Fabrication of Super Invar Micro- and Nano- Structures by Electrodeposition for Low Thermal Expansion Applications

Hana Kim

Department of Chemical Engineering,Northeastern University, Boston MA 02115

Electrodeposited alloys of Super Invar composition, 64 wt % Fe, 31 wt % Ni, and 5 wt % Co, are of both theoretical research and practical interest due to the anomalous codeposition behavior, the preferential deposition of less noble metal, and to the material's low coefficient of thermal expansion, CTE. A better understanding of anomalous codeposition allows for controlling the deposition rates of individual Fe, Ni, and Co metal during codeposition and hence the composition. In addition, electrodeposited Super Invar alloys are of practical interests due to their potential applications in industry, particularly in micro-electro-mechanical systems (MEMS) due to the material's near zero CTE at room temperature in bulk materials. In practice, however, it is difficult to predict the alloy composition and control the composition gradient along the direction of growth, due to the anomalous codeposition behavior. Super Invar has not been widely studied; most studies of the ternary FeNiCo have been focused on Co-rich alloys due to their magnetic property over the last twenty years. In addition, there is an absence of understanding the CTE in electrodeposited Super Invar structures and how it is influenced by the electrodeposition parameters.

The objective of this research is to study the electrodeposition of Super Invar alloy thin films and micro-posts, and nanowires. First, thin film deposition of FeNiCo alloys is investigated in order to determine the most appropriate applied current density into desired recessed features. Based on the conditions of thin films, different pulse plating parameters are applied and the deposit composition distribution along the length of the microstructures ~~was~~ examined. Pulse plating is critical in achieving a uniform composition distribution in 100 μm deep features. Using a pulse scheme with a low, non-zero current step introduces more cracks into the deposit, characterized by SEM. The coefficient of thermal expansion (CTE) of the micro-post arrays is also determined and exhibits negative values in the axial direction, *i.e.*, the micro-posts shrink with temperature. Structures with micron size cracks exhibit large, negative CTE values during the first heat cycle of the CTE test and then exhibited less dimensional change with a subsequent heat cycle. The effect of the additives: glycine, Cu, and 2-butyne-1,4-diol (BD), into the Super Invar electrolyte is examined for better appearance, corrosion resistance and for the fabrication of Super Invar nanowires. Addition of glycine is desirable to thin film deposition in order to fabricate a surface smooth, acting as a leveling agent, however, it was not helpful to micro-recessed pulsed electrodeposition because it did not eliminate cracking, that may be due to local pH rises and corrosion during the relaxation part of the pulse. Corrosion resistance is improved when Cu is added into Super Invar electrolyte, but Cu ions simultaneously reduce in the electrolyte during deposition. To increase the stability of this electrolyte, potassium tartrate and Triton X-100, were added in FeNiCoCu. With these additives, the ratio of Fe to Ni and Fe to Co is altered. Microposts deposited were very dendritic on account of the transport controlled Cu reaction. With the presence of BD, a known corrosion inhibitor, each Fe, Ni, and Co reaction rate during deposition is altered, depending on different rotation rates, and different BD concentrations. FeNiCo nanowires without and with BD were prepared and the corrosion that is observed during pulsing is decreased when BD was added.