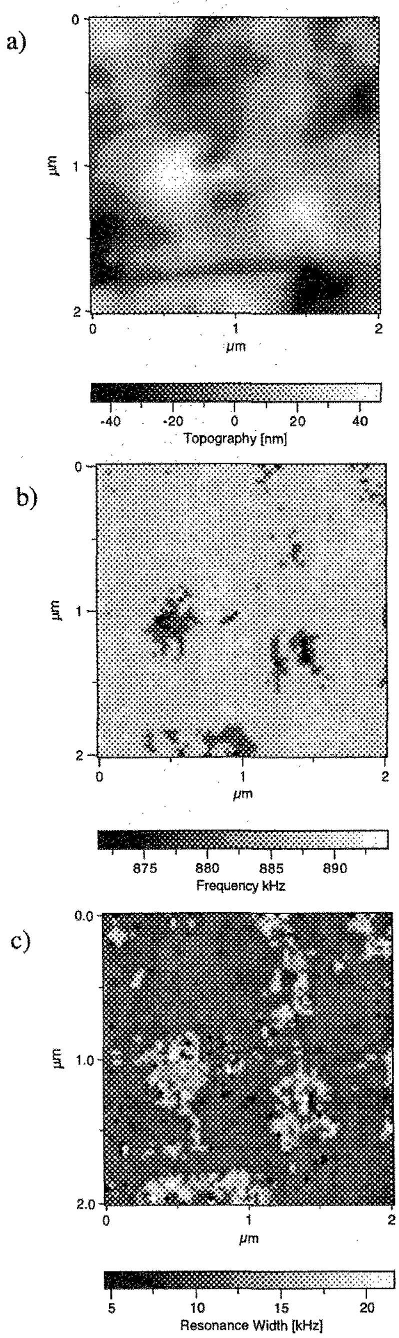

Fig. 4 Images of α-Al_2O_3 films fabricated by AD method using UAFM, a) topography, b) resonance frequency image, c) width of resonance (at -3 dB).

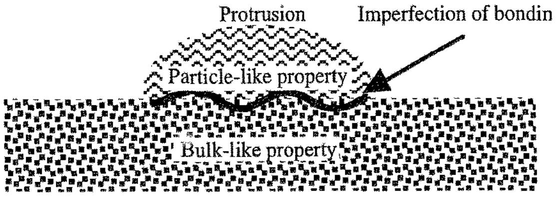

Fig. 5 Model of a-Al_2O_3 film fabricated AD method.

fabricated by AD method, we conducted an UAFM observation. The UAFM observation successfully enabled to measure the elastic property distribution of α-Al_2O_3 film with high spatial resolution, and revealed the domain structure of elastic properties with the size of several hundreds nanometer. The image indicated that some particles imperfectly bonded to the fabricated film. These findings show the effectiveness of the UAFM.

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