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Gallium Phase Formation in Cu and Other FCC Metals During Near-Normal Incidence Ga-FIB Milling and Techniques to Avoid this Phenomenon

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Focused ion beam (FIB) circuit modification of devices with Cu-based interconnect is well known to be problematic [1, 2] due to the vast difference in sputter rate as a function of Cu grain orientation. Measurements of FIB sputter rates on single crystal Cu specimens [3, and Table I] show sputter rate variation of 3.6 times between fast milling orientations such as (111) and slow milling orientations such as (110). This difference in sputter rate poses severe difficulties in uniformly cutting Cu interconnect, but at first glance produces only an annoyance in terms of a requirement of extra milling time to produce TEM specimens by FIB. However, the reasons for this differential sputter rate conceal more alarming issues that need to be taken into account by those producing TEM specimens, particularly as these effects do not seem to be limited only to Cu, but have been observed at Fibics to appear to occur in certain Au and Ni based systems.

It is tempting to attribute this sputter rate difference to ion channeling effects, nevertheless it appears that the slow sputtering of the (110) orientation is not solely due to differences in channeling. In fact this effect may result, to a significant extent, from the formation of an anomalous metal–gallium (M_xGa_y) phase during FIB milling under conditions in which the incident FIB beam hits the specimen at angles far from glancing and closer to normal incidence.

During FIB milling of Cu, “stubborn” grains frequently take on a “dark” appearance when viewed in FIB secondary electron mode; these “dark” grains grow and spread as the ion dose increases. In the case of the single crystal (110) Cu, these “dark” regions first appear at a low dose, and persist and grow throughout the milling operation (Figure 1).

TEM cross-sections of FIB “craters” have been prepared through these dark grains in (110) Cu. These specimens revealed a layer of Cu material rich in Ga, approximately 85 nm in thickness, at the floor of the sputter crater (Figure 2). The electron beam in the TEM was focused to a fine spot and convergent beam electron diffraction (CBED) patterns were obtained from both the substrate Cu and the Ga-rich layer at the crater floor. Analysis of the patterns identified the Ga-rich layer to be Cu_3Ga. (matching JCPDS powder diffraction file 44-1117). This suggests that not only was the (110) crystal slow to mill because of channeling, but also enough Ga was implanting into the sample to transform the bottom floor of the crater into Cu_3Ga, which significantly resisted sputtering.

When examining the edge of the sputter crater in the TEM, it was noticed that the Cu_3Ga phase was not evident until the point at which the crater floor was perpendicular to the incident ion beam (Figure 3). This suggests that the Cu_3Ga phase would only be produced when the incident ion beam was effectively parallel to the (110) surface normal. These are the same conditions where substantial channeling would be expected into pure Cu. These conditions are quite common during FIB circuit edit of Cu interconnect but are rare during conventional FIB-TEM specimen preparation ***unless*** the specimen’s protective surface layer (usually FIB-deposited W or Pt) is lost, or the specimen is tilted towards the beam for coarse machining which can occur when performing the “Lift-Out” preparation technique.

A patented and patent pending set of techniques known collectively by FEI as “CoppeRx” [2, 3, 4] can successfully prevent this anomalous phase formation, and care to avoid implantation conditions during TEM specimen preparation will also reduce this problem, but microscopists should be aware of the potential existence of this artifact. Due to the extremely high concentration of Ga present in this phase, the artifact is readily detected by both EDX and diffraction in the TEM. Before panic ensues, it should be noted that Fibics has produced over two hundred TEM specimens of Cu, Ni or Au, rarely observing anomalous phase formation, nor are there references in the FIB literature, so it is clear that standard FIB-TEM specimen preparation is not in jeopardy. This presentation will demonstrate techniques to identify and avoid anomalous gallium phase formation at the early stage of specimen preparation while observing the sample in the FIB system.

References
1. S. Herschbein, L.S. Fisher, T.L. Kane, M.P. Tenney, A.D. Shore, Proc. ISTFA, 127, (1998).
2. J.D. Casey, K.E. Noll, R. Shuman, C. Chandler, M. Megordan, M. Phaneuf and J. Li, LSI Testing Symposium 2000, Osaka, Japan (2000).
3. M.W. Phaneuf, J. Li, R.F. Shuman, K. Noll, J.D. Casey Jr., U.S. Patent Application 20010053605 (March 2000).
4. R.F. Shuman, K. Noll, J.D. Casey Jr., U.S. Patent 6,322,672 B1 (Issued November 2001).
5. The authors wish to thank Dr. Shigeo Saimoto of Queen's University at Kingston, Ontario, for providing the single crystal copper specimens (Dr. Saimoto is supported by Materials and Manufacturing Ontario) and also Dr. Graham Carpenter, Emeritus TEM Scientist, Materials Technology Laboratory (MTL), Natural Resources Canada and Dr. Gianluigi Botton formerly of MTL and now at McMaster University, for many useful discussions and TEM analyses over the years.

