

CHARACTERIZATION OF HELIOSTAT CORROSION

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ABSTRACT

Corroded mirrors from a heliostat exposed at Livermore, CA for eight months have been characterized by a variety of techniques including optical and electron microscopy, reflectance and transmission spectroscopy, and surface-enhanced Raman spectroscopy. The results indicate that the chemical methods used in previous studies to remove the paint from the back surface of corroded mirrors significantly altered the morphology of the corroded film. A mechanical stripping technique has been developed which eliminates these problems. Use of this new technique, combined with studies of the reflective surface as viewed through the glass, have revealed that the corrosion spots are areas of agglomerated silver which are only loosely bonded to the glass superstrate. These agglomerates can be removed by wiping with paper tissue or by placing the sample in water or glacial acetic acid for a few seconds. These observations have led to the suggestion of a new model for the corrosion process.

1. INTRODUCTION

Current heliostat designs for large solar thermal central power systems use second-surface mirrors manufactured by conventional commercial silvering processes. Recent observations^{1,2} of heliostats exposed at Livermore, CA for less than eight months indicate that the appearance of the reflective surface of these heliostats has changed with time. Most of this deterioration appears as small dark spots. The formation of these spots has been linked to the presence of liquid water at the silver/glass interface of the heliostat module. This paper presents the results of a detailed study of this degradation.

2. EXPERIMENTAL

2.1. Materials

The corroded specimens studied were obtained from a heliostat at Sandia Laboratories Livermore (SLL). The mirror specimens were removed from the heliostat after seven months of outdoor exposure (February - September, 1978) at a heliostat test facility at SLL. Details regarding the origin and construction of this heliostat can be obtained from the authors.

Most of the work of the present study involved two particular regions of the heliostat described above. These regions are designated as CM1 and CM2 (the abbreviation CM represents "corroded mirror"). The corroded areas of these samples were readily observed when the sample was illuminated with intense collimated light and viewed at an angle other than the specular reflectance angle. Measurements also were made on an accelerated test sample (CM3) which had been exposed on the painted surface to water at 77°C for 625 hours. This specimen exhibited much more corrosion than did the two field-exposed samples, in that a dense, random, spotting effect was observed even under normal viewing conditions. Microscopic examination of this specimen revealed that the corrosion pattern observed was qualitatively similar to that of the field-exposed samples.

2.2. Paint Removal

The process used to remove the protective layer of paint from the mirror prior to study of the metal surface is shown here to have an extremely important effect on the results obtained. Earlier studies at PNL² and Sandia Laboratories used dimethyl sulfoxide or glacial acetic acid to remove the paint. In both cases, the sample was immersed in warm liquid for a few minutes until the paint layer appeared to shrink and drift away from the

mirror surface. The results presented here will demonstrate that this technique actually altered the results of the study. Subsequent studies at Sandia Laboratories have used a mechanical process to avoid chemical reactions between the stripping procedure and possible corrosion products. In this process, the paint side of the sample is bonded to a thin piece of styrofoam using 3M brand EC-3549 adhesive. The sample is then cut to size on a diamond saw and the edges ground to minimize fracturing of the glass during subsequent treatment. Next, the sample is dipped into liquid nitrogen until boiling of the nitrogen ceases. The paint is then removed while the sample is cold by inserting a scapel blade under the edge of the paint and lifting the paint away from the mirror surface. While this technique does not always results in a uniformly clean surface, it does, unlike chemical stripping, preserve the structure of the corrosion spots as observed in reflected light micrographs of unstripped corroded mirrors. It also allows analysis of the paint removed and a comparison of the morphology of the paint surface with that of the matching mirror surface.

2.3. Heat Treatments

A number of samples were heat treated in air. Before treatment, paint was removed with glacial acetic acid. For selected samples, the copper also was removed with ammonium persulfate. The samples were placed in a vitreous silica tube open to the atmosphere, and the tube was inserted into a pre-heated furnace. The samples reached the desired temperature (150-450°C) within five minutes and were held at temperature within $\pm 5^\circ\text{C}$ for one hour. The samples were then removed from the furnace and allowed to cool to room temperature inside the vitreous silica tube.

2.4. Microscopy

Optical micrographs were made with a conventional microscope using transmitted and/or reflected light separately or simultaneously. The type of illumination was determined by the requirements of the specimen, e.g., a specimen which had not been stripped of paint could be viewed only in reflected light. A xenon light was used for all reflected light micrographs.

