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Solid-state reactions and microstructure in the Ni-Ti-O system

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Abstract – A two-phase ceramic material consisting of $NiTiO_3$ (ilmenite phase) and NiO has been studied as a model system for investigating phase evolution and precipitate formation at different annealing temperatures. A variety of electron microscopy techniques have been used to characterize the microstructure. This presentation will discuss the interaction of the phase transformation behavior and the topography of the sample surface and discuss the relevance of the surface grooves to the study of triple junctions in other ceramics, including those used for SOFCs.

The specimens have been characterized using a combination of field emission scanning electron microscopy (FESEM), energy dispersive spectroscopy (EDS), atomic force microscopy (AFM), electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). Interesting geometrical patterns of precipitates are observed after heating the samples at 1200 °C and 1400 °C for 0.3 hr. While TEM confirms the presence of NiO, $NiTiO_3$ (ilmenite) and Ni_2TiO_4 (spinel) phases before heat treatment, EBSD and EDS indicate the appearance of $NiTiO_3$ precipitates with preferred orientations in the surrounding matrix of NiO after heat treatment. TEM of focused-ion beam (FIB) milled specimens revealed the orientation relationships, subsurface morphology and the presence of interfacial dislocations. TEM results indicate that dislocations play a significant role in the phase transformation and that the precipitate morphology is strongly influenced by the presence of the sample surface. Thermal grooving occurs at the interfaces between the $NiTiO_3$ precipitates and the NiO matrix. The widths of the precipitates increase, and morphology and spacing change with temperature. A periodic hill-and-valley morphology appears on the precipitate surface.