UDK 620.18:543.2:669.715
Professional article/Strokovni članek

ISSN 1580-2949
MTAEC9, 42(1)45(2008)

USING A FIB TO PREPARE $Al(OH)_3$ SAMPLES FOR THE TEM

UPORABA FIB ZA PRIPRAVO VZORCEV $Al(OH)_3$ ZA TEM

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Prejem rokopisa – received: 2007-09-25; sprejem za objavo – accepted for publication: 2007-10-23

One of the basic applications of a focused ion beam (FIB) tool is the preparation of samples for transmission electron microscopy (TEM) analysis. A dual-beam FIB system combines multiple experimental systems within the same chamber and the combinations of different techniques can be accommodated by unique sample preparation, manipulation and analysis methods. The system contains both the focused Ga+ ion beam and a field-emission scanning electron column. In this paper we described a procedure for preparing $Al(OH)_3$ samples for TEM analysis using a FIB.

Key words: focused ion beam, $Al(OH)_3$, milling, deposition, sample preparation

Ena od temeljnih vrst uporabe fokusiranega ionskega curka (FIB) je priprava vzorcev za presevno elektronsko mikroskopijo. Dualni FIB-sistem kombinira v isti komori več eksperimentalnih sistemov, kombinacija različnih tehnik pa omogoča enkratno pripravo vzorcev, manipulacijo in analizo. Sistem ima oboje: fokusirani in ozki curek Ga^+ in vrstično elektronsko kolono s poljsko emisijo. V članku je opisan postopek uporabe FIB za pripravo vzorcev $Al(OH)_3$ za presevno elektronsko mikroskopijo.

Ključne besede: fokusirani ionski curek, $Al(OH)_3$, mletje, depozicija, priprava vzorcev

1 INTRODUCTION

The two main features of the FIB tool are the capability to remove material from the sample by sputtering (micro-machining) and to add materials to the sample by depositing at sub-micron dimensions. There are a wide variety of applications for a FIB, but they can be segmented into three main categories [1-3], i.e.,

- IC review and modification, where sputtering is used to create a cross-section or modification to the sample, and where cutting is desired together with the deposition of either metallic or insulating materials to either modify existing structures or to create new structures.
- TEM and STEM sample preparation, where the FIB is used to form a thin slice of material by sputtering trenches on either side of a slice.
- Producing thin-film specimens.

In this paper we explain a step-by-step approach to preparing samples of $Al(OH)_3$ for an investigation with transmission electron microscopy using an FEI Strata Dual Beam FIB. Investigating the mechanism of $Al(OH)_3$ crystal growth required the use of both scanning and transmission electron microscopy (SEM and TEM). The data obtained with the SEM are not sufficient to draw a reliable conclusion about the mechanism of $Al(OH)_3$ growth, with respect features such as twinning, and a determination of the twinning plane and direction. This means that TEM is required and a special way of preparing samples for such an investigation needs to be developed.

2 EXPERIMENT

The crystals of $Al(OH)_3$ were obtained by a crystallization process from a caustic soda solution. They crystallized in the form of small particles joined together in larger agglomerates. When such particles were exposed to the ion beam, they started to dissipate. In order to prevent the dissipation process we put $Al(OH)_3$ powder in the cannon mod and then filled it up with liquid epoxy. The vacuum, liquid epoxy filled up all the vacancies inside the $Al(OH)_3$ crystals. After 12 h the epoxy was solidified and made the particle interior stronger. After this, the cannon samples were polished in order to remove excess epoxy and coated with gold so they were conductive. The samples prepared in such a way were ready for the FIB treatment.

3 DISCUSSION

The preparation of the $Al(OH)_3$ sample for the TEM analysis using the FIB was carried out in the National Center for Electron Microscopy, Lawrence Berkeley Laboratory, University of Berkeley, California. A picture of the FIB is shown in the **Figure 1**.

Figure 2 shows a step-by-step approach to the preparation of a sample for transmission electron microscopy using the selective focused ion beam (FIB) milling technique.

