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Texture and Phase Analysis in Nanocrystalline Ni Thin Films by Precession Electron Diffraction Microscopy

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Grain refinement has been considered a powerful method to obtain high strength in metals and alloys. However, in materials with nano grain sizes, rapid and abnormal grain growth may occur when they are subjected to an annealing treatment. In addition, as grain growth occurs, the local texture may evolve, which will also affect the mechanical properties. Therefore, to achieve nanocrystalline metal thin films with high strength, it is critical to correlate the film thickness with grain size and local texture as a function of annealing temperature.

In this work, nanocrystalline Ni thin films with thicknesses of 30 nm and 120 nm were subjected to an annealing treatment at 350 C for 20-80 minutes. To identify the average grain size, grain orientation and phase fraction, precession electron diffraction was used on all of the aforementioned films. For this work, a 200kV JEOL 2010F TEM was used with a 5nm near-parallel beam (convergence angle < 1 mrad). The probe was scanned across the sample (510×510 nm^2 in this study) with a step size of 3nm and processed at an angle of 0.4^o at each step using the DigiSTAR^{TM} system from NanoMEGAS. This process is fully automated.

However, the reliability of the precession analysis is sometimes low due to the contribution of various grains to the diffraction pattern (DP), particularly when there are many grains below 5 nm. Thus, to improve the overall analysis, a novel method was used where the DP is filtered for the following parameters: noise threshold, spot enhance loop, gamma, spot radius and softening loop [1]. In this fashion, the reliability was improved by 5-10%. Finally, the orientation images acquired after indexing were then exported to the TexSEM Laboratories Orientation Imaging (TSL OIM^{TM}) software for further filtering, in particular a reliability threshold of 15% to make sure the results are consistent.

The analysis shows the presence of fcc and hcp phases (Fig. 1) in all samples [2]. In terms of grain orientation, the as-deposited 30nm films show a strong [110] // Normal Direction (ND) texture, while the annealing treatment promotes the formation of [100] // ND in the films. As for the 120nm thickness, a [111]//ND texture in observed in the as-deposited films and a near [112]//ND texture is observed when the films are subjected to various annealing times (Fig. 2). Our theoretical calculations show that the strain energy between the film and the substrate plays an important role in the observed texture for both the 30nm and 120nm films.

References:

[1]A. Kobler *et al*. Ultramicroscopy, **128** (2013), p.68.
[2] S. Rajasekhara *et al*. Scripta Materialia, **67** (2012), p.189.
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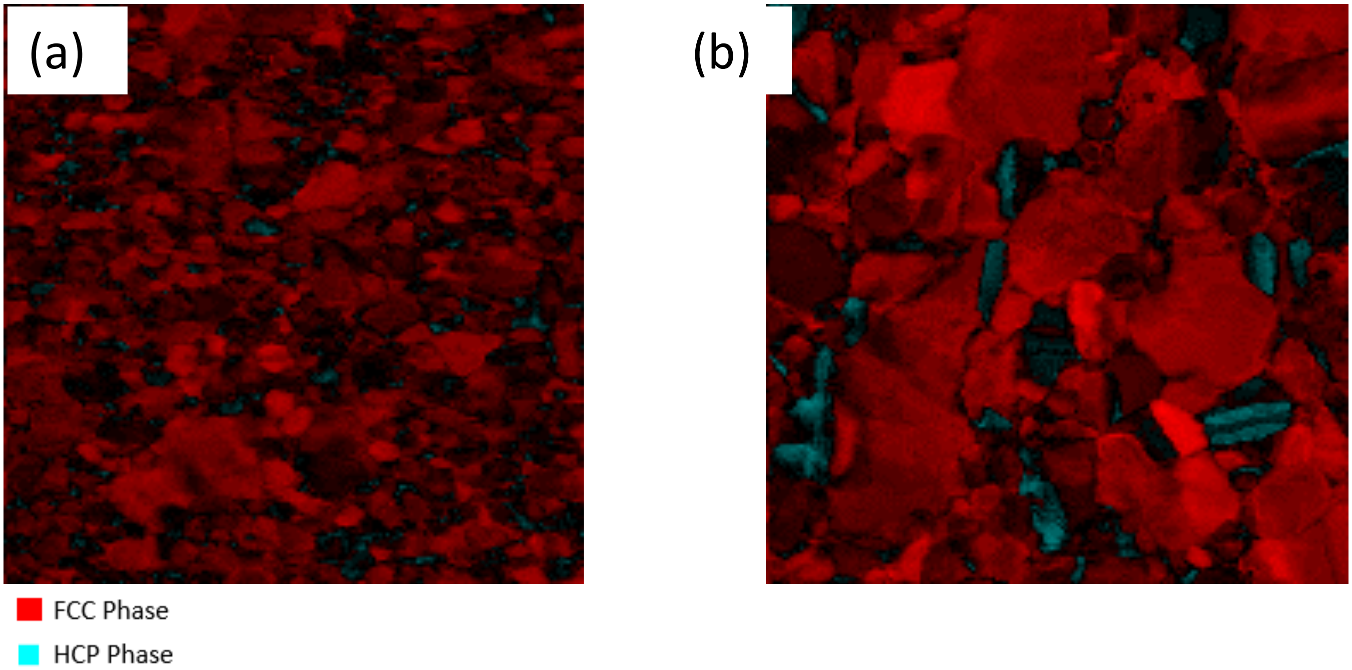

