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In situ Transmission Electron Microscopy of Rapidly Solidifying Metals and Alloys

A. Kulovits,* J.M.K. Wiezorek,* T. LaGrange,** Bryan W. Reed**, and G.H. Campbell**

* Department of Mechanical Engineering and Materials Science, University of Pittsburgh, 648 Benedum Hall, 3700 O Hara Street,
** Lawrence Livermore National Laboratory, Physical and Life Science Directorate, Condensed Matter and Materials Division, 7000 East Avenue, P.O. 808 L-356

We used the unprecedent nanometer spatial and 15ns temporal resolution of the dynamical transmission electron microscope DTEM at Lawrence Livermore National Laboratory to study morphological and structural changes in metallic thin films induced by rapid liquid-solid transformations (Figure 1a) [1]. The unprecedent spatiotemporal resolution of the DTEM allowed us to study phenomena associated with ultrafast solidification processes in pure Al, Cu, Ag and two Al-Cu alloys. The in situ experiments were performed by first melting the thin metal or alloy films with a single 12ns, 1064nm laser pulse (Figure 1b). After a pre-selected time delay a 15ns laser pulse (at FWHM) illuminated the previously molten area to acquire either a bright field image or a diffraction pattern (Figure 1b). The acquisition of a series of images and accompanying diffraction patterns (DP) with different time delays enables direct observation of morphological and structural changes during the entire solidification process.

Figure 1c shows bright field images of an Al thin film area after successful melting and solidification. Elongated grains formed during solidification stretch from the edge to the center of the melt pool. Figure 2 shows a series of images and diffraction patterns for different time delays for an Al thin film. Before the melting, the image of the as-deposited nanocrystalline thin film shows minimal contrast due to the low, ~4000x, magnification and ~ 20 nm grain size. The DP shows a ring pattern characteristic of the nanocrystalline Al film and the diffuse background from the amorphous Si_3N_4 substrate. After 0.5 μs time delay the contrast variations disappear and the sharp ring pattern in the DP disappeared, indicating that the Al is in the liquid state. After 5 μs a planar solidification front was in the field of view. Discrete diffraction spots associated with elongated grains, formed by rapid lateral solidification, close to a Bragg condition appear in the DP. After solidification the DP showed a reduced diffuse background and distinct diffraction spots instead of diffraction rings, due to a significantly reduced number of grains in the field of view. Figure 3 shows select images of delay time sequences for Ag, Cu, a dilute Al-5at.%Cu and a concentrated Al-33at.%Cu alloy. Observing solid liquid interface dynamics during the transformation using snap shot images enabled determination of the solidification front velocity, e.g. ~ 3-4 m/s in the case of the Al thin films. Post mortem TEM studies revealed differences in defect content and structure for Al, Cu and Ag, three FCC metals. . The morphology and structure of the advancing liquid solid interface of two Al-Cu alloys of two different compositions were investigated and compared to the structure of the liquid solid interface of pure Al. Figure 3 shows that the planar solidification front destabilizes as the Cu content increases. Future work will include detailed investigation of the dependence of the interfacial stability on chemical composition of the Al-Cu alloys, in order to provide experimental data for theoretical model verification.

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[1] T. LaGrange et al., *Appl. Phys. Let.* 89 (2006) 0044105

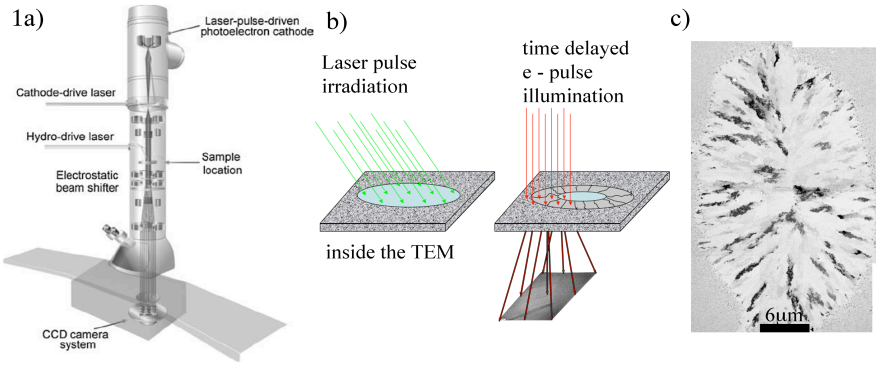

FIG. 1. a) Schematic of the modified TEM instrument, DTEM; b) Schematic of the experimental sequence; c) Bright field image montage of a molten and re-solidified area of Al thin film.

