Microstructural Evolution at Room Temperature in Electrodeposited Copper Metallization

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Understanding and controlling the microstructure of conductor materials such as electrodeposited copper[1-5] for metallization is increasingly important. It has been observed that the microstructure of both sputtered and electrodeposited copper can change considerably with time, even at room temperature. Work reported to date has generally emphasized the evolution of the grain structure to larger grain size (i.e. grain growth) with corrresponding changes in film characteristics such as a decrease in resistivity. However, recently we reported[6] the observation of room temperature recrystallization of electrodeposited copper metallization in real time by atomic force microscopy (AFM) and found that the nucleated grains appeared to be significantly smaller than the existing grains. This may have important implications for surface reactivity and the stability and evolution of microstructure. The results closely reflected the classical three stages of annealing: recovery, recrystallization and grain growth. Thus, an induction period was observed before the onset of recrystallization and subsequently grain growth was found to occur. This paper will present further results on the microstructural evolution of electrodeposited copper.

AFM images of the surface of a typical 31 nm copper film electrodeposited on a gold substrate from an acidic $CuSO_4$ bath with 1.3×10^{-4} mol dm^{-3} thiourea are shown in Fig 1(a-c). The scan time for each image is 110 s. Fig 1(a) was imaged beginning 360 s after electrodeposition and shows the surface morphology for an as-deposited film. Typical feature size is 120 - 180 nm. Fig 1(b) shows the same area imaged beginning 800 s after electrodeposition. The feature size is now observed to be considerably smaller, typically 40 - 90 nm. This indicates that room temperature recrystallization has already occurred at this time and that the nucleated grains are significantly smaller than the as the as-deposited grains. Subsequent images showed growth of these grains. In fact, a secondary recrystallization of these grains was also observed, again followed by grain growth. Fig 1(c) shows an image of the same sample obtained 7 days later. Clearly, significant grain growth has occurred.

Scaling analysis of AFM images before and after recrystallization will be presented which shows a decrease in characteristic length and an increase in roughness. Results of the ongoing investigation using other techniques including x-ray diffraction, focussed ion beam imaging and resistance measurements will be discussed.

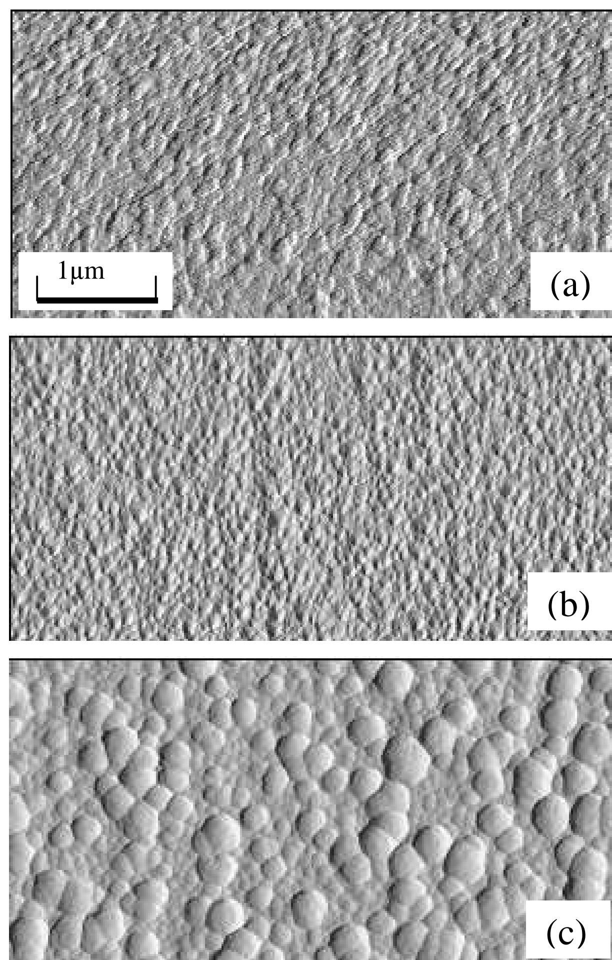

Fig. 1. AFM images of 31 nm electrodeposited copper film (a) 360 s (b) 800 s and (c) 7 days after electrodeposition.

References

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