A number of specimens were also studied with a scanning electron microscope. Although some of these specimens required carbon coating to minimize electrical charging effects under the electron beam, many of the specimens were studied in the as-stripped state.

2.5. Reflectance and Transmittance Measurements

The spectral dependence of the specular reflectance of corroded and uncorroded mirrors was measured from 0.35 to 2.50 micrometers using a V-W technique which has been described in detail elsewhere.³ Transmission spectra were measured with a Cary 17I spectrometer.³ No attempt was made to remove the paint and other backing material (adhesive, styrofoam) prior to the first reflectance measurements. After the initial measurements, the backing removed, and the paint was stripped from the uncorroded mirror, CM1, and CM2 with glacial acetic acid. The uncorroded mirror was also treated with ammonium persulfate to remove the copper. The spectral reflectance of these three samples was then remeasured and found to be identical with the initial measurements.

2.6. Raman Measurements

Raman spectra were obtained with a conventional Raman backscattering system consisting of a cw argon laser, collection and sample illumination optics, double grating monochromator, photon counting electronics, and

computer-controlled scanning and data acquisition system. Measurements on intact, corroded mirrors were performed by illuminating and collecting scattered light from the glass side in a right angle scattering geometry. Mirrors stripped of paint and copper and heat-treated (Section 2.3.) were examined from the rear (metal layer) side. A 488.0 nm laser wavelength was used to excite the spectra reported here. A 514.5 nm line was occasionally used to distinguish Raman scattering from fluorescence. The incident laser power was focused with a cylindrical lens and kept below ~ 100 mW to minimize sample heating.

3. RESULTS

3.1. Microscopy

3.1.1. Corroded Mirrors

Most of the early work on the corrosion of heliostat mirrors utilized chemical methods to remove the paint from the mirror surface so that the metallic film could be viewed from the copper side. A few relatively low magnification ($\leq 150X$) micrographs taken through the glass superstrate were reported by Burolla and Roche¹ on Livermore heliostats. Similar micrographs were reported by Lind et al.² for severely corroded mirrors which had been stored for 17 years outdoors at Carolina Mirror Corp. These early attempts to view the silver-glass interface through the glass revealed that the corroded spots consisted of somewhat concentric rings surrounding a darker nucleus. Similar micrographs were obtained in the present study at much higher magnification (600X) by grinding and polishing the glass to a thickness of less than 0.5 mm so that a higher magnification lens could be used to view the sample. The results, shown in Figure 1A, indicate that the apparent ring structure is less clearly defined than was indicated by the lower magnification micrographs. The darker central portion of the corrosion spot appears to be very similar to that observed in earlier studies.

Mechanical stripping of the paint from the sample with liquid nitrogen (LN) has essentially no effect on the morphology of the corrosion areas, as is shown in Figure 1B. This result suggests that samples prepared by this technique should be much more representative of the actual condition of the undisturbed corroded mirror than those stripped of paint by other techniques. For comparison, the results of chemical stripping with glacial acetic acid is shown in Figure 1C. The removal of the corrosion product from the holes is

evident in Figure 1C. A similar effect was noted for samples stripped of paint with dimethyl sulfoxide, and for a mechanically stripped mirror which was rinsed in running water for a few seconds and then wiped dry with a paper tissue. Further study revealed that a solvent is not needed to remove the corrosion product lying within the holes in the metallic film. This material can be removed easily by wiping the sample with a dry paper tissue, as shown in Figure 1D. The reader should note that the area shown in Figure 1D is identical to that shown in Figure 1B.

The appearance of the mirror when viewed from the silver side is also a strong function of the stripping technique used (Figure 2). The left portion of the micrograph in Figure 2 represents a region of the specimen which was stripped by the liquid nitrogen technique. The holes through the metal film were filled with material when viewed immediately after stripping. The entire specimen was then placed in warm glacial acetic acid for 60 seconds to remove the paint from the remainder of the specimen. Such an area is shown in the right portion of this micrograph. Note that the holes in the film in the area which had been mechanically stripped still contain a residue of material, whereas those in the area stripped chemically are quite clean. The light areas surrounding the holes in both regions of the sample are bare silver, whereas the darker matrix represents areas which are covered with copper. The physical appearance of the matrix, however, is also quite different in the two areas of the sample. Close examination of the mechanically stripped area reveals that many small particles of paint still cling to the metal film in this region of the specimen.

