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Solute distribution in electrodeposited Ni-Mn alloys by atom probe tomography

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The combination of high mechanical strength with thermal stability and low residual stress in electrodeposited (ED) Ni is highly desirable for many applications, though rarely achievable in practice [1]. High strength and hardness generally require extremely fine grain size, which in turn, leads to greater driving force for recrystallization and grain growth, and therefore, decreased thermal stability [1]. Mn additions, however, lead to enhanced thermal stability [2], manifested as resistance to both anneal softening and grain growth (Fig.1). This work aims at elucidating the responsible mechanisms.

Characterization of the compositional structure of these materials at a nanometer scale is challenging because of both the fine scale of the microstructure and the low solute concentrations. We have used atom-probe tomography (APT) to provide a description of the spatial distribution of Mn and other solutes in dc and pulse-plated Ni-Mn alloys after deposition and subsequent annealing treatments. Films are deposited from a Ni sulfamate solution at 3 mA/cm^2 for dc plating and with alternating current between 3 and 15 mA/cm2 during pulse-plating.

Despite the different plating conditions, Mn modulations are observed in both dc and pulse plated films (Fig.2). Depending on the location in the deposit, these modulations take the form of reasonably well-defined layers or more complex, ill-defined, three-dimensional structures. No concentration enhancement of any solute is observed at grain boundaries in the as deposited state.

After annealing at 600^oC for 1 hour that corresponds to the onset of grain growth for the Ni-Mn alloys, the Mn concentration modulations disappear by diffusion, and segregation of Mn, C and S/O is observed at some of the grain boundaries, possibly affecting grain growth kinetics. Small nanometer-sized Mn-rich clusters are also observed (Fig. 3). The composition suggests the formation of the metastable Ni_3Mn phase, with S and/or O local concentrations up to 2 at.%. The S/O segregation behavior at the interface of these precipitates may constitute an efficient mechanism for preventing sulfur and/or oxygen from segregating to the grain boundaries. Evidence of grain boundary pinning given by transmission electron microscopy observations could also imply slower grain boundary velocities than in the case of ED Ni films.

[1] J.W. Dini, Electrodeposition: the Materials Science of Coatings and Substrates, Noyes Publication: New York, 1993
[2] J.J. Kelly, S.H. Goods, N.Y.C. Yang, Electroch. and Solid-State Letters, 6 (6) (2003) C88
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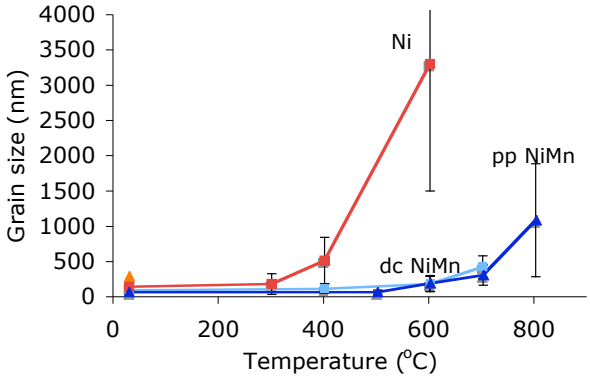

Fig. 1. Grain size versus annealing temperature for Ni sulfamate, dc Ni-Mn and pulse plated Ni-Mn films. All annealing times are 1 hour.

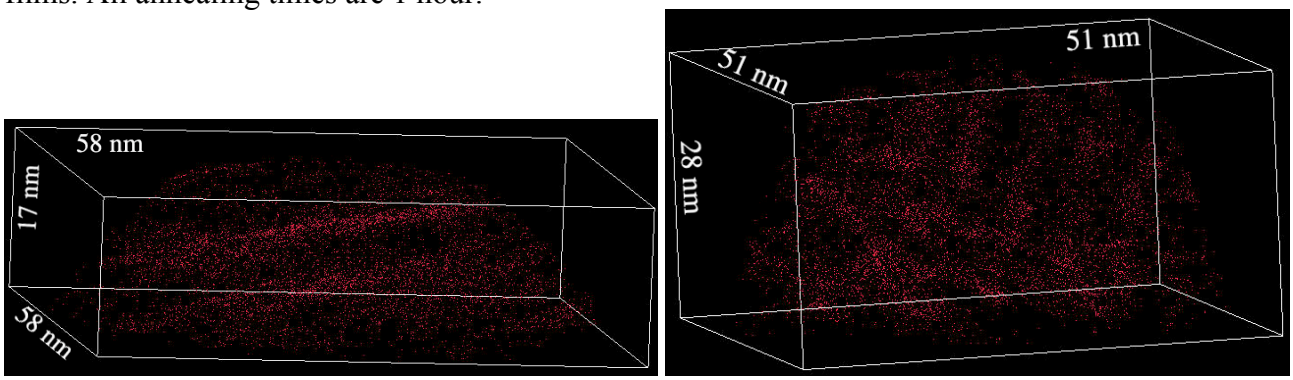

Fig. 2. 3D reconstruction of as-deposited NiMn films (a) dc plating condition (b) pulse plating Mn atoms are in red, all other atoms have been omitted for image clarity.

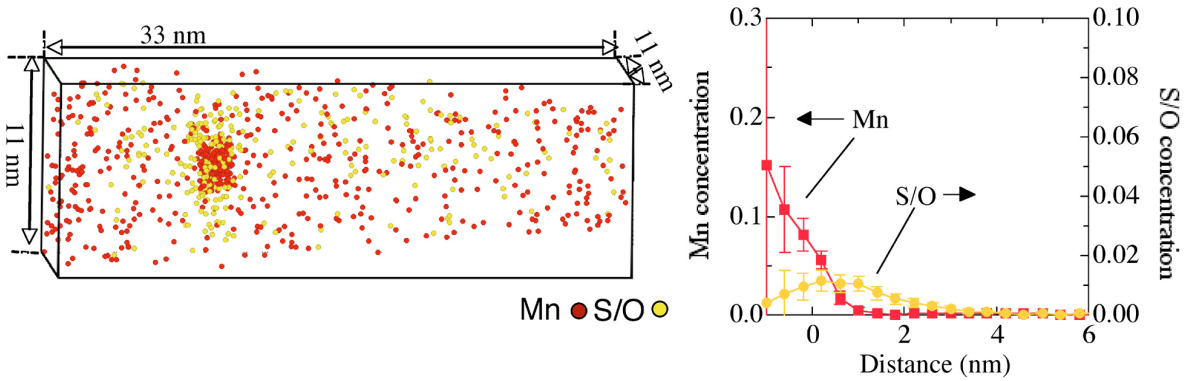

Fig. 3. 3D reconstruction of a pulse plated NiMn film after annealing at 600°C for 1 hour. Mn atoms are in red, S/O in yellow and all other atoms have been omitted for image clarity. The proximity histogram of the Mn-rich precipitate indicate S and/or O segregation at its interface.

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