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PARTIAL ANALYSIS OF EXPERIMENT LDEF A-0114

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Dr. J. C. Gregory, Principal Investigator

Institute for Surface Science
and Chemistry Department
The University of Alabama In Hunstville
Huntsville, AL 35899

Introduction

Due to delays in manifesting the return of the Long Duration Exposure Facility from space, attention has remained concentrated on extracting the maximum information from the EIOM-2 experiment flown on STS-8 in September 1983. During the period covered by this report an analysis was made of the optical surfaces exposed during that flight and an assessment made of the effect of the 5eV atomic oxygen upon their physical and chemical properties. This work, which has now appeared in the journal "Applied Optics" Vol. 25, page 1290 on April 15, 1986 was entitled "Effects on Optical Systems from Interactions with Oxygen Atoms in Low Earth Orbits". Its authors were P. N. Peters, J. C. Gregory, and J. T. Swann.

Experimental Measurements

Surfaces of various samples were exposed in the orbital direction as part of The University of Alabama in Huntsville module of the oxygen interaction with materials experiment (EOIM-2) which flew on Shuttle Flight STS-8 in September 1983.[1]

Approximately 95% of the total atom fluence of ~3.5 x 10^{20} oxygen atoms cm^{-2} was produced while surfaces were oriented within 1° of the orbital direction.

The surfaces studied were of two types: high-purity thin films sputtered or evaporated onto 2.54-cm diam λ/20 fused silica optical flats, and highly polished bulk samples. Films were prepared with optical densities of ~2.5 or less, as listed in Table II. Measurement of optical densities using a Perkin-Elmer PDS scanning microdensitometer allowed sensitive determination of any changes to these thin films produced by the exposure. By masking one-half of each sample during flight as a control it was possible to measure changes in

optical density of ~0.01, corresponding to a few percent change in thickness of ~10 nm thick films. (No increase in scattering was assumed, since most films became smoother; changes in reflection due to very small oxide thickness increases were also neglected.) In several cases the sensitivity of the method was ±1 monolayer of surface atoms. Total film thicknesses were accurately measured by step height changes in stylus traces of the film/substrate surfaces using a Tencor Alpha-Step 200 stylus profilometer with a nominal 2 μm radius diamond stylus. Decreases in both the optical density and total film thickness were attributed to metal film removal. A decrease in optical density but an increase in total film thickness was attributed to film expansion due to formation of a nonvolatile oxide of lower density than the original film. To obtain optimum resolution of step heights from stylus measurements, fine lines were scraped to the bare substrates, except for the niobium and nickel films, which could not be removed without damaging the substrate. The arithmetical average of the step plateaus (average absolute distance from a center line level for up to 2000 data points) gave indicated surface roughness of the order of 0.5 nm for the smoothest surfaces. Although some smoothing probably occurred from the stylus riding over microscopic defects and compressing the surface somewhat, changes of ±1 nm in relative film heights between exposed and unexposed surfaces were measurable when averaging was used on both levels of sharp steps. Topographic measurements were also made on a number of surfaces with an optical heterodyne instrument using recent techniques.[2] These measurements provided an optical determination of surface roughnesses for comparison to stylus results as well as several correlation functions from the same measurement.

Estimates of metallic film thicknesses in exposed and unexposed areas were calculated from the optical density measurements using equations for transmission and reflection.[3] Published optical constants for metals[3,4] were used

for calculations except for noted cases, such as for niobium, where a value of 28 nm thickness per unit optical density change had been accurately determined for our sputtered niobium films. Air, metal, and fused silica substrate media were assumed. This neglected the effect of very thin transparent oxides on absorption and reflection for normal incidence on these samples. Neglecting such effects on metals forming thick oxides, such as for silver, would introduce unacceptable errors, however.

Osmium and iridium films were purchased, the platinum overcoatings on the osmium were electron-beam evaporated, and most of the other films were sputtered.

Sensitive step height measurements on bulk samples such as Os, C, and optical plastics were obtained with the diamond stylus. Most of the bulk sample surfaces had 10 nm arithmetic roughness and were not very flat. In most cases this lack of precision for bulk sample measurements was unimportant since large effects were produced. Step height determination was enhanced on most bulk samples by sputtering a protective niobium film ~220 nm thick in a resolution pattern of small rectangles spaced over the whole sample surface before mounting for flight. Although both knife-edges and film masks were used, much sharper steps were obtained with films than with mask knife-edges, because intimate contact between substrate and film mask was assured, and effects due to shadowing of areas of the substrate next to taller mask structures did not occur. Accurate niobium mask thicknesses were obtained in the unexposed areas and subtracted from the step heights in the exposed areas. Typically, large negative steps were produced on combustible materials by oxygen exposure; however, in the case of silver, a large expansion occurred.

The polycarbonate (CR-39) and polymenthyl methacryalte (Lucite) bulk plastics were cut into disks and cleaned with ethanol. The other bulk materials in

Table III, except for the diamond sample, were ground and polished reasonably flat with progressively finer abrasives (aluminum oxide or diamond) to ~10 nm arithmetic roughness and cleaned with strong solvents followed with alcohol.

Results

Rapid etching of carbon and carbonaceous surfaces was observed with the polycarbonate CR-39 showing the largest etch (over 22 μm) of any substrate yet flown and measured. Of the metals, only osmium and silver showed large effects, the former being heavily etched and the latter forming a very thick layer of oxide. The first measurable (but small) effects on iridium. aluminum, nickel, tungsten and niobium thin films were reported. Except for iridium the change in optical properties was ascribed to a small increase in the thickness of the metal oxide layer which covers these metals after they have been exposed to the normal atmosphere. Iridium appears to have been very lightly etched and this effect will be studied further as other exposures become available. Full details of the observations are available in the Applied Optics paper and are not repeated here.

References

1. J. C. Gregory and P. N. Peters, "The Production of Glow Precursors by Oxidative Erosion of Spacecraft Surfaces," NASA CP-2391, Second Workshop on Spacecraft Glow (1985), pp. 174-179.

2. G. E. Sommargren, "Optical Heterodyne Profilometry," Appl. Opt. 20, 610 (1981).

3. G. Hass and L. Hadley, "Optical Properties of Metals," in <u>AIP Handbook</u>, D. E. Gray, Ed. (McGraw-Hill, New York, 1963), Sec. 69.

4. D. W. Lynch and W. R. Hunter, "Comments on the Optical Constants of Metals and an Introduction to the Data for Several Metals," in <u>Handbook of Optical Constants of Solids</u>, E. D. Palik, Ed. (Academic, Orlando, 1985).

5. P. N. Peters, private communication.