Electrodeposition of Nano-crystalline Nickel-Molybdenum Alloys

R. Washio¹, M. Mizumoto², T. Ohgai²*, A. Kagawa², K. Takao²
N. Uchida³, Y. Tanaka³ and S. Sumita³

¹Graduate School of Science & Technology, ²Department of Materials Science and Engineering,
³Nagasaki University, 1-14 Bunkyo-machi, Nagasaki 852-8521, JAPAN
*Tel/Fax: +81-95-819-2638, E-mail: ohgai@nagasaki-u.ac.jp

Abstract
Nano-crystalline nickel-molybdenum alloys were electrochemically synthesized from aqueous solution. With increasing molybdenum content in the alloy up to 23.6%, the crystal grain size decreased down to several nanometers scale. The magnetic coercive force decreased to several oersteds with increasing the molybdenum content. Soft magnetic property of the alloy was improved compared with that of pure nickel.

Keywords: electrodeposition, nano-crystal, nickel, molybdenum, magnetic property

Introduction
It is well known that metallic molybdenum can not electrodeposit from aqueous solution containing molybdate ions only. However, in the solution containing iron-group metal ions such as Ni²⁺, Co²⁺, and Fe²⁺, molybdenum can co-electrodeposit with iron-group metal as metallic state. Usually the electrodeposited iron-group metal and molybdenum alloy consist of small crystals with the diameter less-than one micrometer. On the other hand, nano-crystalline iron-group metal shows excellent soft magnetic properties, which can be applied to high sensitive magnetic field sensors [1, 2]. In this study, structure and magnetic property of electrodeposited nano-crystalline nickel-molybdenum alloy were investigated.

Experimental
Aqueous electrolytic solution was synthesized from nickel sulfate, sodium molybdate, and sodium citrate. Optimum condition for nickel-molybdenum alloy deposition was determined by the cathodic polarizarion curve measured at a wide potential range. Nickel-molybdenum alloy was electrodeposited potentiostatically on a copper sheet. To observe the cross-sectional area of electrodeposited alloy films, the samples were whittled using focused ion beam (FIB). Transmission electron microscope (TEM) was used for observing the structure of nano-crystalline nickel molybdenum alloy. The magnetic property was investigated by using a vibrating sample magnetometer (VSM).
Results and Discussion

Figure 1 shows scanning ion microscopy (SIM) images of cross-sectional area in the pure nickel film (Ni) and the nickel-molybdenum alloy films (Ni-Mo) electrodeposited on copper electrode. Electrodeposited Ni consists of large crystal grains with the diameter of several micro-meters, while Ni$_{95.5}$Mo$_{4.5}$ alloy consists of pillar-shaped crystals oriented vertically to the film surface. On the other hand, Ni$_{76.4}$Mo$_{23.6}$ alloy consists of very small crystal grains with the diameter of several nano-meters. With increasing Mo content in the alloy, the film thickness was decreased. The film thickness of electrodeposited Ni was around 12 μm, the Ni$_{95.5}$Mo$_{4.5}$ alloy was around 5.6 μm, and the Ni$_{76.4}$Mo$_{23.6}$ alloy was about 2.8 μm. These results were determined by the decreasing current efficiency due to increasing molybdate ions in the electrolytic solution. The transmission electron diffraction pattern obtained by electrodeposited Ni was consisted from spots, which corresponds to the typical large crystals. While, the pattern of Ni$_{95.5}$Mo$_{4.5}$ alloy was consisted from rings and the one of Ni$_{76.4}$Mo$_{23.6}$ alloy was hallow pattern, which corresponds to the typical nano-crystalline or amorphous phase. Magnetic coercive force obtained from Ni-Mo alloy was decreased down to several oersteds with increasing Mo content.

Conclusion

Ni-Mo alloy consists of nano-sized grains was synthesized from aqueous solution. With increasing Mo content in the alloy, the film thickness was decreased due to decreasing current efficiency of the alloy deposition. Magnetic coercive force of the alloy decreased down to several oersteds with decreasing grain size of the alloy deposit.

Acknowledgements

This work was supported in part by TDK Corporation, Mitutoyo Association for Science & Technology, Yazaki Memorial Foundation for Science & Technology, Research Foundation for Materials Science, Japan Society for the Promotion of Science (Grant-in-aid for Scientific Research C : No.19560734).

References