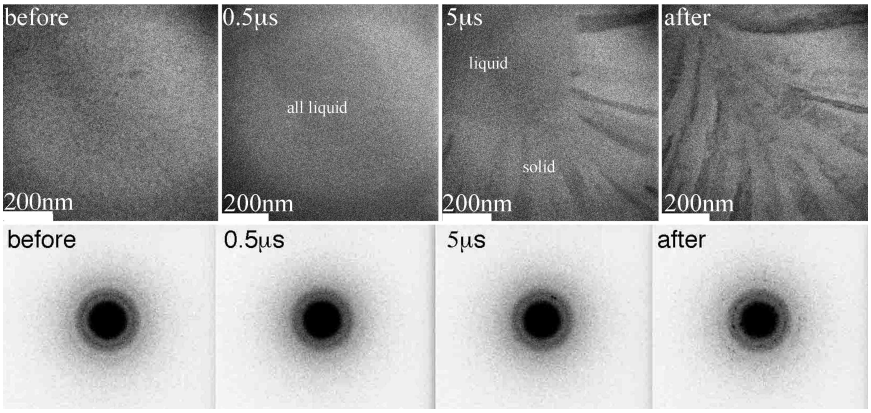

FIG. 2. Time-delay sequence of multi-beam bright field TEM images and diffraction patterns showing morphological and structural evolution during rapid solidification of the Al thin film.

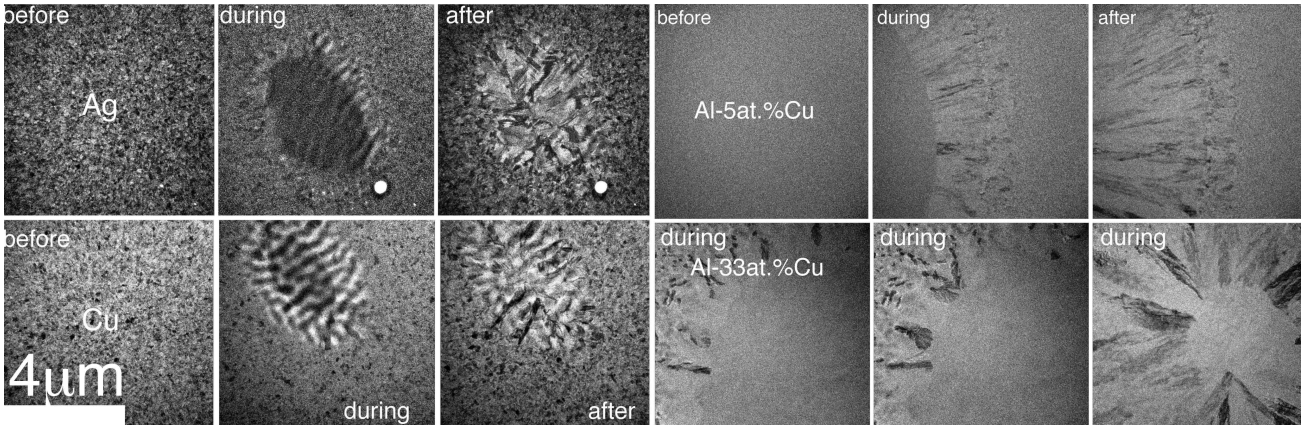

FIG 3. Time-delay sequence of multi-beam bright field TEM images showing morphological and structural evolution during rapid solidification of the pure Ag, Cu and two Al-Cu thin films.

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