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Understanding Microstructural Changes in Metals Induced by Gallium Ion Beam Irradiation

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The irradiation of metallic surfaces with a focused beam of Ga ions is not a benign process. It is well known that energetic ions can cause sputtering of the target, but other microstructural changes have also been observed.[1] The appearance of dark imaging areas during ion imaging and milling of metals is common and has been observed in face centered cubic (FCC), body centered cubic (BCC) and hexagonal crystal structures. An example is shown in Figure 1 where a 30kV Ga ion beam has been rastered over electroplated Cu. The extensive use of FIB milling for sample preparation and the use of ion channeling contrast to characterize grain structures and measure grain sizes requires that the modification of the surface by the ion beam be properly understood and no longer ignored.[1]

Sputter deposited films of Cu, Au and W 1-2 μm thick were imaged using secondary electrons generated by 30kV Ga^+ ions in an FEI Helios dual beam FIB. Areas of 100 μm^2 were scanned using ion beam currents of 48 and 280 pA for 1 to 10 minutes to produce a range of ion doses (10^{16} to 10^{18} ions/cm^2) and dose rates. Electron backscatter diffraction (EBSD) was used to characterize the orientation and crystalline structure 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 the depth of the modified microstructure.

Dark imaging regions were observed in Cu, Au and W during Ga ion induced secondary electron imaging at 30 kV and a variety of beam currents. EBSD inverse pole figure maps revealed that the ion irradiation developed dark regions had undergone a change in grain orientation with respect to the sample normal (the ion beam direction). Note that the ion irradiation conditions were sufficient to cause sputtering of the metal samples. These areas were always reoriented so that the easiest channeling orientation (<110> for FCC, <111> for BCC and <11-20> for HCP) was aligned with the ion beam direction. An example inverse pole figure for Cu with respect to the surface normal is shown in Figure 2 that demonstrates the high degree of <101> preferred orientation present after irradiation. In order to verify that the oriented regions were responsible for the dark imaging regions of the sample, the area fraction of oriented grains in EBSD maps were compared with area percent of oriented regions under the same ion irradiation conditions. The results are shown in Figure 3 for Cu and W. Note that there is good agreement between the fraction of dark imaging grains and the fraction of reoriented grains in each material, clearly showing the connection between the two. It is also interesting to note that the orientation modification is more extensive in Cu as compared to W for the same total ion doses. EBSD has also shown that at increased doses (longer exposures) the intermetallic phase Cu_3Ga forms with <11-20> parallel to the beam direction.[1] No second phase formation was found in Au or W.

The mechanism driving formation of the dark reoriented areas during irradiation with Ga ions is not entirely clear. Differential damage models have been proposed to describe the microstructural alterations that are observed.[2,3] This model considers that grains oriented in the easy channeling direction will develop less damage than those in a non-channel orientation causing grain boundary migration into the grain with more damage. It is difficult to reconcile the details observed in this study entirely by the differential damage model. Further work is needed to obtain a more complete understanding of this phenomenon. Finally, these results indicate that caution is required when ion beam imaging and/or milling is applied to fine grained crystalline materials as the resulting microstructure may not be representative of the starting material.

References

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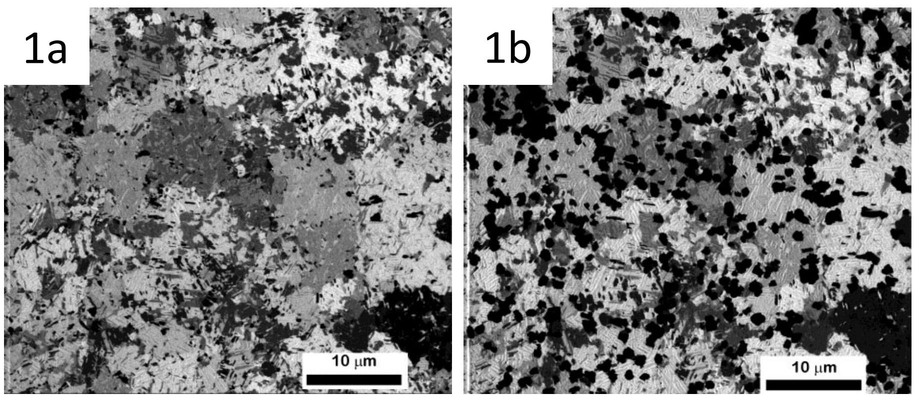
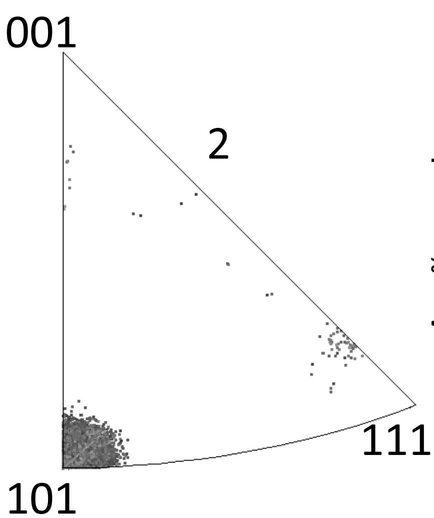
[4] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United Stated Department of Energy (DOE) under contract DE-AC0494AL85000.





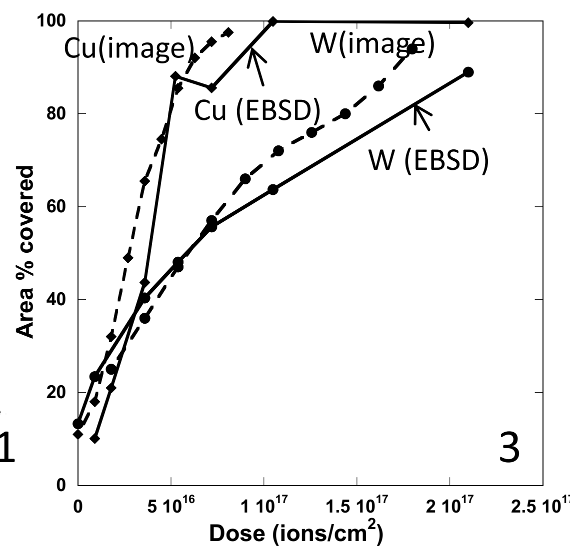


Figure 1. Ion induced SE images of Cu after irradiation a)lower dose, b) higher dose. Figure 2. Inverse pole figure with respect to the surface normal of Cu after irradiation. Figure 3. Plot of area percent covered by the reoriented (solid lines) or dark imaging regions(dotted lines) as a function of the ion dose for W and Cu.

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