Scanning electron micrographs of the chemically and mechanically stripped mirrors emphasize the differences discussed above. Furthermore, EDX spectra confirm the identification of the various areas observed in the optical

micrographs. These spectra indicate that the holes observed for chemically stripped mirrors contain only glass (Figure 3, point D), that the ring surrounding the hole is bare silver over glass (Figure 3, point B), and that the matrix represents a normal area of the mirror with films of both silver and copper over the glass (point C). The high magnification of this micrograph relative to that of the optical micrographs, however, reveals that there is a small (1-2) micrometer) area of material along the edge of the hole which could not be detected in the optical micrographs. EDX analysis (point A) reveals that this material is very rich in silver, with no indication of the presence of any other elements except those found in the underlying glass. While this type of features is not found near all of the holes through the metal film, such features are by no means unusual.

Some features observed in the SEM micrographs of the mechanically stripped mirrors are identical to those observed for the chemically stripped samples. The area surrounding the holes appear to be a smooth film of silver over glass (point B in Figure 4). The areas within the corrosion spots which are not covered with particles (point C) appear to be primarily glass with a very small amount of both silver and copper. The particles themselves (point D) appear to be strongly enriched in silver, suggesting the formation of silver particles thicker than the sampling depth ($\sim 1\mu\text{m}$) of the electron beam. There is no indication of other foreign elements such as sulfur or chlorine which might indicate the presence of silver compounds. (It should be noted that EDX will not detect elements below sodium in atomic number, so that the presence of carbon, oxygen, nitrogen, and fluorine cannot be eliminated by these results). The matrix area surrounding the corrosion spots is covered with paint, as is indicated by the EDX spectrum for point A in this figure.

The identification of the material lying within the corroded holes in Figure 4 has been confirmed by transmission electron microscopy and electron diffraction of material removed during replication of this surface. The diffraction pattern clearly indicated that most of this material is metallic silver. There were also traces of another material in the diffraction patterns which may be a silver halogen (either chloride or bromide) compound. The intensity of the diffraction pattern for the minor phase decreased with time. Further work is underway to identify this material and to ascertain its source.

3.1.2. Thermal Treatments

Thermal treatment of uncorroded mirrors yields some features which bear a remarkable resemblance to that of corroded mirrors. The micrographs shown in Figure 5 exhibit structures with a morphology very similar to that shown by the corroded mirrors. (A direct comparison at identical magnification of a corroded specimen with a heat treated specimen is shown in Figure 6). The number density, size, and distribution of the light areas (which are bright blue when viewed in transmitted light) in the micrographs of the heat-treated specimens are all quite similar to those of the holes through the metal film in the corroded specimens. Heat treatment of an uncorroded specimen at 275°C or above, in fact, results in the formation of what appear to be holes in the metallic film. These holes appear to result from the disappearance of the blue regions observed in specimens heat-treated at lower temperatures. Examination of these holes at 1000X indicates that the "holes" are actually filled with many small particles (approximately 1 micrometer in diameter). The morphology of the material within these areas is very similar to that observed when silver films without a copper overcoat are given a similar heat

treatment. Study of these films with the scanning electron microscope indicates that the particles are essentially pure silver. Although the specimens which were heat-treated with both the silver and copper films have not as yet been examined by SEM, it does not appear unreasonable to suggest that the small particles formed in these specimens are very similar to those obtained for the specimens which were covered only with a silver film.

3.2. Reflectance and Transmittance Measurements

3.2.1. Specular Reflectance

Measurements were made to determine the change in reflectivity of the silver-glass interface upon outdoor exposure (CM1 and CM2) and upon accelerated aging (CM3), and to relate the observed changes in optical properties to structural features characteristic of the corrosion process.

The reflectivity of the silver-glass interface (ρ_{sg}) was determined from the reflectance of the mirror (ρ_m), and transmittance (τ_g) and reflectance (ρ_g) of the glass superstrate according to the following relation:

$$\rho_{sg} = \frac{\rho_m - \rho_g}{[(1 - \rho_g)^2 + \rho_g(\rho_m - \rho_g)]X^2} \quad (1)$$

where X is the internal transmittance of the glass, which, for $X=1$, is given by $X = \tau_g + \rho_g$. Measured values of ρ_m and τ_g are given in Figure 7a and 7b, respectively. (The slight increase in transmittance in the outdoor exposed samples is probably due to the recent observed solar-induced decrease in Fe^{2+} absorption³. Based on earlier work⁴, a wavelength independent value of $\rho_g = 0.074$ was used. was used. The resulting values for the reflectivities of the silver-glass interfaces are given in Figure 7c. Difference spectra, obtained by subtracting the reflectance of the corroded interface from that of the uncorroded interface,

are shown in Figure 7d. The difference spectra for all three corroded mirrors are qualitatively similar, i.e., no sharp spectral features are observed, the degradation has a shallow minimum between 0.8-1.0 μm , and the degradation increases for wavelengths less than 0.7 μm .

