Characterization of As-Received and In-Situ and Ex-Situ Annealed Electrodeposited Nanocrystalline Nickel Using Automated Crystal Orientation Mapping in TEM and SEM

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The microstructure and texture characteristics of the as-received and both in-situ and ex-situ annealed electrodeposited nanocrystalline nickel were investigated using the recently introduced ASTAR automated crystal orientation mapping in a transmission electron microscope (TEM), complemented by electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM). The former technique, permitting to achieve a spatial resolution of several nanometers, has been employed for a detailed study of the nanocrystalline specimen state. The TEM data acquisition was performed using a NanoMEGAS Digistar device, attached to a JEOL JEM 2100F transmission electron microscope operated at 200 kV. The above device performs scanning and simultaneous precession of the focused primary electron beam. The obtained nanobeam spot (disc) diffraction patterns are recorded by an external ultrafast optical camera. The pattern indexing (orientation determination) is performed off-line by dedicated software, using cross-correlation with a pre-calculated set of templates, and the resulting crystallite orientation maps are then constructed. The EBSD study was carried out on annealed samples using a Zeiss LEO 1530 field-emission gun SEM, operated at 20 kV, equipped with an HKL Technology/Oxford Instruments EBSD attachment. The HKL Channel 5 software was used to perform EBSD data acquisition and post-processing.

The as-deposited material consisted of nanograins interspersed with coarser (sub)grain clusters, arranged in large mesoscale colonies and characterized by a dominant <001> fibre texture aligned with the deposition direction (DD). A large fraction of nanograin/cluster boundaries displayed a low-coincidence site lattice or twin character, which indicates a tendency for these boundaries to adopt a low-energy state during the electrodeposition process. The EBSD study confirmed the previously suggested presence of the "cobblestone" type mesotexture, characterized by a local <001> fibre axis approximately perpendicular to the hemispherical growth surface of a mesoscale colony, in the above material. The (sub)grain clusters contained low-angle boundaries and displayed large misorientation gradients, nevertheless, their orientations did not statistically differ from the surrounding nanograins. Contrary to some previous suggestions, these clusters neither seemed to serve as nuclei for the observed abnormal grain growth during annealing nor displayed a strong tendency for widespread (sub)grain coalescence. The <001> // DD to <111> // DD fibre texture transition occurring during annealing did not seem to result from the growth of pre-existing suitably oriented nuclei. Instead, copious twinning occurring along the migration front of the abnormally growing grains appears to be primarily responsible for the above transition.

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