Fig. 1 Phase maps combined with reliability maps for samples of thickness (a) 30nm and (b) 120nm. The presence of fcc and hcp phases in both samples is evident.

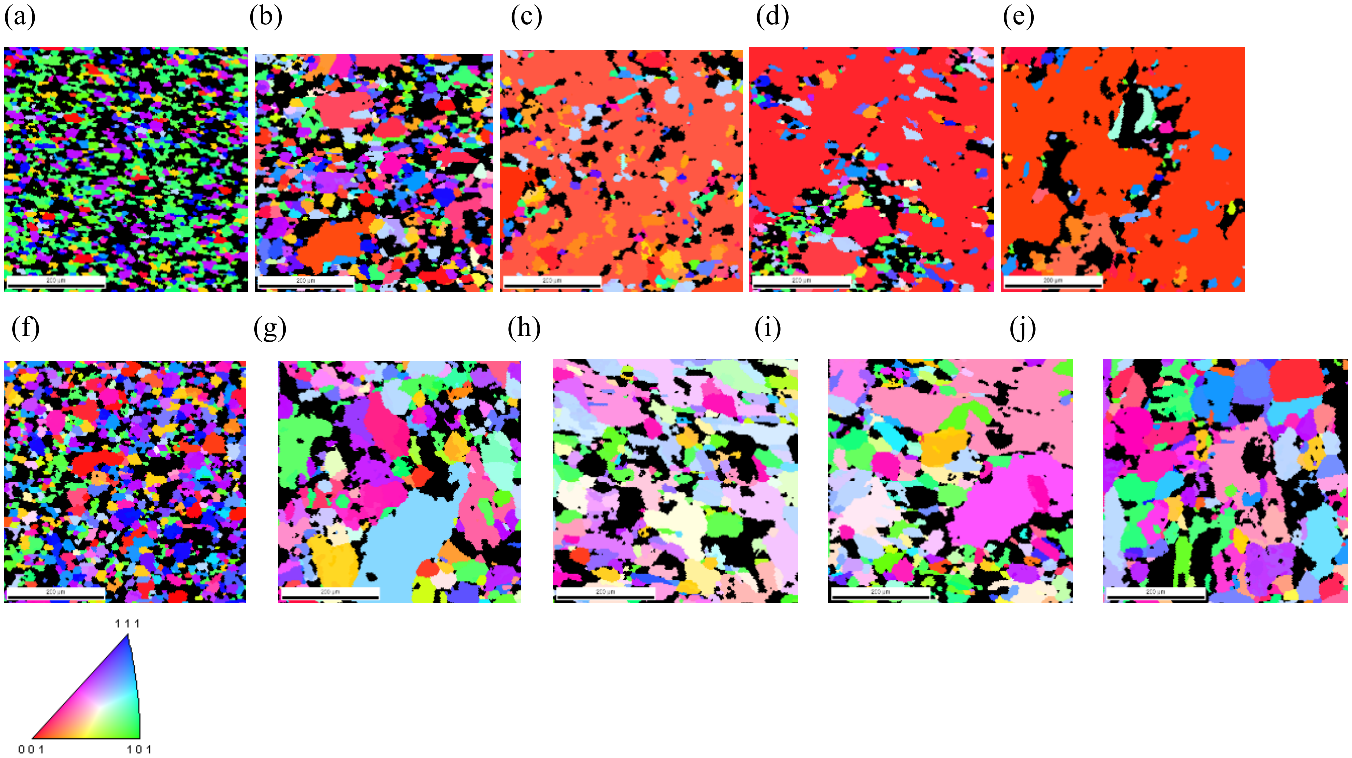

Fig. 2 Orientation maps combined with reliability maps. (a) as-deposited 30 nm films and annealed for (b) 20min, (c) 40 min, (d) 60 min and (e) 80min. (f) as-deposited 120nm film and annealed for (g) 20min (h) 40 min, (i) 60min and (j) 80 min.