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Gallium Phase Formation in Cu During 30kV Ga^+ FIB Milling

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The appearance of dark areas during ion imaging of many face centered cubic metals is common and have been noted to appear quite quickly in fine-grained samples. Focused ion beam milling with Ga^+ of Cu and other face centered cubic metals produces modified surface microstructures that are described as either ion beam induced oriented grain growth or Ga-intermetallic growth. [1,2] The extensive use of FIB milling for sample preparation and the use of ion channeling contrast to characterize and measure grain sizes require that the modification of the surface by the ion beam be properly understood.

Sputter deposited films of Cu and Au 1-2 μm thick were imaged using secondary electrons generated by 30kV Ga^+ ions in an FEI DB235 dual beam system. Areas of 100 μm^2 were scanned using a variety of ion beam currents for 1 to 10 minutes to produce a range of ion doses. Electron backscatter diffraction (EBSD) was used to identify the Ga-intermetallics that formed and to characterize the orientation of the milled surface. TEM and STEM microanalysis and EBSD of the cross sections were carried out to determine the extent of Ga penetration and phase transformation.

EBSD inverse pole figure maps with respect to the surface normal are shown in Fig. 1 of milled areas for ion doses of $25x10^{16}$ and $38x10^{16}$ ions/cm^2. The EBSD patterns from the milled area of Fig1a are indexed as a cubic phase (probably an ordered Cu-Ga phase) and in Fig. 1b, at a higher ion dose of $38x10^{16}$ ions/cm^2, the region has fully transformed to a hexagonal phase (Cu_3Ga). The cubic phase and the hexagonal phase have a <110> or a <11-20> out of plane texture as indicated by the green color in the inverse pole figure maps and the inset legends. The FCC and HCP orientation relationship is: $<110>_{FCC} \| <11\text{-}20>_{HCP}$ and $\{111\}_{FCC} \| \{0001\}_{HCP}$. Figure 2 is an EBSD inverse pole figure map with respect to the milling direction obtained from a cross section of the milled area of Fig.1a. This map clearly shows that the surface modified region extends over 100 nm into the sample. This depth is much thicker than the 10 nm Ga ion range predicted by TRIM. However, TRIM does not account for channeling. Figure 3 shows composition profiles obtained in STEM of the two regions. Note that the surface is highly enriched in Ga and extends over 100 nm into the film.

The formation of the dark imaging regions as a result of ion beam milling can be explained in the following manner. During ion milling with 30 kV Ga^+ ions ($25x10^{16}$ ions/cm^2) , the surface of the sample becomes highly enriched in Ga and results in the precipitation of an oriented Cu-Ga cubic ordered phase. Further exposure to the ion beam ($38x10^{16}$ ions/cm^2) results in the formation of a thin surface layer of hexagonal Cu_3Ga (on top of the cubic phase) which is also oriented with respect to the milling direction. The orientation relationship observed in this study is commonly found in the precipitation of HCP from FCC phases. The dark regions are a consequence of the strong channeling orientations of the cubic <110> and hexagonal phases <11-20> parallel to the ion beam. These orientations result in strong ion channeling and a decrease in the number of emitted secondary electrons. Channeling also increases the ion range resulting in the relatively thick surface modified layer observed in this study.

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[3] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United Stated Department of Energy (DOE) under contract DE-AC0494AL85000.

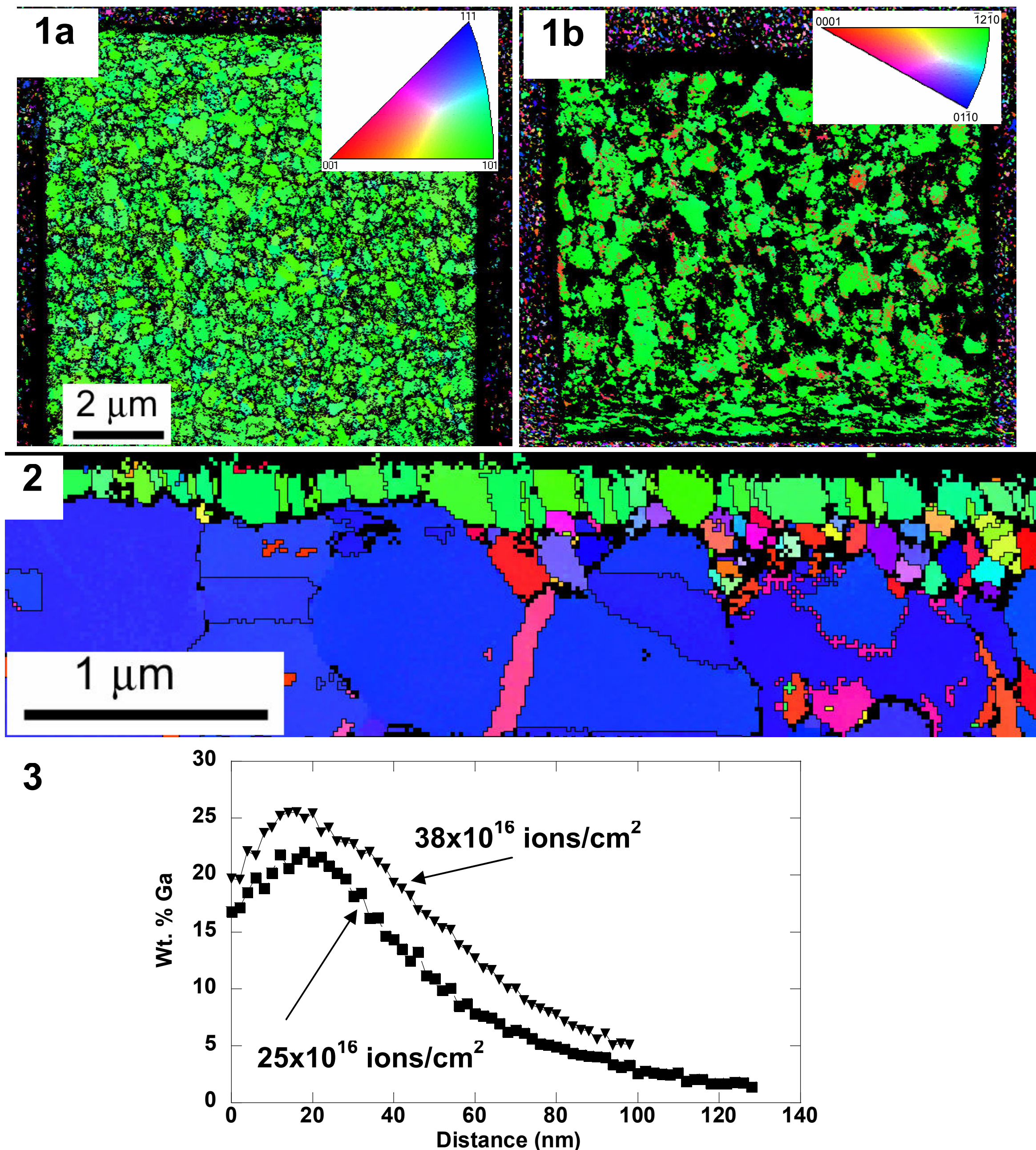

Figure 1. EBSD orientation maps with respect to the milling direction of CVD Cu after FIB milling a)$25x10^{16}$ ions/cm^2 resulting in a cubic phase at the surface, b) $38x10^{16}$ ions/cm^2 resulting in hexagonal Cu_3Ga. Figure 2. EBSD orientation map with respect to the growth direction of the sample shown in Fig1a. Figure 3. Ga concentration profiles from the milled areas shown in Fig 1a and 1b.

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