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Composition and Crystal Orientation Mapping of nano-scale multi-phase Rapid Solidification Microstructures in hypo-eutectic Al-Cu Alloy Thin Films

Jörg M.K. Wiezorek[1], Can Liu[1], Sahar Farjami[1], Kai W. Zweiacker[1,3], Joseph T. Mckeown[2] and Geoffrey H. Campbell[2]

[1.] Department of Mechanical Engineering and Materials Science, Swanson School of Engineering, 636 Benedum Hall, 3600 O'Hara Street, University of Pittsburgh, Pittsburgh, PA 15261, USA.
[2.] Materials Science Division, Physical and Life Science Directorate, Lawrence Livermore National Laboratory, 7000 East Avenue, Livermore, CA, 94551, USA
[3.] Now at EMPA, Swiss Federal Laboratories for Materials Science and Technology Überlandstrasse 129, CH-8600 Dübendorf, Switzerland.

Recent nano-scale spatio-temporal resolution *in situ* transmission electron microscopy (TEM) studies of the rapid solidification (RS) of elemental Al and hypo-eutectic Al-Cu alloys after pulsed laser melting permitted measurements of the evolution of crystal growth rates and correlation with crystal growth mode changes responsible for the microstructure formation under non-equilibrium conditions [1-4]. The morphological characteristics of the multi-phase RS microstructures in the electron transparent thin film Al-Cu alloys were identical to those reported for equivalent composition bulk alloys [2-6]. Here we combine precession electron diffraction assisted automated crystal orientation mapping (PED-ACOM) with scanning (TEM) (STEM) imaging and energy dispersive X-ray spectroscopy composition mapping (EDXSM) to elaborate constitutional and crystallographic aspects of the development of multi-phase RS microstructures in hypo-eutectic Al-Cu alloys with 4at% to 12at% Cu.

Fig. 1a and 1b illustrate the refined scale and distinct morphology of the RS microstructures of hypoeutectic Al-Cu with the heat affected zone (HAZ, Zone 1), a narrow transition region with elongated α-Al grains (Zone 2), the RS crystal growth region with columnar morphology cellular two-phase growth (Zone 3a), and the banded-morphology growth region (Zone 3b) [2, 6]. The formation of these different microstructural zones is related to the changes in RS interface velocity after pulsed laser induced melting (e.g. Fig. 1c). After incubation periods that last from ~5μs to ~25μs (increasing with Cu%) the directional RS crystal growth forms the Zone 2, Zone 3a and Zone 3b multiphase microstructures at crystal growth rates of 0.1m/s to ~2.0m/s (e.g. Fig. 1c for Al-4Cu). RS front velocity changes profoundly affect the coupled growth of the α-Al (dark, Fig. 1b) and Al_2Cu (bright, Fig. 1b). Fig. 2 shows STEM DF-imaging and EDXSM data obtained for Zones 1 and 2 in Al-11Cu. For this alloy the α-Al grains in Zone 1 contained 3.6at%Cu (e.g. circles, Fig. 2a), which is about the same as for the e-beam deposited films prior to RS [6]. The Al_2Cu phase in the triple junction of the continuous Cu-enriched network in Zone 1 and Zone 2 (TJ in Fig. 2a) contained on average 31.5at%Cu. Notably, the cellular growth zone (Zone 3a) exhibited discontinuous fine-scale copper enriched metastable Al_2Cu (θ') phase within the supersaturated α-Al matrix grains, while the Cu-enriched continuous network in Zone 1 and Zone 2 contained θ-Al_2Cu phase (Figs. 1 and 2) [2-4]. Under equilibrium conditions hypoeutectic Al-Cu alloys contain the face-centered cubic α-Al matrix (≤2.6at%Cu) and tetragonal θ-Al_2Cu (~33at%Cu) phase as primary pro-eutectic and secondary lamellar eutectic microstructural constituents. The PEDACOM and EDXSM analyses of the hypo-eutectic Al-Cu alloys showed deviations of the phase fractions and the compositions from those of the as-deposited stated and those predicted by the equilibrium phase diagram for α-Al and Al_2Cu for the RS microstructure Zones 2, 3a and 3b. EDXSM consistently showed severe composition gradients for α-Al in the narrow Zone 2 transition region

(≤ 1µm) from slow solidification rates below 0.1m/s to RS velocities ≥ 0.5m/s, while the supersaturated α-Al phase compositions remained constant in Zone 3a and reached alloy composition for the single-phase crystallization at transition to Zone 3b. The discontinuous morphology of the Al_2Cu (θ') phase in the Zone 3a implies continuous growth of the matrix Al crystal and repeated nucleation and growth for the minority Al_2Cu crystals during RS at interface velocities in excess of 0.5m/s [3, 4]. Finally, two different crystallographic orientation relationships between the α-Al matrix and the Al_2Cu (θ') phase that facilitate coherent interphase interface formation have been identified by PEDACOM.

