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Kyoto University
Observation of electromigration in a Cu thin line by in situ coherent x-ray diffraction microscopy

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Electromigration (EM) in a 1-μm-thick Cu thin line was investigated by in situ coherent x-ray diffraction microscopy (CXDM). Characteristic x-ray speckle patterns due to both EM-induced voids and thermal deformation in the thin line were observed in the coherent x-ray diffraction patterns. Both parts of the voids and the deformation were successfully visualized in the images reconstructed from the diffraction patterns. This result not only represents the first demonstration of the visualization of structural changes in metallic materials by in situ CXDM but is also an important step toward studying the structural dynamics of nanomaterials using x-ray free-electron lasers in the near future. © 2009 American Institute of Physics. [DOI: 10.1063/1.3151855]

I. INTRODUCTION

Atomic diffusion caused by momentum transfer from conducting electrons is a phenomenon known as electromigration (EM).1 Under a high current density of ~106 A/cm², voids and hillocks form around the interconnecting wires due to EM and often result in the failure of Cu wiring layers in large-scale integration (LSI) circuits.2,3 To enhance the reliability of LSI circuits and design new LSI structures, understanding the mechanism of deterioration in Cu wiring layers is important. The evaluation of structural changes in wiring layers induced by EM has been carried out by electron microscopy, x-ray microscopy,4,5 and x-ray microdiffraction.6 Transmission electron microscopy (TEM) is a well-established microscopy technique with atomic resolution. Recently, EM-induced surface atomic steps in Cu lines have been observed by in situ TEM.7 TEM is a powerful tool for evaluating local structures in metallic foils thinner than a few hundred nanometers. Hard x-ray microscopy equipped with a Fresnel zone plate is a promising method of x-ray microscopy and was recently developed at synchrotron x-ray radiation facilities, which can currently achieve about 30 nm resolution.8 Until now, both Cu thin lines and EM-induced voids in a LSI structure have been three-dimensionally observed by hard x-ray tomographic microscopy.9,10 Mass transport of the early stages of EM in a Cu line has been investigated by dynamical x-ray microscopy.5 Polychromatic microdiffraction equipped with Kirkpatrick–Baez mirrors is also a powerful tool for characterizing mesoscopic length scale structures of polycrystalline materials.6 Until now,

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which has been developed at SPring-8, as an important step toward the use of XFELs.

II. EXPERIMENT

Figure 1(a) shows a scanning electron microscope (SEM) image of the Cu thin line sample. A 1-μm-thick Cu film was deposited on a 270-nm-thick SiN membrane by electron-beam evaporation using a mask.\cite{34} 1-μm-wide thin lines and 300-nm-wide adjacent connections, which simulate via structures in LSI circuits, were fabricated in the Cu film using a focused ion beam. The Cu/SiN in a 60×60 μm² area around the connections was completely removed to avoid scattering x rays from the membrane. Figure 1(b) shows a schematic of the system used for applying dc to the thin line sample during CXDM measurements. The SiN membrane chip supporting the Cu thin line was held between a Bakelite base and acrylic resin clips. The sample holder was mounted on a stage in the vacuum chamber. A dc of 32 mA was applied to the Cu thin line via Cu wires from the outside the chamber. Estimated current density was 3.2×10⁶ A/cm² for the 1-μm-wide thin lines and 5.3×10⁶ A/cm² for the 300-nm-wide adjacent connections. Figure 1(c) shows the time dependence of the increase in resistance of the Cu thin line when a dc of 32 mA was applied.

