Understanding abnormal grain growth in nanograined nickel through the combination of *in situ* TEM and precession microscopy

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Nanograined materials have generated great interest due to the unique microstructures that have been observed and the often unexpected resulting mechanical properties. For many metal systems, it has been shown that a decrease in grain size results in an increase in hardness, as well as a decrease in ductility. To recuperate the loss in ductility, several processing concepts have been proposed, including the development of a bimodal grain size distribution [1].

To study the thermal stability of nanograined metals and the underlying mechanisms controlling the abnormal grain growth necessary to create a bimodal grain size distribution, this research has used *in situ* transmission electron microscopy (TEM) heating experiments of pulsed laser deposited (PLD) nanograined nickel thin films. Advancements in TEM heating stage technology (Figure 1A-C) has provided greater sample stability at elevated temperatures permitting the application of advanced TEM techniques during heating experiments. Through these studies, the microstructural evolution of nanograined nickel via abnormal grain growth was observed in real time. The resulted grain size analysis provides significant information about single grain boundary migration kinetics, as well as statistical information regarding the grain growth dynamics. Two unexpected microstructures, metastable hexagonal closed packed (HCP) nickel grains and large, high defect density face centered cubic (FCC) grains, were observed at temperature during these *in situ* TEM annealing studies [2-3]. These unique structures raised many questions regarding the influence of grain boundaries, local orientations, and overall film texture on the formation and evolution of nanograined films.

Until recent advancements in electron microscopy technique, the investigation of the role of texture and grain boundaries on the origin and evolution of these structures was not possible due to lack of an automated technique with the needed spatial resolution. However, recent advancements in precession microscopy that allow for less than 5 nm resolution texture maps with automated acquisition and identification of diffraction patterns have enabled detailed investigation of local orientation measurements inside the TEM [4-5]. Figure 1D shows a ray diagram for the precession microscopy technique that produces a nearly kinematical diffraction pattern from the sample. Using this technique, it was determined that the HCP nickel phase was indeed present in the as-deposited films at percentage levels indistinguishable to either selected area electron diffraction or X-ray diffraction. However, there remained questions about the evolution of the metastable HCP phase grains and what local microstructural variables were at play in promoting or hindering growth of those grains. In order to determine the texture evolution as a function of annealing condition, *in situ* TEM heating experiments were combined with precession electron microscopy. This combination of techniques has provided enhanced insight into the microstructural evolution of the nanograined PLD nickel that was not possible prior to the advancements in the stability of *in situ* heating stages and the development and commercialization of the precession system. In Figure 2, a nanograined nickel film was analysed with precession diffraction at room temperature, and then again after 120 s and 300 s of in-situ observation at 700 ºC. The same region was observed at each point, as indicated by the arrowed fiducial markers in each micrograph. The inverse pole figures accompanying each TEM image show out of plane texture evolution in both the FCC and HCP grains present. This presentation will discuss the observed influence of texture and grain boundaries on grain growth in nanograined nickel, and will also highlight the application of precession diffraction in combination with other *in situ* TEM techniques not previously studied [6].

References

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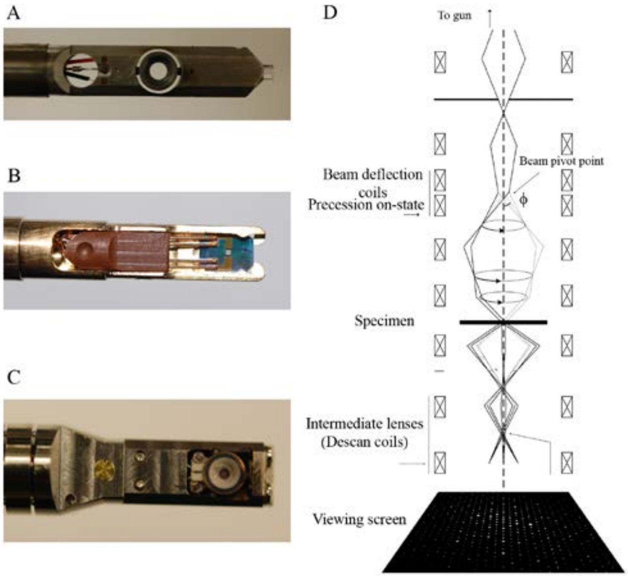

Figure 1. *A) Ray diagram that depicts precession of the electron beam resulting in a nearly kinematical diffraction pattern on the viewing screen, B) Gatan heating stage tip, C) Protochips Aduro heating stage tip, and D) Hummingbird heating stage tip.*

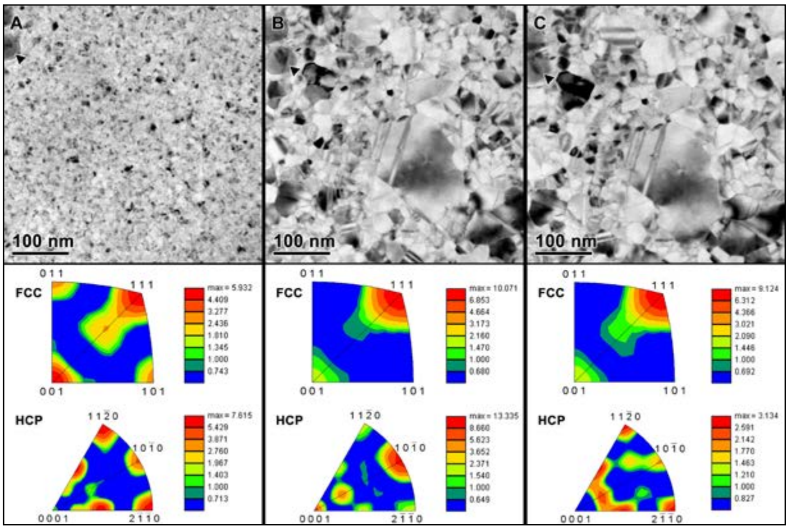

Figure 2. *A set of bright field micrographs and associated inverse pole figures taken during an annealing experiment performed at 700 ºC A) Initial set at room temperature, B) After 120s at 700 ºC, C) After 300s at 700 ºC.*