

Preparation of flat cross section of thin films by perforation fracture method

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Among many analyzing methods for thin films, atomic force microscopy (AFM) is one of a most powerful tool which enables to analyze local properties as well as microstructure observation in nm scale, but generally it is only applied to the surface of the films and not to the cross section of the films. One of a main reason preventing the cross sectional AFM analysis will be a difficulty in preparation of a flat cross section. In the present study, the authors demonstrate a novel method for obtaining a flat cross section of stacked films deposited on substrate, i.e. perforation fracture method, where preliminary prepared pits by focused ion beam (FIB) on the surface promote controlled crack propagation and flat cross section of the film is obtained. As a practical example, preparation of cross section and AFM observation for a BT/LNO film deposited on a Pt/Ti/SiO₂/Si substrate is introduced.

Key-words : Film, Cross section, Perforation, Fracture, AFM, FIB

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1. Introduction

There have been reported a lot of research works of ceramics materials in thin film form.¹⁻⁵ As a basic approach for investigating microstructure of the film is microscopic techniques such as atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), etc. Among these methods, AFM enables us to analyze variety of different properties (e.g. electrical, electrophysical, electrochemical, magnetic, etc.) by choosing appropriate cantilevers and measurement methods.⁶⁻⁷ Generally, AFM is used only for surface analysis and observation of the film and a cross sectional analysis is usually done by SEM and/or TEM. If the AFM is applicable for the cross section of the films, further informative analyses becomes available, e.g. local analyses at the interface between the film and substrate, etc. The authors have reported an ion milling method for obtaining the cross section.⁹ The ion milling method is suitable for obtaining flat cross section but it takes considerably long time of ca. 10 h for the ion milling and multiple steps, i.e. cutting, mechanical polishing, and ion milling are needed. For the easy preparation of the cross section, a simple cracking method will be the better way to obtain the cross section of the films.

The authors have tried the AFM observation on the cracked cross section of the film on Si (001) substrate by a conventional way, which is merely cracking the Si substrate along the cleavage direction, [110], of Si after scratching the back surface using a diamond-point pen, but such observation was difficult due to high roughness of the cross section. This method is suitable for the cross sectional SEM imaging, since the cracking is the easiest way to prepare the cross section, but such cross section is too much rough and not suitable for AFM observation. One may consider the flat cross section can be easily obtained using the focused ion beam (FIB) method, but such cross section polished using heavy Ga ion leads considerable damage (amorphization, contamination, redeposition, etc.) on it⁹ and will not be suitable for the analysis using AFM which directly observes the properties of the cross section. From the view point of low damage preparation of the cross section, the cracking method is the ideal process.

There have been only a few reports about the cross sectional AFM analysis in which the cross section of the film was prepared by cracking method.¹⁰⁻¹³ In those cases, their film samples are suitable for the cracking method since they are amorphous film or their cleavage plane is identical to that of the substrate. However, most of the thin films in practical use is not only such films
but composed of multiple layers and components without specific lattice orientation suitable for cleavage and it results in the high roughness of the cross section due to the uncontrolled crack propagation. In order to obtain flat cross section for such films, controlling crack propagation is required.

In the present study, for controlling the crack propagation of the film, a row of pits was preliminary prepared by the FIB on the film on the substrate. The pits are expected to act as a guide for the crack propagation similar to the perforation which is generally used for the easy cutting of papers, tickets, etc. without using a knife or scissors. The cracked cross section at the region between the pits is expected to become flat and low damaged, and therefore suitable for the cross sectional AFM observation.

2. Experimental

In the present study, perforation and fracture method was demonstrated for a bare and a film deposited Pt/Ti/SiO$_2$/Si(001) substrate. The deposited film was BaTiO$_3$ (BT) and LaNiO$_3$ (LNO) prepared by chemical solution deposition method. Details of the preparation method and the electrical properties of the films have been reported in our previous studies. First, the bare substrate was used to optimize the sizes and intervals of the pits for obtaining the flat cross section (the size and intervals of the pits are shown in Table 1). The pits for the perforation was prepared by FIB (JIB4500, JEOL Ltd., Japan) using 30.0 nC/cm$^2$ of Ga$^+$ ion beam irradiated on the surface of the film. The substrate after the pit prepared was cracked as schematically shown in the Fig. 1. In order to initiate the origin of the crack at the edge of the substrate, a tiny scratch was made by a diamond point pen at the one end of the perforation prior to the fracture. The obtained cross section of the substrate was observed by AFM using a perpendicular specimen holder, which can tightly hold the substrate with a screw as shown in the Fig. 3. Cross sectional AFM images of the substrates prepared by the perforation fracture method was shown in Figs. 4(a) to 4(c). A cross sectional AFM image of the same substrate prepared by conventional fracture method (without perforation) was shown in Fig. 4(d). An SEM image of the same substrate was shown in Fig. 4(e) for comparison, where the thickness of the Pt is about 180 nm. Comparing the images (a) to (c) with (d), it is obviously understood that the top surface of the substrate was not clearly confirmed in the specimen prepared by a conventional fracture method, whereas the substrates perforation fractured shows relatively obvious edge of the top surface. Among the three specimens with different pit sizes and different intervals, differences in the root mean square (RMS) roughness near the edge of the top surface arose. The RMS values obtained at the square regions indicated by the red dashed line in the images (a), (b), and (c) were (a) 51 nm, (b) 33 nm, and (c) 6 nm, respectively. From the results, the authors considered that the larger pit

