DOE/TE/00070--T/

FINAL REPORT

**ENVIRONMENTAL SCANNING ELECTRON
MICROSCOPE (ESEM)**

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UNIVERSITY RESEARCH INSTRUMENTATION
PROGRAM**

GRANT: DE-FG02-95TE00070

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INTRODUCTION

The Environmental Scanning Electron Microscope (ESEM) was acquired by a grant from the Department of Energy University Research Instrumentation Program and matching funds from Lehigh University and industry. The equipment is installed as part of our electron microscopy laboratories and is being utilized on a regular basis. Over 20 graduate and undergraduate students from the Department of Materials Science and Engineering as well as other departments in the University have included this instrument in their research. In addition, the ESEM has been used in several courses including MAT 427 - Advanced Scanning Electron Microscopy, a graduate course offered every other year. What follows are examples of how the ESEM has been included in our research programs.

ESEM RESEARCH EXAMPLES

ELECTRODEPOSITED COATINGS

Nickel matrix/aluminum particle composite coatings were produced by an electrodeposition process. Figure 1 displays a light optical (LOM) photomicrograph of an as-deposited coating. After deposition, the coatings were heat treated to produce a Ni-Al alloy. The heat treated microstructure contains Ni_3Al blocks in a γ solid solution matrix as shown in Figure 2. Kirkendall voids are also present throughout the coating. The diffused Ni-Al coatings are expected to have excellent high temperature corrosion resistance. The objective of the environmental scanning electron microscope (ESEM) study was to directly view the morphological changes which occur during heat treatment, i.e. from the coating morphology in Figure 1 to the morphology in Figure 2. The coating *surface* was viewed in the hot stage of the ESEM during heating to and isothermal holding at 600°C for approximately one hour. Although it was difficult to discern the morphological changes taking place, some evidence of heat treatment and oxidation were found during the ESEM study. To improve the imaging quality, future ESEM work will be performed on polished and lightly etched coating *cross-sections* rather than on the coating surface.

OXIDATION BEHAVIOR OF IRON AND ALUMINUM

The initial oxidation behavior of plain carbon steel (AISI 1018), Figure 3, and commercially pure (99.9%) aluminum, Figure 4, was investigated in an environmental scanning electron microscope (Electroscan 2020) during heating between 25°C and 660°C at 25°C/s . Dry air was injected directly above the sample heating stage using a special injection system. Water vapor (H_2O) pressure in the chamber was adjusted as necessary for sample imaging. Samples were wet ground using 1200 grit SiC abrasive paper. During heating, oxide whiskers (diameter $\sim 200\text{nm}$) nucleated on the surface of the iron sample but sample vibration hindered more quantitative measurements. Once at temperature, the oxide whiskers were prominent and filamentary whiskers also appeared within 30 minutes. In contrast, the surface of the aluminum remained featureless upon heating from 25°C to 660°C (near the melting point) for a period extending over 30 minutes.

PASSIVATION IN MICROELECTRONIC INTERCONNECTS

Environmental scanning electron microscopy has been used to image microelectronic interconnects under the insulating passivation layer while maintaining sufficient resolution for meaningful analysis. In situ electromigration lifetime tests were also performed on an exploratory basis and recorded on videotape. Figure 5 contains images captured from the videotape over a time span of about 5 seconds just before catastrophic failure of the line, with experimental conditions listed below. Arrows mark equivalent positions in each image, and evidence of void nucleation, growth, motion and coalescence can clearly be seen.

