

CHARACTERIZATION OF NICKEL-BASED IRON-ZINC COMPOSITES SHEET FORMED BY ELECTROFORMING

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Introduction

Nickel based composites sheet is useful for electronics and information display materials because of its high conductivity and deformability [1]. The nickel composites sheet can be formed by various methods like electroplating, electro-less plating and other mechanical deforming processes [2]. Although each process can make nickel sheet, its physical and chemical properties are slightly different due to microstructure and impurity because nucleation and growth behaviors of the nickel grains are dependent upon fabrication environment [3]. That kind of little difference of the properties is so important for the fabrication of high quality products in a leading edge science and industrial field. Recently, electroforming is one of useful techniques to prepare thin and thick sheet for industrial materials. Although the electroforming of pure nickel has been well established, little information have been available, especially about nickel based composite sheet for electromagnetic insulator [4]. Hence, nickel-based iron-zinc composites sheets were fabricated by electroforming followed by cold rolling. Emphases are on the analysis of mechanical and corrosion behaviors with its composition.

Experimental Methods

Electroforming was carried out in a rotating drum-cell containing a modified nickel sulfate bath in which iron and zinc ions are present. Fig. 1 is schematic diagram of the electroforming unit. Table 1 is the solution chemistry of the nickel sulfate bath. The anode and the cathode were titanium electrode and 316 stainless steels, respectively. Electroforming was carried out at the condition of 45-55°C, pH=4.8±0.1, 7-10 mA/dm², and 6V. Ultrasonic agitation at 500W was applied to electro-deposition cell with an ultrasonic generator (BH-50, Korea) during electro-deposition. The electroplating was carried out with a regulator (Jisan-400, Korea) and electroformed nickel strip was separated from

cathode by using a doctor blade. Microstructure observation and chemical analysis of the products were performed by using transmission electron microscopy (HR-TEM, JSM Joel 2010-H, Japan) and energy dispersive spectroscopy (EDX, Oxford, UK), respectively. TEM specimen was prepared by twin jet-polishing (Struers Tenupol-3, Denmark). Nano-indentation (Park's Scientific Inc. USA) was carried out to determine hardness change of the composite sheet at 500 µN.

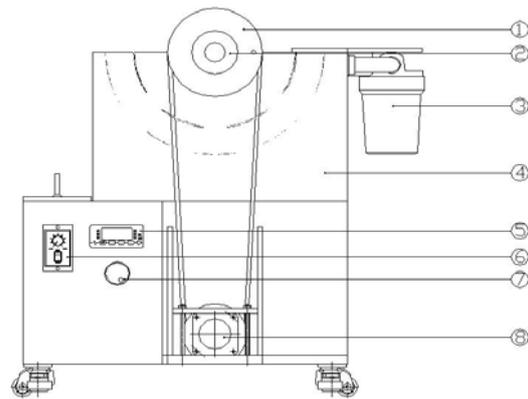


Fig. 1. Schematic diagram of the electroforming unit : (1) aluminum drum (2) pulley (3) filtration (4) bath (5) temperature controller (6) drum speed controller (7) main switch (8) motor

Table 1. Solution chemistry of a modified nickel sulfate bath [wt. %]

NH ₄ Ni(SO ₄) ₂	NiCl ₂	H ₃ BO ₃	ZnCl ₂	FeCl ₃
79	4	8	1	0.8

Results and Discussion

Since electroformed nickel composites sheet is relatively thin thickness of about 500 nm, nano-indentation technology was applied to determine hardness after fine polishing its surface with diamond paste. Hardness of determined by nano-indenter was 6.7 GPa for pure nickel sheet, whereas 7.1 GPa for composites sheet, respectively. The enhanced hardness of the

composites is related to solid solution hardening or second phase hardening due to addition of iron and zinc in the nickel matrix. Fig. 2 is TEM image and selected area diffraction (SAD) pattern of nickel-based iron zinc composites sheet. As shown in Fig. 2, matrix is polycrystalline nickel in which dislocation and twin are present. Fig. 3 is EDX spectra of the sheet, in which the sheet is mainly nickel and additional iron and zinc are clearly observed. It means that nickel based iron-zinc composite sheet can be formed by electroforming with a modified nickel sulfate bath with zinc and iron chlorides.



Fig. 2. TEM image and SAD pattern of nickel based iron-zinc composites sheet

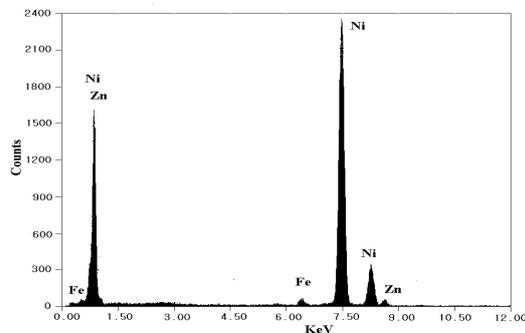


Fig. 3. EDX spectra of nickel based iron-zinc composite sheet

Table 2 shows the corrosion potentials and rates of the electroformed nickel sheets in aqueous 50% NaOH solution with chloride ions. The corrosion potential and rate of pure nickel sheet in 50% NaOH solution were $-782.5 \text{ mV}_{\text{SHE}}$ and $3.6 \times 10^{-4} \text{ Acm}^{-2}$, respectively. Those of the composites in 50% NaOH solution were $-860.7 \text{ mV}_{\text{SHE}}$ and $4.8 \times 10^{-4} \text{ Acm}^{-2}$, respectively. When the chloride ions were present in the corrosion solution, the corrosion potential decreased, whereas, the corrosion rate increased with and without iron and zinc content in composites sheets.

Table 2. Corrosion potential and corrosion rate of nickel based iron-zinc composites sheet with chloride ion content

HCl ₂ content [%] in 50% NaOH	Corrosion Potential [mV_{SHE}]	Corrosion Rate [$10^{-4} \times \text{Acm}^{-2}$]
0	-860.7	4.8
3	-974.2	7.9

It is difficult to find the site where corrosion initiates at this moment. However, the fact that electromotive force (EMF) of nickel is higher than those of iron and zinc supports that pure nickel is less active than the nickel composites in the corrosive solution. Hence, the corrosion resistance was decreased by adding iron and zinc because less EMF and the chloride ions which act as aggressive ions and to make pits on the metallic surface.

3. Conclusion

Nickel based iron-zinc composites sheets were fabricated by electroforming with a modified nickel sulfate bath with iron and zinc chloride. The composite sheets are mainly polycrystalline nickel with iron and zinc inside. Hardness increased about 16% by adding iron and zinc. Corrosion resistance of the nickel based-iron-zinc composites sheet in aqueous 50% NaOH decreased, especially in a corrosive solution with chloride ions

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