After the area of interest is found, the first step is the deposition of platinum. The platinum is deposited, either under an ion or electron beam, in the strip with dimensions of approximately 25 x 1 x 2) µm (length,

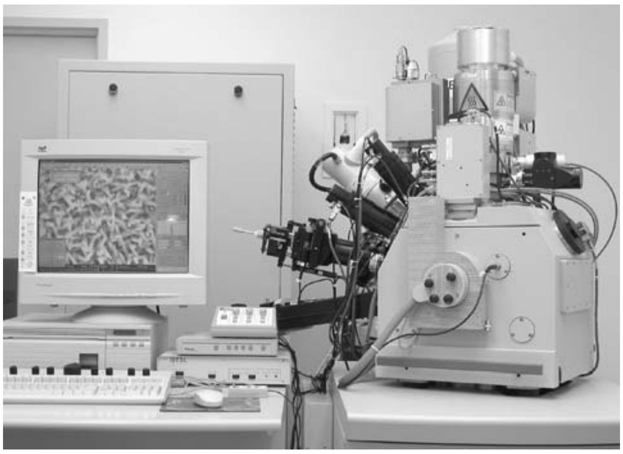

Figure 1: FEI Strata Dual Beam FIB in NCEM LBNL, University of California, Berkeley
Slika 1: Strata dualni curek FIB pri NCEM LBNL, Univerza v Kaliforniji, Berkeley

width, height). We used an ion beam with a beam current corresponding to double the area of the deposition (**Figure 2a**).

The next step is milling of the cleanup trenches on both sides of a platinum strip (**Figure 2b**). Before the milling procedure begins, the milling stair step pattern needs to be defined in terms of shape and position. The shape of the stair-step pattern is shown in **Figure 3**, [1].

The milling procedure consists of two steps. A large beam current (20 000 pA) is used in the first step of the bulk removal with the aim to reduce the time of milling when the ion beam is positioned far from the platinum strip. In the next step, after the bulk of the material has been removed, the face of the specimen slice must be cleaned up using a lower beam current. Again, a stair step pattern must be defined. After the final milling the platinum strip must retain the defined thickness.

After the final milling is finished, the sample is tilted by 45° in order to make a cross-section of the obtained sample slice. Under a beam current of 7000 pA a thin slice of the specimen is cut free from the three sides leaving a small "bridge" in the upper-left corner (**Figure 2c**).

In the next stage (**Figure 2d**), the sample stage in the chamber (z-position) is lowered (to avoid any needle damage) and a platinum needle is inserted into the chamber. Then, the sample stage is raised again. Using a manipulator the Pt needle is navigated and attached to the free upper (right) corner. The needle is welded to the specimen slice by depositing platinum in the same way as in the case of the deposition of a platinum strip. For this test a smaller pattern (approximately (1x1) µm) and a lower beam current were used. Finally, the remaining bridge connecting the slice sample to the sample bulk is cut free using the 300 pA beam current. In the next step the sample stage in the chamber must be lowered slowly to lift out the membrane – the thin specimen slice.