| Table 1. Pit preparation condition by FIB |
|---------------------------------------------|
| Ga ion beam current/nA | 30.0 |
| Acceleration voltage/kV | 30.0 |
| Pit width/µm | 2 |
| Pit length/µm | 10, 15, 20 |
| Interval between pits/µm | 10, 15, 20 |
| Dose of Ga ion for each pit/nC/µm$^2$ | 30.0 |

that the line structure was prepared without causing major damage to the sample. From the cross sectional SEM images, it is understood that the perforation considerably acted as a guide of the crack propagation since the cross section of the pit were obviously observed. Although the dose of the Ga$^+$ ion was same for the all three sizes of the pits, the depth of the pit was different. The reason of the depth difference will be re-deposition during the FIB processing, i.e. the small pit tended to generate the re-deposition on the wall of the pit whereas the larger pit could reduce the re-deposition. It is worth to note that not only the depth of the pits, but also the shape of the pit is sharper for the larger pits.

For the cross sectional AFM observation, tight holding of the specimen was important to obtain high resolution AFM image with high reproducibility. The fractured substrate was observed by AFM using a perpendicular specimen holder, which can tightly hold the substrate with a screw as shown in the Fig. 3. Cross sectional AFM images of the substrates prepared by the perforation fracture method was shown in Figs. 4(a) to 4(c). A cross sectional AFM image of the same substrate prepared by conventional fracture method (without perforation) was shown in Fig. 4(d). An SEM image of the same substrate was shown in Fig. 4(e) for comparison, where the thickness of the Pt is about 180 nm. Comparing the images (a) to (c) with (d), it is obviously understood that the top surface of the substrate was not clearly confirmed in the specimen prepared by a conventional fracture method, whereas the substrates perforation fractured shows relatively obvious edge of the top surface. Among the three specimens with different pit sizes and different intervals, differences in the root mean square (RMS) roughness near the edge of the top surface arose. The RMS values obtained at the square regions indicated by the red dashed line in the images (a), (b), and (c) were (a) 51 nm, (b) 33 nm, and (c) 6 nm, respectively. From the results, the authors considered that the larger pit

Figure 2 shows SEM images of the typical pits prepared by FIB [(a) to (c)] and the cross section of the substrate obtained by the perforation fracture method [(d) to (f)]. From the surface observation, one can understand

![Fig. 1. Schematic of the pits prepared by FIB on the surface of the substrate (a) and fracture along the perforation (pit) prepared by FIB.](image-url)
size and larger intervals resulted in the flatter cross section. Regarding the intervals between the pits, the shorter interval was expected to result in the flatter interface since the unguided region is short, but the larger intervals resulted in the flat cross section. The reason of this tendency might be caused by the depth and sharpness of the pits prepared by FIB since the deeper pit and also the sharper edge of the pit is considered to enhance tensile stress at the edge of the pit. We consider that among the three pit preparation conditions, the pit depth is more effective to obtain the flat cross section.

From the RMS values, the authors set the pit size and intervals of 20 µm as the optimum perforation condition. The optimum pits were then prepared on the BT/LNO...
films deposited on the same substrate and the cross section was prepared. Cross sectional AFM image of the film was shown in Fig. 5(a) and 5(b) as well as the cross sectional SEM of the same specimen (c). From the cross sectional AFM images, one can easily recognize the film on the Si substrate and the thickness of the film was also identical to the one observed in the SEM. It is not very clearly observed, but the cross sectional AFM images of the BT film look like relatively columnar structure and the Pt and LNO regions are relatively granular and porous. These structures much well with the SEM image and also our previous reports. From these results, it was successfully demonstrated that the perforation fracture method was effective to obtain a flat cross section of the stacked films. The authors are expecting further AFM analysis (electrical conductivity, ferroelectricity, etc.) will be done in the future on the fractured cross section of the perforation fracture method.

4. Conclusion

As a novel method for obtaining a flat cross section of the stacked films on the substrate, perforation fracture method was demonstrated for BT/LNO film on Pt/Ti/SiO2/Si substrate. By preparing pits on the surface of the film by FIB, crack propagation was effectively controlled and flat cross section was obtained compared to the one without pits preparation. The optimum size and intervals of the pits in the present study for obtaining flat cross section for comparison, which was obtained with a conventional fracture method. One can recognize that the similar thickness and similar microstructure of the film was observed by SEM and AFM.

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