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Automated Orientation Imaging Microscopy of Nanoscale Al Thin Films After Rapid Solidification using SEM and TEM

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We investigated the microstructure evolution in nanocrystalline Al thin films prior to and after rapid solidification using orientation imaging microscopy (OIM) with a field emission gun (FEG) scanning electron microscope (SEM) and a LaB_6-gun equipped transmission electron microscope (TEM), operated at 25kV and 200kV respectively. We sputter-deposited 80 nm thick Al films on a 100nm thick amorphous Si_3N_4 membrane of windowed TEM grids. The Al exhibited an average grain size of ~ 20 nm prior to pulsed laser induced local melting. Rapid solidification of the melted region was completed within ~10 µs [1], yielding grains, 80 nm thick, 0.5 µm to 1 µm wide and 4µm to 35 µm long (Fig. 1b). The resulting polycrystalline thin film microstructure thus comprised the nanocrystalline parent constituent and the microcrystalline product of the rapid solidification.

We used SEM OIM to determine whether a growth texture existed in the solidification product. Figure 1a shows a typical area analyzed by SEM OIM using a step size of 200 nm. Figures 2a and 2b display the corresponding confidence index and orientation maps, where about 60 elongated grains are discernible. Forming statistically significant datasets by combining data from several such fields of view facilitates determination of the growth texture (*for brevity not shown here*). Figure 2 also demonstrates that the lateral resolution limit of SEM OIM, typically estimated at ~ 30 nm to 60 nm [2-4], prevented collecting robust OIM data sets for the nanocrystalline parent microstructure, which provides the grain seeds at the melt-pool edge for the rapid growth of the solid. Additionally, significant fractions of the area analyzed by SEM OIM are affected by sampling EBSD data from multiple grains due to the presence of inclined grain boundaries between the 20nm diameter grains. Hence, we cannot study the occlusion process occurring during rapid solidification by SEM OIM. The lateral resolution also limits the accuracy of information on the position and character of grain boundaries in the microcrystalline grains of the rapid solidification product (Figs. 2b and d).

We used an automated OIM system in the TEM [5] to overcome the limitations of and to complement the results obtained by SEM OIM. The OIM in the TEM we used here relies on indexing of precession diffraction patterns to determine crystallographic orientations [5]. The main advantage of TEM OIM is the lateral spatial resolution, here ~ 15 nm, which enables robust OIM analyses of nanocrystalline grains. Aside from sample preparation, the main disadvantage of TEM OIM relates to the limited field of view and the associated difficulty to acquire statistically significant data sets necessary for quantitative texture analysis if grain sizes exceed about 100 nm.

Figure 1b shows a TEM image of a laser melted and re-solidified area that has been analyzed by TEM OIM. We used step sizes of 40 nm and 20 nm to scan the 7.5 µm by 7.5 µm fields of view. OIM in the TEM and SEM yielded similar confidence indices, ~0.15, while the TEM OIM confidence index map (Fig. 2e) is more detailed due to the superior lateral resolution. Nanocrystalline grains are clearly detectable in the orientation maps (Figs. 2f and g) enabling detailed analysis of the occlusion process during rapid solidification. The improved lateral spatial resolution of the TEM OIM also permitted more accurate detection of grain boundary positions and character than the SEM OIM (compare Figs. 2b, 2d with Figs. 2f, 2g and 2h).

Combining automated OIM in the SEM and TEM enabled us to address the challenge posed by the highly bi-modal grain size distribution, namely ~20 nm in the nanocrystalline parent constituent and >1µm in the rapidly solidified microcrystalline grains, of the laser processed Al thin films. We successfully acquired statistically significant datasets for growth texture determination by SEM OIM and measured orientation changes with lateral spatial resolution of 20 nm by TEM OIM to analyze the occlusion processes during the early stages of rapid solidification after laser melting Al thin film. We acknowledge access to facilities of the Materials Micro-Characterization Laboratory of the Department of Mechanical Engineering and Materials Science, University of Pittsburgh.