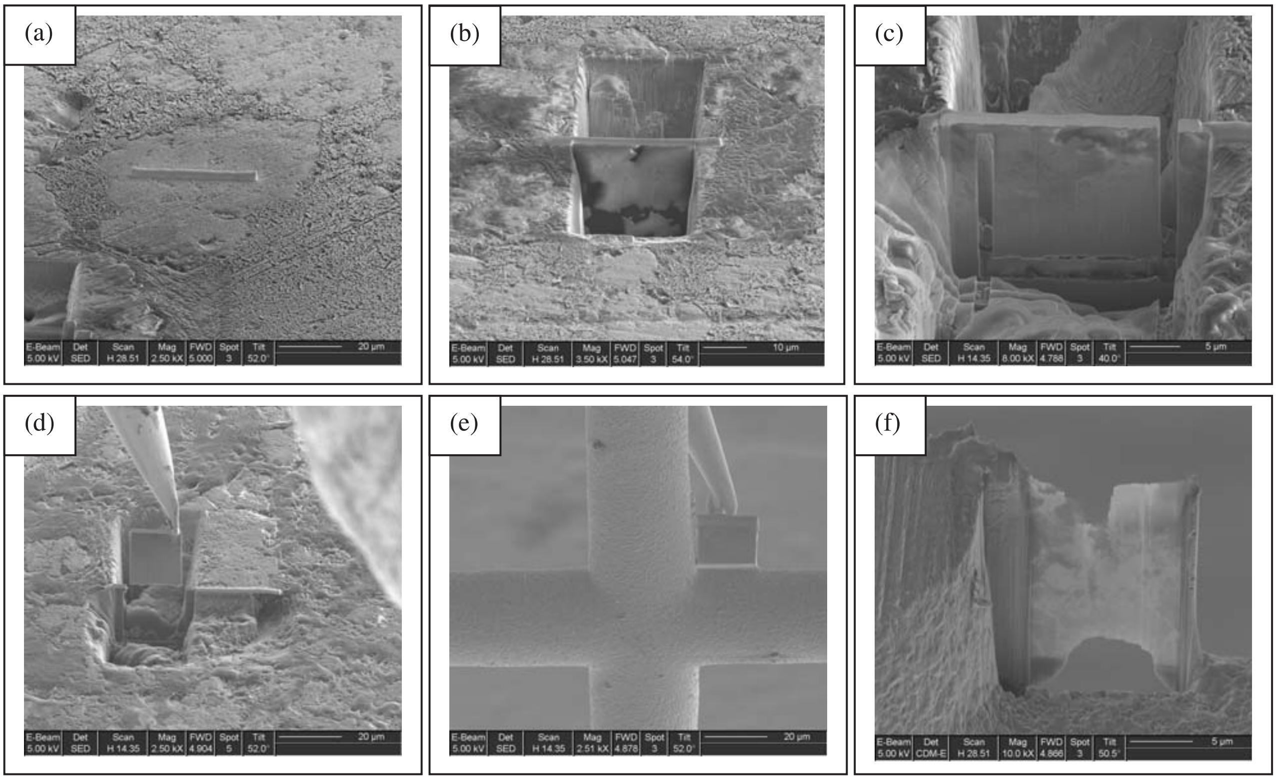

Figure 2: Step-by-step approach to preparing $Al(OH)_3$ samples for TEM using a FIB
Slika 2: Stopnje priprave vzorcev za TEM z uporabo FIB

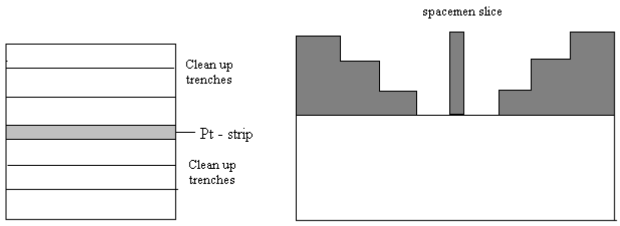

Figure 3: Shape of stair-step pattern used for the bulk removal of material
Slika 3: Shema stopničastega načina odvzema materiala

The last sequence is welding the membrane to the half-cut copper grid. Prior to this the copper grid is placed in the holder in the chamber and aligned horizontally. The membrane is then placed in a pre-milled trench located near the flat side of the grid and welded to the grid with platinum deposition (**Figure 2e**). When the membrane is welded to the grid, the platinum needle is cut free from the membrane. After this step the specimen slice is still too thick for any TEM analysis and the bulk of the FIB dust (the result of milling) is present on the surface of the membrane. Thus, a final thinning on the grid is necessary and, indeed, vital; this is the last step in the preparation of the $Al(OH)_3$ sample for TEM analysis. There is, however, a final step; it is good practice to let the ion beam scan (for a short time) the thin section using a low beam current (50–100 pA). The picture of the sample after the final thinning on the grid is shown in **Figure 2f**. Such a thin sample (about 50 nm) is appropriate for a TEM investigation.

4 CONCLUSION

The application of a focused ion beam in the preparation of $Al(OH)_3$ crystals for TEM is shown to be a good technique. Problems related to the dissipation of $Al(OH)_3$ particles under the ion beam can be successfully avoided by using epoxy. A general problem related to the FIB technique is the re-deposition of dust (removed material) during the milling step. In addition, the problem that may occur during the preparation of the sample for the TEM analysis is related to the last steps when the sample slice is very thin and the handling needs to be very delicate. The procedure of cleaning the sample slice on the grid must be carefully performed with the choice of an appropriate beam current. The lower beam current is recommended, as is the repetition of the cleaning procedure several times until the desired sample thickness is achieved. If the sample is too thin the sample slice may break.

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