The degradation for wavelengths greater than 1.2 μm appears readily explicable in terms of corrosion spots consisting of holes containing agglomerated silver. Such features would give rise to a wavelength-independent decrease in reflectance equal to the fractional hole area plus a slight increase above this value (and possibly a slight spectral dependence) due to scattering by the agglomerated silver. This is consistent with the observed decrease in infra-red reflectance of CM1 of 0.062 vs. the measured fractional area of holes for this sample of 0.076.

However, such a model will not explain the significant increase in degradation for wavelengths less than 0.7 μm . Some possible explanation for this behavior invoke a spectral dependence introduced by the thinning of copper, absorption by the agglomerated material, reflectance of the paint showing through the holes, and the presence of additional corrosion features. Additional measurements tend to argue against the first three explanations and support the latter. Removal of the backing paint and copper overlayer did not alter the reflectance of the uncorroded mirror. Removal of the backing paint and silver agglomerates did not alter the reflectance of corroded mirrors CM1 and CM2. The reflectance of the paint was found to be <0.01 over the entire wavelength range. However, reflected light micrographs taken through the glass (see Figure 1) did reveal another corrosion feature, i.e., corrosion holes were often encircled by a ring of material having low reflectivity than the rest of the silver film. The contrast between the ring and the rest of the silver is enhanced in violet light and muted in red light, consistent with

the spectral dependence observed in Figure 7. It follows that this new feature may explain the increase in degradation at shorter wavelengths. It is not currently known whether the change in optical properties of the ring results from a change in the morphology or composition of the parent film.

3.2.2. Transmittance

The proposed model relating structural and spectral features can also be tested by measuring the transmittance through the mirror after removal of the backing paint. Figure 8 shows the results of such measurements on an outdoor exposed mirror sample (CM1b) obtained from an area adjacent to CM1 of the preceding section. The transmittance of the sample was measured immediately after LN stripping ("filled" holes) and after rinsing the LN stripped sample in water and wiping dry (empty holes). The corresponding microstructure for each of these transmittance measurements is shown in Figure 9. Since the spectrophotometer has a limited acceptance angle, diffraction of light by the small corrosion features will also give rise to "transmittance loss", and hence will complicate interpretation of these spectra.

However, allowing for these diffraction effects, certain conclusions can be drawn from the data of Figure 8: (a) removal of the agglomerated material significantly increases the transmittance of the sample; (b) the transmittance of the rinsed sample is in reasonable agreement with the measured fractional hole area and reflectance for wavelengths less than $0.7 \mu\text{m}$ appears insufficient to explain the corresponding decrease in reflectance, suggesting that this portion of the reflectance decrease is associated with absorptive losses; and (d) there are no sharp spectral features associated with the agglomerated silver over the wavelength region $0.35\text{-}2.5 \mu\text{m}$.

3.3. Raman Spectra

Heat treatments of stripped mirrors after removal of the copper overcoatings consisted of 30-minute exposures to wet or dry air at 300°C. In each case, the reflective silver layer was transformed into a milky, semi-transparent layer. A representative Raman spectrum from a heat-treated film is shown in Figure 10. Peaks associated with scattering by the film are indicated with arrows, while the peaks at lower Raman shifts (near 600, 800, and 1100 cm^{-1}) arise from scattering by the glass substrate. A broad, relatively featureless background intensity is also present, with a maximum intensity near 2000 cm^{-1} . (The rising intensity below 400 cm^{-1} is due to elastic scattering.) The background arises partly from fluorescence originating in the glass substrate and, possibly, in the metal film.

The arrow-labeled features in the heat-treated spectrum are similar to features reported in the literature dealing with "enhanced" Raman scattering from roughened silver interfaces.^{5,6} These features are not a result of scattering by monolayers of molecules absorbed on the silver surfaces. Although molecular monolayers are not normally detectable in spontaneous Raman spectra, such "surface enhanced" signals are believed to result from electronic interactions between the absorbates and the silver substrate. The degree of enhancement, and hence the observed signal strengths, has been reported to depend crucially on the degree of surface "roughness" in the silver substrate. Exceptionally smooth, evaporated films were found to exhibit no observable surface enhanced signals; hence the effect is considered to be extremely sensitive to this, as yet uncharacterized, aspect of the silver morphology.