	Depth of FIB Crater (μm)		
Trial Number	**(1 1 1)**	**(1 1 0)**	**(1 0 0)**
1	2.46	0.66	1.60
2	2.46	0.69	1.44
3	2.52	0.66	1.60
4	2.41	0.72	1.60
5	2.41	0.66	1.60
Average Depth (μm):	**2.45**	**0.68**	**1.57**
Std. Deviation (μm):	**0.04**	**0.02**	**0.06**

TABLE I

Measured sputter crater depths into electropolished single crystal copper of known orientation for a dose of 5 nC / μm^2 per crater at constant beam current and pixel spacing.

Depth ratios (Proportional to Sputter Rates)

(1 1 1) / (1 1 0) = 3.62   (1 0 0) / (1 1 0) = 2.31

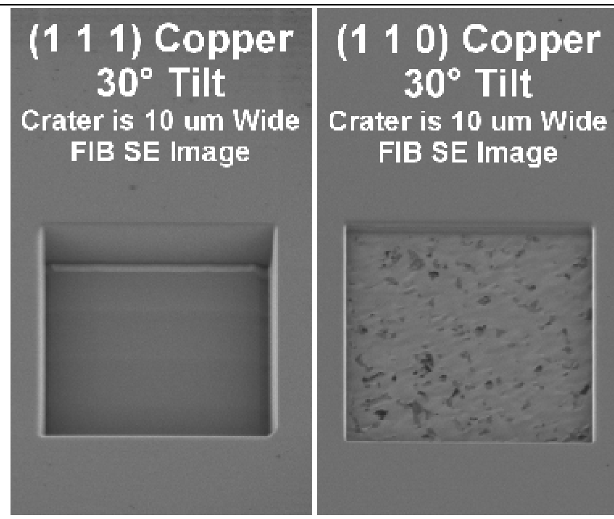

FIG 1. Comparison of Cu single crystals after equal milling. Note the mottled and dark grains in the (1 1 0) crater floor.

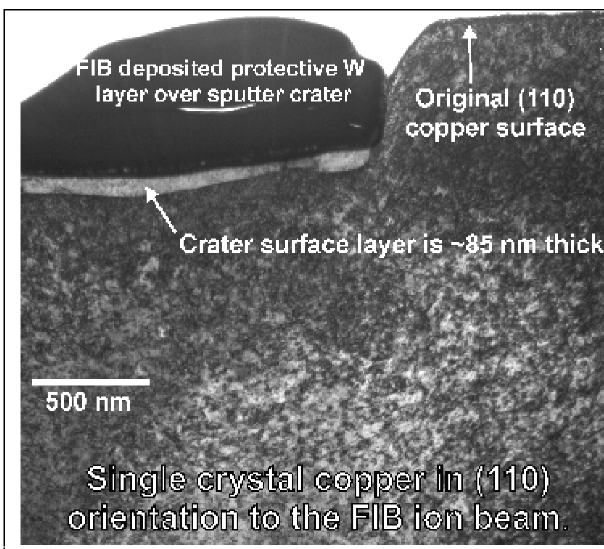

FIG 2. TEM BF image of a FIB-prepared cross-section from the edge of a (110) sputter crater prepared as in FIG 1. A thin layer of gold was sputter deposited on the surface of the crater prior to FIB deposition of tungsten. The ~85 nm thick "surface layer" proved (by CBED) to be Cu_3Ga.

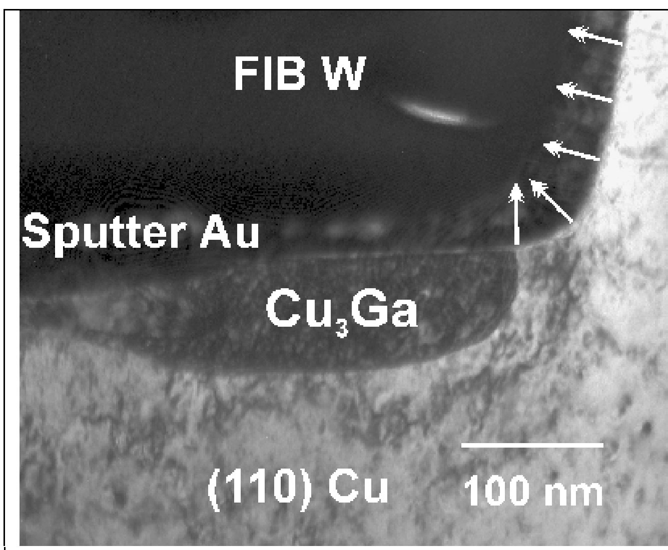

FIG 3. TEM BF image of the crater edge. The protective Au and W coatings preserved the original surface of the crater. Note that the Cu_3Ga phase only begins to form when the surface normal of the crater wall (white, double headed arrows) becomes essentially parallel to the incident FIB Ga beam during sputtering.

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