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The Influence of Film Thickness on the Microstructure of Nanocrystalline Nickel Films: A Precession Electron Diffraction Microscopy Study

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Recent studies have indicated that pulsed laser deposition (PLD) nanocrystalline nickel films exhibit special properties relative to PLD nanocrystalline copper or gold films [1]. In particular, *in-situ* TEM heating experiments have demonstrated the presence of large *hcp* phase nickel grains (> 100 nm) within a *fcc* phase nanocrystalline nickel matrix [2, 3]. However, it is currently unknown whether nanoscale *hcp* nickel grains are present in as-deposited films due to the limited spatial resolution of conventional characterization techniques for phase mapping. To address this issue, a Diffraction-Scanning Transmission Electron Microscopy (D-STEM) type lens configuration [4] combined with precession electron microscopy [5], capable of spatial resolution < 10 nm, has been used to determine the distribution of *fcc* and *hcp* nickel phases, as well as the grain size distribution of nanocrystalline nickel films of different thicknesses (50 and 100 nm).

Bright-field TEM images from the 50 and 100 nm-thick PLD nickel films are shown in Figs. 1a and 1b, respectively. Although several nanoscale grains are visible in these images, dynamical electron scattering, coupled with the fact that some grains are oriented off-zone axis, severely limits the overall microstructure analysis. In contrast, the reconstructed image containing phase and reliablity information obtained by D-STEM and precession electron microscopy clearly shows, for the first time, the true morphology of the nanocrystalline PLD nickel film (Figs. 1c and 1d). Both *hcp* and *fcc* phase grains with distinct grain boundaries are visible in the 50 nm film, while in the case of the 100 nm-thick film distinct *hcp* nickel grains appear to be located at the *fcc* grain boundaries. A careful analysis of approximately 831 and 956 *hcp* phase grains in the 50 and the 100 nm films show that the *hcp* phase grains are on average 10 and 11 nm in size, respectively. In addition, the 50 nm-thick film consists of 9% *hcp* phase, whereas the 100 nm-thick film contains 6% *hcp* phase. For comparison, the average grain size of the *fcc* matrix phase was determined by measuring 178 grains from the corresponding reliability maps (Figs. 2a and 2b). The *fcc* grains in the 50 nm film exhibit a narrower Gaussian distribution centered at approximately 23 nm (Fig. 2c), while those in the 100 nm-thick film show a broader distribution centered at around 40 nm (Fig. 2d).

In summary, we demonstrate for the first time the morphology of nanoscale *hcp* phase nickel that is present in as-deposited nanocrystalline PLD nickel films of different thicknesses.

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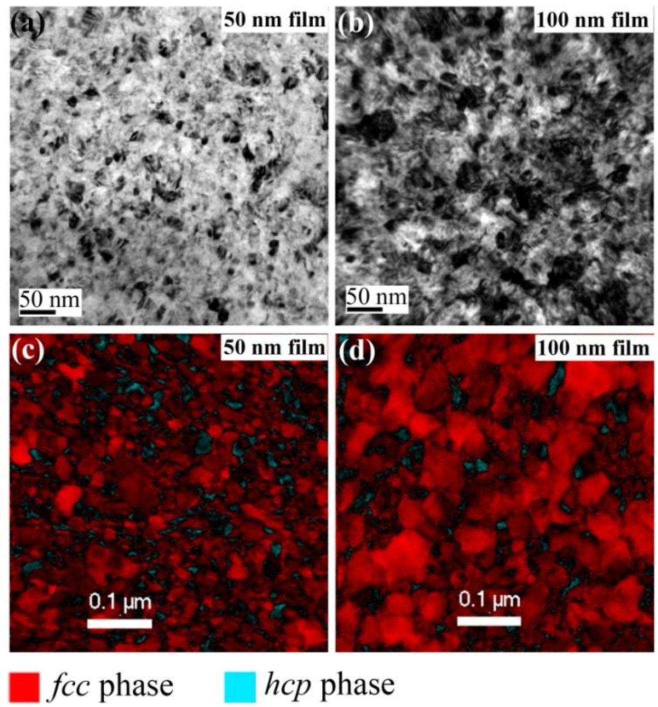

FIG. 1. (a) and (b) Bright-field TEM image of the 50 and 100 nm films, (c) and (d) combined phase and reliability maps from the 50 nm film showing both *hcp* and *fcc* phase grains, and from the 100 nm film showing *hcp* phase grains primarily at the *fcc* phase grain boundaries.

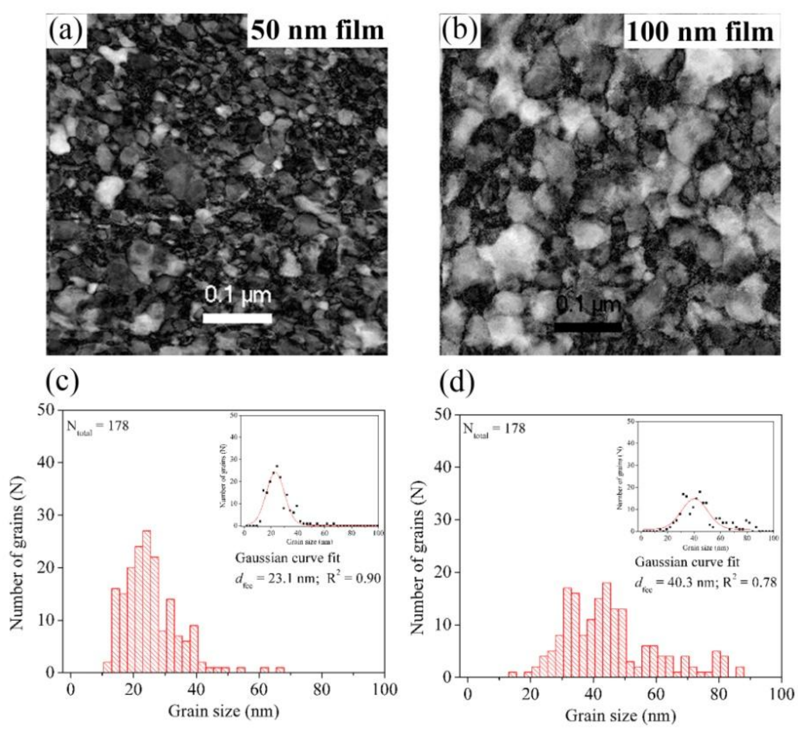

FIG. 2.
(a) and (b) Reliability maps corresponding to FIG. 1 (c) and (d) for the films 50 and 100 nm thick, which are used to determine *fcc* grain size in the two films, (c) and (d) *fcc* grain size distribution in the 50 and 100 nm films.

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