Conventional TEM Investigation Of The FIB Damage In Copper

D. Kiener\textsuperscript{a, b}, T. Jörg\textsuperscript{a}, M. Rester\textsuperscript{a}, C. Motz\textsuperscript{a}, G. Dehm\textsuperscript{a, c}

\textsuperscript{a}Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria
\textsuperscript{b}Materials Center Leoben, Forschungs GmbH, Leoben, Austria
\textsuperscript{c}Department of Material Physics, Montanuniversität Leoben, Leoben, Austria

Several years ago, the focussed ion beam (FIB) technique has found a broad variety of applications in material science [1]. Recently, the FIB became popular as a tool for making TEM samples as well as machining miniaturized mechanical test samples [2-7] to investigate the influence of sample dimensions on mechanical properties. As the surface to volume ratio is large for submicron sized test structures, any surface modifications by ion bombardment and implantation may critically alter the mechanical properties.

To investigate the Ga\textsuperscript{+} ion damage by TEM, a 150 nm thick polycrystalline Cu film with an average grain size of \(\sim\)300 nm deposited on a \(\sim\)100 nm thick amorphous SiN\textsubscript{X} membrane was used. This Cu film was intentionally damaged with Ga\textsuperscript{+} ions using a dual-beam workstation consisting of a scanning electron microscope and an integrated scanning Ga\textsuperscript{+} FIB column. The kinetic energy of the primary Ga\textsuperscript{+} ions was varied between 5 keV and 30 keV.

The formation of an amorphous layer under grazing Ga\textsuperscript{+} ion impact was investigated using line patterns milled with the FIB. Bright-field TEM images of a line milled with 50 pA and an accelerating voltage of 30 kV reveal an amorphous layer with a thickness of \(\sim\)10 nm. EDX measurements show a significant Ga concentration in the damaged layer. Bright-field TEM images of a line milled with 50 pA and an accelerating voltage of 5 kV show a broader damaged zone due to the less well focussed ion beam at low acceleration voltages.

The damage structure due to perpendicular Ga\textsuperscript{+} ion impact was investigated using areas damaged by FIB milling in deposition mode. Fig. 1(a) shows a dark-field TEM image of a Cu grain situated at the border of an ion damaged and undamaged region. The right part of the grain was exposed to Ga\textsuperscript{+} ions with a kinetic energy of 30 keV and an ion current of 50 pA. A dislocation-like network with a spacing of \(\sim\)30 nm is observed. A reduction in kinetic energy from 30 keV to 5 keV did not lead to a significant reduction in damage formation (see Fig. 1(b), showing a bright-field TEM micrograph of the damage pattern of a grain bombarded by 5 keV Ga\textsuperscript{+} ions and an ion current of 100 pA).

The damage pattern changes in contrast with the diffraction vector and even vanishes for certain conditions, as can be seen when comparing Figs. 2(a) and (b), indicating different sets of dislocations. Further complications arise if the overlap between succeeding milling areas is insufficient, leading to an additional thickness contrast (see Fig. 3).

To investigate the ion damage under grazing ion impact, TEM samples were prepared from single-crystal Cu using the FIB and an in-situ lift out technique. No protection layer was applied to ensure comparability to previous investigations. Fig. 4(a) shows a TEM bright-field image of a sample thinned with a kinetic energy of 30 keV and a final milling current of 50 pA. A significant damage reduction was observed. Fig 4(b) shows a TEM bright-field micrograph of a region of the same sample milled with 5 keV grazing incident Ga\textsuperscript{+} ions and a final milling current of 100 pA. This reduction in ion energy along with the grazing ion impact led to the desired low amount of damage [8].

Fig. 1(a): Dark-field TEM image of a Cu grain partly exposed to Ga$^+$ ions with a kinetic energy of 30 keV and an ion current of 50 pA. (b) Bright-field TEM micrograph of the damage pattern of a grain bombarded by 5 keV Ga$^+$ ions and an ion current of 100 pA.

Figs. 2(a) and (b): The damage pattern changes in contrast with the diffraction vector, indicating different sets of dislocations.

Fig. 3: Thickness contrast due to insufficient overlap between succeeding milling areas.

Fig. 4(a): TEM bright-field image of a sample thinned with a kinetic energy of 30 keV and a final milling current of 50 pA. (b) TEM bright-field micrograph of a region of the same sample milled with 5 keV grazing incident Ga$^+$ ions and a final milling current of 100 pA.