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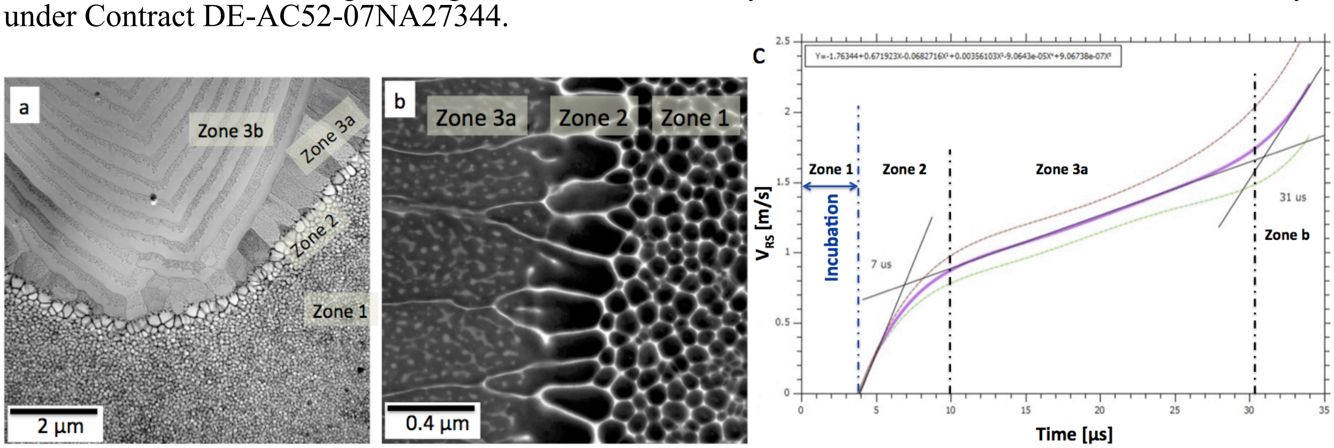

Figure 1. The RS microstructure zones, a) BF-, b) HAADF STEM images for Al-9Cu and c) RS interface velocity evolution for hypo-eutectic Al-4Cu after PL melting for Al-4Cu.

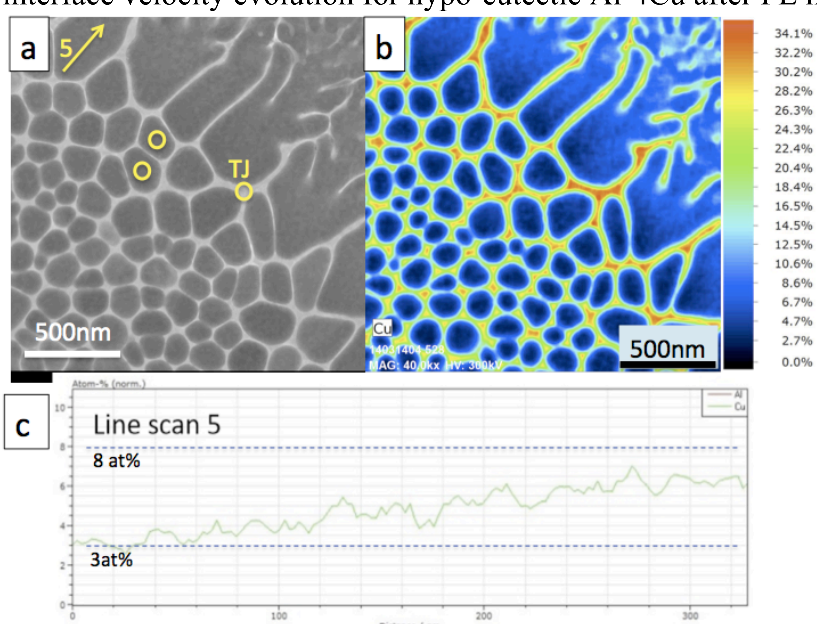

Figure 2. a) HAADF STEM image for Al-11Cu alloy with locations of example line scan for Zone 2 and compositions of α-Al grains and triple junctions (TJ) in the continuous network of Cu-enriched phase in Zone 1, b) Cu% heat map for Cu%≤35at%, c) example of 350nm length Cu% line scan for elongated α-Al grain in Zone 2 (marked in a)) with Cu% increasing from 3at% to 6at%.

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