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X-Ray fluorescence imaging of Co/Cu electrodeposits

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Introduction

Novel X-ray fluorescence (XRF) imaging has been developed recently [1]. This method is based on the excitation of the whole specimen surface by a wide beam and detection of the fluorescent X-rays by a two dimensional detector. It offers fast imaging as compared to conventional 2D–scanning technique.

The objective of this work was to study electrodeposition of metals from mixed solutions. While electro–analytical techniques have a long history in preparation of common alloys [2], there are some metals that are being scarcely investigated at all. XRF imaging was used to determine the geometrical and elemental structure of deposited metals (Co and Cu) in a thin electrolysis cell.

Experimental

Stock solutions (0.1M) were prepared from $CoCl_2$ and $CuSO_4$, mixed (1:1), and presented for electro deposition. A 2.6cm diameter electrolytic cell was used with a Ni cathode. Constant potential was maintained during the electrolysis, U=7V. Details of the XRF imaging camera system are described elsewhere [1]. By changing the excitation energy, XRF images can be obtained for Co and Co+Cu. The X-ray images were recorded for 1s acquisition and 2s read-out time.

Result and Discussion

The binary system of Co and Cu is immiscible. A single-phase solid solution was obtained by quenching, where the alloying was restricted to few percent of Co (or Cu) [3]. An early electrochemical study concluded with a gross admixture of the two metals [4]. Optical images of Co-Cu deposit in our cell seem to exhibit phase separation in agreement with previous observations. The deposit showed two alternating colors: reddish and white parts.

Observation by optical microscope can be performed, but it is not easy to differentiate between elements in the optical image by color. Elemental mapping however, is an inherent capability of XRF imaging. The XRF images of Co-Cu deposit are shown in Fig.1 and Fig.2, obtained at different excitation energies. Looking at the images, they seem to be quite similar. That means Co and Cu are not segregated completely, but distributed almost homogeneously in the deposit. Another feature of the deposition process is the Cu-rich area around the cathode relative to Co. Evaluation of the optical image of deposit revealed a characteristic feature: the outer rim is a fine geometry with fractal dimension, FD=1.2–1.3, and the inner part is space filling.

Electroplating is known to produce compact metal at low current densities, therefore it has practical importance. The structure of the electrodeposited metals can be a very fine micro-crystal phase. This provides an opportunity for the interpretation of our results. Microstructure investigation of the Co-Cu deposit is proposed. The

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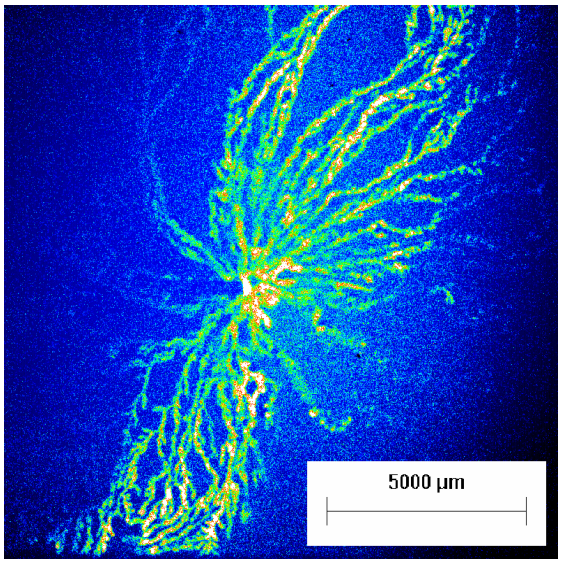

Figure 1: XRF image of Co-Cu deposits at E=10keV (combined image of Co and Cu)..

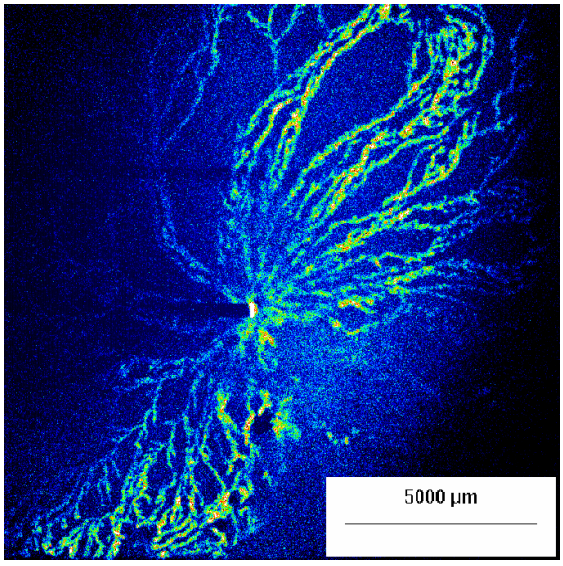

Figure 2: XRF image of Co-Cu deposits at E=8.6keV (image of Co).