ZnO DUST IN FILTER BAGS

The Electroscan ESEM 2020 was used to analyze filter bags used in a baghouse to remove ZnO dust from a stream of hot air. As ZnO dust collects on the surface of the filter bag, reducing its permeability, the filter bag is periodically shaken to remove the ZnO dust and allow more to collect on its surface. Over time, a hard substance collects on the surface of the filter bag which is not removed with ZnO dust during the periodic shakings. This layer eventually clogs off the filter bag and must be scraped off by hand. The sponsor was interested in trying to determine if the suspect layer could be imaged in the ESEM and composition of the layer determined using EDS X-ray Microanalysis and X-ray mapping. XRD of scrapings from the filter bag suggested that a deliquescent compound such as ZnCl_2 was present in the suspect layer. Analysis began with the preparation of cross-sections of the filter bag and oxide layers (60 μm thick). These cross-sections were imaged in the ESEM at 20 keV under a water vapor partial pressure of 1.6 Torr, which allowed for observation without the need for specimen coating. SE images of the filter bag/oxide cross-sections were taken at 500x. EDS spectra were collected for 100 sec. live time and 20-40% dead time. The EDS spectra were used to set up energy windows for which x-ray maps (128 x 128 pixels) were produced using a 100 ms dwell time per pixel. A composite micrograph of the cross-section showing the SE image and x-ray maps for Zn and O can be seen in Figure 6. X-ray maps for additional elements such as Cl, Na, S, Pb, F and K were also collected, but unfortunately did not provide much information as to the composition of the suspect layer. Difficulties in the preparation of the smooth and undisturbed oxide cross-sections was a major limitation in the analysis of these samples.

DYNAMIC ANALYSIS OF CLAY PLATELETS

Scanning electron microscopy (SEM) has been extensively used to examine individual and groups of clay platelets at high magnification. SEM and other optical and electron microscopy require that the pore fluid be removed, replaced, or frozen. To remove the pore water without disturbance of the original fabric is difficult. The environmental scanning electron microscope (ESEM) is a relatively new instrument that examines materials including liquids and oils in their natural state with no prior sample preparation. The ESEM is able to examine materials including liquids and oils in their natural state with no prior sample preparation. The environmental chamber can contain gas or vapor at a pressure as high as 50 Torr. The gas in the environmental chamber operates as a cascade electron multiplier to enhance the secondary electron signal and to provide a source of positive ions which neutralizes any negative charge which might accumulate on the sample. The ability to manipulate temperature, pressure, gas and add or remove fluids in the ESEM greatly increases the range and diversity of experimental conditions. Accessory equipment for cooling, heating, and applying tension and compression makes it possible to image the effects dynamic processes such as freezing, melting, desiccation, and consolidation on soil microstructure. In this study, the ESEM was used to quantitatively analyze the effect of dynamic environmental processes on the microstructure of clays. Testing was conducted on Niagara clay using the high temperature stage. Saturated clay samples were placed into the ESEM chamber, and the temperature of the samples were increased. When a specific temperature (100°, 200°, and 300°C) was achieved, a micrograph was obtained, Figure 7. Initial and final micrographs were compared and analyzed using digital image processing.

SUMMARY

The Environmental Electron Microscope (ESEM) has become an integral part of our Electron Optics Laboratory and is contributing to many of our research programs while training undergraduate students and graduate students in this novel microscopy technique.

Figure 1. LOM photomicrograph of an as-deposited Ni matrix/Al particle composite coating.

Figure 2. LOM photomicrograph of the coating morphology after heat treatment at 600°C.

Figure 3. Oxidation of AISI 1018 Steel After 30 Minutes.
[20kV, GSED, 650°C]

Figure 4. Oxidation of Commercially Pure Al After 30 Min.
[15kV, GSED, 663°C]

In situ electromigration test

Figure 5

Al-4wt%Cu under 1 μm passivation

Conditions: $J = 10 \text{ MA/cm}^2$, $T \approx 450^\circ\text{C}$, e^- flow left to right
30 kV, WD=7.5 mm, 2.5 Torr, image width=20 μm

Figure 6

**Figure 7a. ESEM Micrographs of
Niagara Clay at 295 x
Before Drying**

**Figure 7b. ESEM Micrographs of
Niagara Clay at 550 x at
a Temperature of 199°C**