Analysis of effect of additives on crystalline orientation of Ni plating by EBSD

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1. Introduction
Ni plating has been applied to various substrates to improve, for example, their corrosion resistance and appearance. For this purpose structure of plating film, grain size and surface flatness are optimized by using various additives. The actual functions of such additives are, however, not yet understood. In this study, effect of current density and additives on structure and crystalline orientation of Ni plating films was examined by using electron backscatter diffraction (EBSD).

2. Experiments
Ni films of 10 μm in thickness were electrodeposited on Cu substrate from watts bath at 100 Am⁻² for 48 min. 2-butyne-1,4-diol (BD) at 0.0, 0.2, or 1.0 mM and saccharin (SC) at 10 mM were used as additives. For cross-sectional observation and analysis using FE-SEM and EBSD, samples were embedded in conductive resin (Technovit5000), mechanically polished, and etched with Ar ion beam to remove polishing scar.

3. Results and discussion
Crystallographic orientation maps measured for cross-section of Ni films obtained by EBSD are shown in Fig. 1. Film deposited at 0.0 mM BD consisted of columnar grains growing almost perpendicular to the surface (Fig. 1a). Grain size was small at the Ni / Cu interface, and became large with thickening the film. The grains became fine with increase in concentration of BD (Fig. 1b). Almost linear relationship between mean cross-sectional area of grains and BD concentration was found as shown in Fig. 2, confirming that the gain size decreases with increasing in BD concentration. Orientation of grains in the film was mainly (110) at 0.0 mM BD and (111) at 1.0 mM BD. This result also consisted with XRD spectra measured for the films deposited in the same condition. Addition of 10 mM SC to plating bath containing 1.0 mM BD shows drastic refinement of grain size and change in orientation, as shown in Fig. 1c. Combination of BD and SC is used as effective leveler to obtain flat plating surface. The role of this co-adsorption is not yet clarified.

![Fig. 1 EBSD maps of cross-section of Ni plating layer deposited on Cu substrate. a) 0.0 M BD, b) 1.0 mM BD, c) 1.0 mM + 10 mM saccharin.](image1)

![Fig. 2 Dependence of mean grain cross-section area on BD concentration.](image2)