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May 1989



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**APPLICATION OF VIDEOMICROSCOPY TO IN-SITU
STUDIES OF ZINC ELECTRODEPOSITION**

by

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In electrodeposition processes, surface texture development is of considerable scientific and technological interest. Although many researchers have used ex-situ techniques to study deposit structures, these methods don't allow the uninterrupted observation of the developing morphology over the long periods of time typically required for the formation of thick deposits. In this investigation, the development of a videomicroscopy technique is reported that can be used for in-situ studies of microscopic-scale surface features.

The growth of zinc striations on a synthetic graphite substrate from flowing, well supported, acidic chloride electrolytes was observed to evaluate the capabilities of the video microscopy system. Micrographs were made at a magnification of 160X, which corresponds to a video image width of 800 μm . The videotapes were recorded while the experiment was in progress and then edited to produce time lapse movies of the salient features of slowly occurring events. Still photographs were taken from the videotape with a Kodak SV6500 color video printer.

As illustrated in the videomicrographs of Figure 1, several steps are involved in striation formation. Nodules are first deposited randomly over the cathode surface (1a). With the passage of time, the protrusions coalesce (1b), and some agglomerates become large enough to affect the flow in the boundary layer. The eddies formed reduce the mass transport overpotential, causing the growth rate to increase in the direction parallel to the electrolyte flow (1c). As more charge is passed, the nodules coalesce to form ridges which then grow evenly in all directions.

Videomicroscopy can be used to record the progress of electrodeposition in real time. The technique shown here is currently capable of recording the deposition process at a maximum magnifications of 375X, allowing

observation of an electrode area of approximately $300 \times 200 \mu\text{m}$. A magnification of 600X can be achieved with a more intense light source and a higher power eyepiece. An objective lens with a long working distance has been used (50X, N. A. = 0.6). It provides a diffraction limited resolution of $0.5 \mu\text{m}$; the actual resolution is somewhat less because the lens is not designed for the 2 mm thick window used in the channel flow cell. Several areas of the surface can be quasi-simultaneously observed by using a traveling stage, allowing the experimenter to determine the consequences of surface events. With fiberoptic light sources, the images obtained can be of remarkably good quality.

ACKNOWLEDGEMENT

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REFERENCE

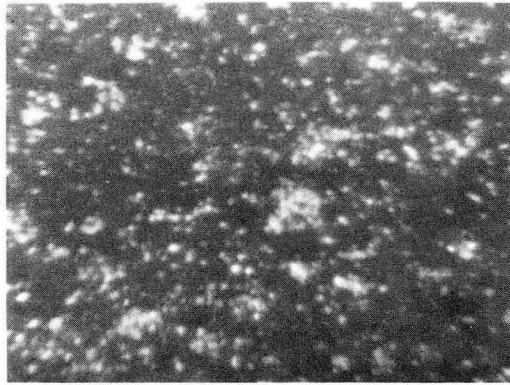
1. Mc Vay, L., R. H. Muller and C. W. Tobias, Application of Videomicroscopy to In-situ Studies of Electrodeposition, LBL report no. 26426, December (1988), Submitted to J. Electrochem. Soc.

FIGURE CAPTION

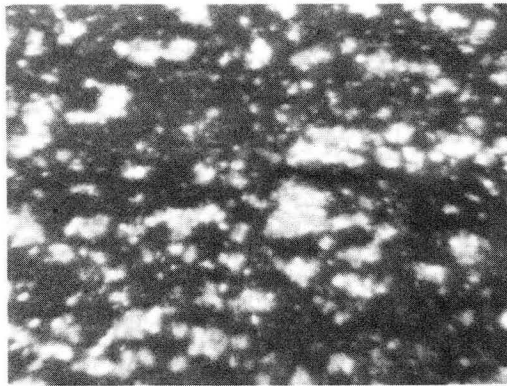
Figure 1- Genesis of a striation from a set of unaligned protrusions: (a) 1 min; (b) 3 min; (c) 5 min.

Deposition conditions: 3M ZnCl₂, 3M KCl, pH = 1.5, 28.5 mA/cm², Re = 4000, 800 μm/image width.

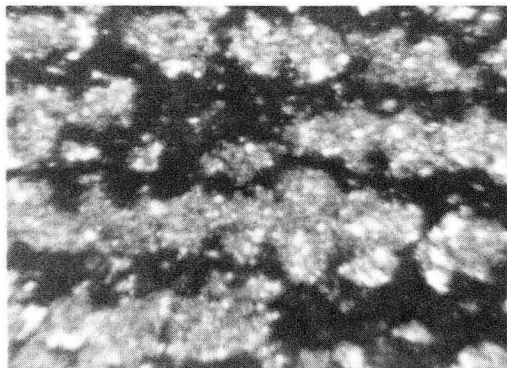
The flow direction is from left to right.



(a)



(b)



(c)

XBB-892-1243A

Figure 1

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