Microstructure and Mechanical Properties of Nanocrystalline Ni-Al₂O₃ Composites by Electrodeposition

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Abstract – Nanocomposite materials consisting of a nanocrystalline Ni matrix (grain size 50-60 nm) and nano-size Al₂O₃ particulates (average particle size: 30 nm) up to 1.5 wt.% have been synthesized by electrodeposition. Hardness of Ni–Al₂O₃ nanocomposite was about 1.7 times that of nanocrystalline pure Ni. However, tensile test of Ni–Al₂O₃ nanocomposite decreased to about 1/2 times that of nanocrystalline Ni. Fracture surface exhibited a typical dimpled surface with dispersed Al₂O₃ particles on it. Concentration of Al₂O₃ on the fracture surface was more than 3 times that of the bulk concentration. Therefore it is considered that the aggregated Al₂O₃ particles caused initiation of cracks and the reduction of tensile stress.

It has been reported that nanocrystalline Ni with grain size less than 100 nm can be synthesized by electrodeposition, and they exhibited extremely high strength by grain refinement. If nanocrystalline materials contain precipitations of nano-size, they may exhibit higher strength by both grain size reduction (Hall-Petch law) and precipitation hardening. The purpose of the study was to fabricate a nanocrystalline Ni-Alumina composite by electrodeposition method and to characterize their microstructure using SEM, TEM, analysis and to determine mechanical properties.

Nanocrystalline Ni-Al₂O₃ composites were electrodeposited in the bath of consisting of 500g/ℓ nickel sulfamate, 15 g/ℓ nickel chloride, 30 g/ℓ boric acid and 2g/ℓ saccharin. γ-Al₂O₃ powders (30nm in average diameter) were suspended in the bath. The pH was adjusted to a constant value of 4 by sulphuric acid at 50±1°C. Direct current was used, and the current density was 30mA/cm². A nickel plate of 99.9% purity was used as the anode, and stainless steel plate was used as the cathode. Plating time was 3 h, and always stirring it by a magnetic stirrer placed at the cell bottom. Grain size was determined by direct measurement on transmission electron microscope (TEM, JEM2100F) bright-field micrographs.

Observation by TEM on Ni-Al₂O₃ composite revealed the average grain size was 55nm. EDS map of the sample is shown in Fig.1. The Al₂O₃ particles are uniformly distributed throughout the Ni matrix. However, many agglomerates of Al₂O₃ particles with cluster sizes about 5µm are also observed. Relationship between Al₂O₃ concentration in the bath and amount of deposit is shown in Fig.2. The amount of Al₂O₃ deposit increased with amount of Al₂O₃ concentration in the bath to 40g/ℓ, but it decreased in 50g/ℓ. It seems that there is an optimum contents of alumina powders in the bath. Relationship between Al₂O₃ content in the deposit and Vickers microhardness is shown in Fig.3. The Vickers microhardness increased with amount of Al₂O₃ content.

Fractured surfaces were observed after tensile test. The surface exhibited a typical dimple surface containing Al₂O₃ particles in the bottom. Some Al₂O₃ particles were cohered in the fracture face. The Al₂O₃ content of fracture surface by EDS analysis was 5-8 wt.%, and it is higher than that of the bulk content. Therefore, it can be considered that the cause of the decrease tensile stress is cohesion of Al₂O₃ content.