III. RESULTS AND DISCUSSION

Figures 2(a)–2(d) show the coherent x-ray diffraction patterns of the Cu thin lines when the increases in resistance were Δ0.3, Δ0.7, Δ1.5, and Δ2.0 Ω. To the right of each diffraction pattern, a closeup of the pattern surrounded by a white square is displayed in a different color. Since the diffraction patterns measured at Δ0, Δ0.05, Δ0.1, and Δ0.3 Ω were almost identical and those at Δ1.0 and Δ1.5 Ω were also the same, these diffraction patterns are represented by those at Δ0.3 and Δ1.5 Ω, respectively, in Fig. 2. In the diffraction pattern at Δ0.3 Ω, the speckle patterns spreading to the upper right and lower left result from the 300-nm-wide adjacent connections in the sample. Similar speckle patterns can also be seen in the other diffraction patterns. The direction in which the speckle patterns spread rotates clockwise as the resistance increases, which is schematically drawn in Fig. 2(e). This implies that the adjacent connections leaned right as the resistance increased. In the diffraction patterns at Δ0.7 Ω and higher, characteristic speckle patterns that were not observed at Δ0.3 Ω can be seen in the upper left and lower right directions. A part of the characteristic speckles is colored and the directions are shown by dotted lines. The process of change in both the speckles and the directions is schematically drawn in Fig. 2(e). The size of the speckles decreases as the resistance increases. The direction in which the speckle patterns spread and the speckles themselves rotate counterclockwise as the resistance increases. This implies that a characteristic structure was produced in the thin line between Δ0.3 and Δ0.7 Ω, and its size increased and shape changed as the resistance increased. The coherent x-ray diffraction intensity corresponds to the magnitude of the Fourier transformation of the projection image of the sample. Both the size and shape of the structure can be estimated from the characteristic speckle. Table I summarizes the estimated structures. The structure is an EM-induced void in the thin line. Many line-shaped speckles can be seen in the diffraction pattern at Δ0.3 Ω, as indicated by the black arrows in Fig. 2(a). As the resistance increases, the line-
To evaluate the process of the EM-induced structural changes in the thin line, images were reconstructed from the diffraction patterns. Even if the sample is one-dimensionally long such as the thin line, when it has a specific structure that is the ladderlike shape in the present sample and the two-dimensional oversampling ratio, which was \( \sim 25 \) in the present measurement, is more than 2, it is possible to reconstruct sample images by using an exact support which is the nonzero region in the real space. The reconstruction was carried out by the following procedure: (i) a random-complex-number array in real space was generated with a size of \( 1201 \times 1201 \) pixels. A fast Fourier transform (FFT) was applied to the real-space array. The magnitudes of the output complex values in the experimentally measured region were replaced with the diffraction data, while the maximum value of the diffraction data was set in every pixel of the central region, which was unmeasured region because of the presence of a direct beamstop. The reconstruction was imperfect, it was confirmed by a simulation that voids around the ladderlike shape can be visualized.

Figures 3(a) and 3(b) show SEM images of the thin line observed before and after measuring all the diffraction patterns, respectively. EM-induced voids are formed at dark ar-

TABLE I. Relationship between the characteristics speckles observed at \( \Delta 0.7, \Delta 1.5, \) and \( \Delta 2.0 \) \( \Omega \) and the objects estimated from the speckles.

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<tr>
<th>( \Delta V )</th>
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<th>Real space</th>
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<tr>
<td>( \Delta 0.7 )</td>
<td>[195 pix, 188 pix]</td>
<td>[410 nm, 410 nm]</td>
</tr>
<tr>
<td>( \Delta 1.5 )</td>
<td>[115 pix, 115 pix]</td>
<td>[192 nm, 192 nm]</td>
</tr>
<tr>
<td>( \Delta 2.0 )</td>
<td>[154 pix, 154 pix]</td>
<td>[220 nm, 220 nm]</td>
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shaped speckles disappear, and the large speckles are produced, which implies that the width of a part of the thin line decreased as the resistance increased.

As the resistance increased, the width of a part of the thin line decreased as the resistance increased.