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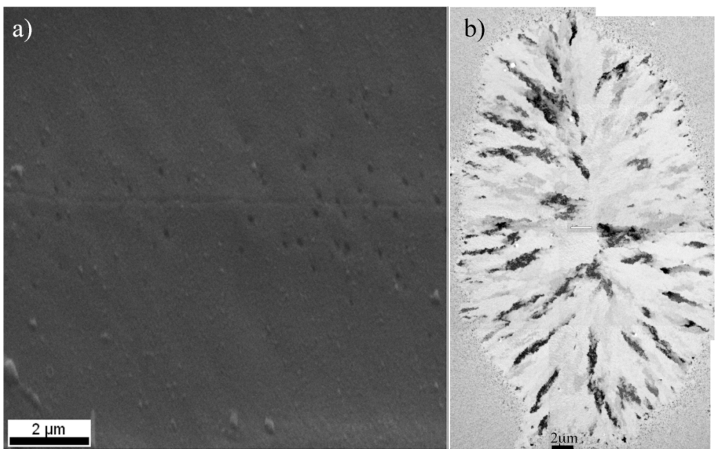

FIG. 1. a) Secondary electron SEM image of a re-solidified area with elongated grains; b) montage of TEM bright field images of a of elliptical shape area rapidly re-solidified after pulsed laser melting.

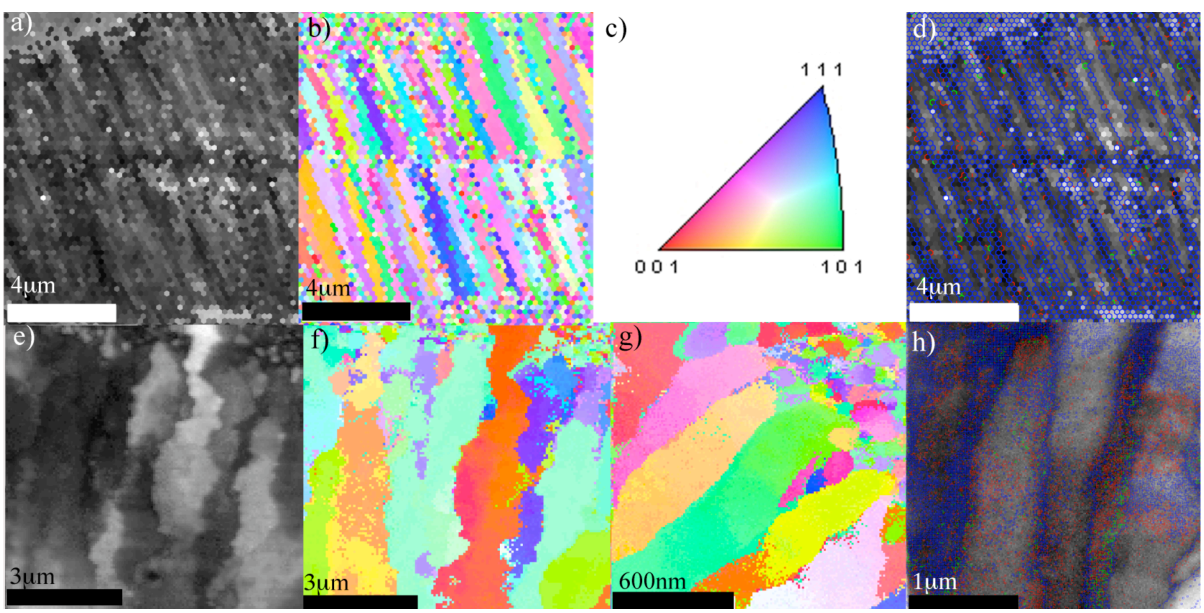

FIG. 2. a) to d) SEM & e) to h) TEM OIM maps - a) & e) Confidence Index (dark = lowest confidence index; white = highest confidence index); b), f) & g) orientation with legend in c); d) & h) Grain boundary misorientation angle, θ (θ >15° in blue, 15 ° ≥ θ > 5° in red, 5°≥ θ in green)

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