

## **ABSTRACT**

The low temperature annealing behavior of nanocrystalline Ni-Fe alloys produced by electrodeposition has been systematically studied for five Ni-rich compositions viz. Ni, Ni-5.5 wt% Fe, Ni-18.5 wt% Fe, Ni-28.5 wt% Fe and Ni-43 wt% Fe. The Design of Experiments consisted of specimen of these five compositions in the as-deposited condition and those subjected to annealing at 573 K, 648 K and 723 K in air for 16 hours. Surface sensitive techniques such as X-ray Photoelectron Spectroscopy (XPS) and Grazing Incidence X-ray Diffraction (GIXRD) were employed to study the nature of surface oxide species while Pole Figures (PF), Inverse Pole Figures (IPF) and 3 Dimensional Orientation Distribution Function (3D-ODF) were generated to investigate the change in preferred orientation or texture in the as-deposited and annealed conditions. Various Ni and Fe oxides formed at different annealing temperatures as identified by GIXRD and XPS. A distorted NiO with twice the lattice parameter of cubic NiO has been identified and the presence of NiFe<sub>2</sub>O<sub>4</sub> using XPS in Ni-18.5 wt% Fe, Ni-28.5 wt% Fe and Ni-43 wt% Fe under certain annealing conditions is shown. In case of Ni-5.5 wt% Fe, FeO was present preferably in NiO-FeO solid solution, At higher annealing temperatures FeO evolved into Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>. With increase in Fe content of the alloys and annealing temperature, there is preferential formation of Fe oxides over Ni oxides. This occurs at lower annealing temperatures for Ni-43 wt% Fe. As-deposited nanocrystalline Ni and Ni-5.5 wt% Fe showed a strong  $\langle 100 \rangle$  preferred orientation, while nanocrystalline Ni-18.5 wt% Fe, Ni-28.5 wt% Fe and Ni-43 wt% Fe showed a mixed  $\langle 111 \rangle \langle 100 \rangle$  preferred orientation. In the case of Ni-18.5 wt% Fe and Ni-43 wt% Fe this change occurred gradually with increase in annealing temperature while in the case of Ni-18.5 wt% Fe, such a change was rapid at 573 K. Grain growth rate depended on the orientation and  $\{111\}$  grains grew at a faster rate than  $\{200\}$  grains and other grains with different orientations indicating the occurrence of abnormal grain growth.

*Keyword:* Electrodeposited nanocrystalline Ni-Fe alloys, GIXRD, XPS, Texture, Annealing, Surface oxides.