FIG. 2. (Color) Coherent x-ray diffraction patterns of the Cu thin line in \( 1201 \times 1201 \) pixels when the increases in resistance are (a) \( \Delta 0.3 \), (b) \( \Delta 0.7 \), (c) \( \Delta 1.5 \), and (d) \( \Delta 2.0 \) \( \Omega \). The pixel size is \( 1.73 \times 10^{-4} \) nm\(^{-1}\). To the right of each diffraction pattern, a closeup of the pattern in 200 × 200 pixels surrounded by the white square is displayed in a different color. The central \( \sim 25 \times 25 \) pixels, which are displayed in white, are not measured due to the beamstop. The directions of speckle patterns spreading to the upper right are traced by solid lines. In the patterns of (b)–(d), speckle patterns spreading to the upper left are colored, and the directions are traced by dotted lines. (e) The relationship between the colored speckles and the lines traced in (a)–(d) is schematically drawn.
The size and shape of the void indicated by the images reconstructed from the diffraction patterns at other areas indicated by red and pink arrows in Fig. 3 (Color) [(a) and (b)] SEM images of the Cu thin line observed (a) before and (b) after measuring all the diffraction patterns. A dc was applied to the thin line from the upper side along the white arrow. (c) Image in 200×800 pixels reconstructed from diffraction pattern of Fig. 2(a). The pixel size is 30.2 nm. The image is displayed in grayscale. [(d)–(g)] Images in 200×200 pixels reconstructed from diffraction patterns of Figs. 2(a)–2(d), respectively, in the area surrounded by the blue square in (c). Pixels with intensity greater than 50% of the maximum intensity in each reconstructed image are displayed in white.

The average brilliance of x rays radiated from in-vacuum undulator installed at BL29XUL in SPring-8 is $\sim 10^{20}$ photons/mm$^2$ mrad$^2$ s in 0.1% bandwidth, while that of the Japanese XFEL is estimated to be $\sim 10^{21}$ photons/mm$^2$ mrad$^2$ s in 0.1% bandwidth. Therefore, if the dynamic range of CCD detectors is improved and the readout time becomes short, the diffraction data can be collected quickly ten times. Moreover, coherent high dense x rays can be produced by focusing XFEL because it has almost full spatial coherence. When x-ray beams are focused, the x-ray illumination area becomes small. For the present method, the maximum value of the size of samples is limited by the focusing spot size. Therefore, according to the size of samples, it is effective to use ptychography or keyhole imaging. As the result, measurement time can be shortened further. On the other hand, radiation damage of the sample will be serious. When the sample is irradiated by high-peak brilliance x rays such as XFEL pulse x rays, it is not understood how the sample is changed. At least, metallic samples should be more resistant to radiation than biological samples.

In the present study, the small angle x-ray scattering data were collected, which offered electron density distribution of the Cu thin line, while the coherent diffraction intensity data near the Bragg peaks offer strain distribution in addition to electron density distribution. For the latter method, the sample is limited to the single crystal or the single grain in a polycrystal. For polycrystalline samples such as the present thin line, CXDM using Bragg peaks is useful to evaluate local structures in samples, such as strain distribution in a single grain near an interconnect.

IV. CONCLUSION

In situ CXDM under the application of a dc was developed at SPring-8 as a step toward the use of XFELs and was applied to evaluate EM in a 1-μm-thick Cu thin line. Both EM-induced voids and thermal deformation in the thin line were made to appear characteristic x-ray speckle patterns in the coherent x-ray diffraction patterns, which were successfully visualized in the images reconstructed from the diffraction patterns. The present result not only represents the first demonstration of the visualization of structural changes in metallic materials by in situ CXDM but is also an important step toward studying the structural dynamics of nanomaterials using XFELs in the near future. To trace faster EM process and to examine the three-dimensional structure of EM-induced voids with better resolution, it is necessary to use more brilliant and almost fully coherent x rays such as those from XFEL. We believe that in situ CXDM will be established as a technique for evaluating EM in LSI circuits in the near future.

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32Technical design report of the European XFEL (http://xfel.desy.de/dr/ddr/).
39See light sources overview in SPring-8 website http://www.spring8.or.jp.