The Raman results are interpreted as indicating the presence of surface-roughened metallic silver in the heat treated mirrors. This identification is supported by the observation of identical signals (peaks at 1350, 1600, and

2930 cm^{-1}) from surfaces of bulk silver roughened by mechanical abrasion. The results of optical and electron microscopy (Section 3.1.2.), together with the Raman data, strongly suggest that the heat treatment produced a textured metallic silver matrix containing voids inhabited by agglomerated silver particles. Whether or not the surface enhanced scattering is associated with the particulate or the textured silver is under investigation.

Raman spectra from a heliostat exhibiting large ($\sim 1\mu\text{m}$) dark corrosion spots are also shown in Figure 10. The lower spectrum was obtained from an uncorroded region of the reflective silver layer. The peaks labeled "glass" and the smooth background intensity result from Raman scattering and fluorescence in the glass, respectively (the laser was incident from the glass side of the unstripped mirror). The lack of surface enhanced scattering is attributed to the relative smoothness of the uncorroded silver surface.

Examination of a corroded region revealed additional peaks similar to those observed in the heat-treated mirror (see the middle curve in Figure 10). An added background intensity was also observed which is believed to result partly from fluorescence by the protective paint backing. This result is consistent with the previous observation (3.1.1.) that light is transmitted through the silver and copper layers in severely corroded regions. The appearance of prominent surface enhanced features further suggest that metallic silver is also present within the degraded region. Spectra taken with the laser focused to a point at different locations in the region show increased surface enhanced intensities in the outer, rather than central areas. These results are consistent with particulate or roughened metallic silver existing within the corroded area, and possibly less abundant in the core region. The similarity of the corroded spot spectra with the spectra from heat-treated mirrors suggests a similarity in silver morphology, i.e., agglomerated or

textured metallic silver which is only loosely bonded to the glass substrate. It should be noted that the presence of corrosion products other than the silver absorbate species could not be confirmed. Further work will be undertaken to identify the absorbate species and to characterize the dependence of the surface enhanced signals on silver morphology.

4. DISCUSSION

The results of the present study suggest a new model is needed for the corrosion of mirrors in the environment usually encountered by heliostats. The use of chemical methods to remove the paint and other backing materials in earlier work altered the morphology of the corroded mirrors, and led to corrosion models involving either electrochemical corrosion or corrosion resulting from interactions between the metal film and the glass superstrate. These models placed considerable emphasis on the absence of corrosion products within the corroded films. However, the use of microscopic and spectroscopic techniques to study mirrors without stripping of the backing, combined with the discovery of a mechanical stripping technique which does not alter the corrosion morphology, has demonstrated conclusively that the corroded areas are, in fact, filled with a significant amount of material. The results of EDX and Raman analysis indicate that this material is essentially pure silver. Optical and scanning electron microscopy have shown that this material is in the form of small agglomerates (1-2 micrometers or less) which are similar to those obtained by heating silver films in air. The area surrounding these holes appears to be delaminated silver containing a significant density of small holes similar in size to the agglomerated particles. Although the cause of this agglomeration cannot be explained fully at this time, any corrosion model offered in the future must explain this phenomenon.

Results of this study also suggest that there is a strong relationship between the morphology of the degraded mirrors and that of as-manufactured mirrors. Close examination of the as-manufactured mirrors by transmitted light microscopy reveals a mottled appearance, suggesting that copper coating over the silver is quite thin in some places. Heat treatment ($\leq 250^{\circ}\text{C}$ in air) emphasizes this effect and reveals the similarity of this pre-existing

morphology to that observed in corroded mirrors. Heat treatment at higher temperatures (275°C) in air results in the formation of holes which are filled with agglomerated silver. This result strongly suggests that the process observed during heat treatment is related to that occurring during corrosion in this field.

Although a detailed model cannot be proposed at this time, a scenario now can be suggested for the mirror corrosion process. It appears that the copper is removed first from localized regions of the metallic film. The observations of (apparently) thin copper regions on the as-manufactured mirrors suggests that these areas may act as sites for the initiation of the corrosion process. Since copper is known to be soluble in water, this process can be explained if liquid water reaches the metal surface. The silver in the areas exposed by the removal of the copper appears to delaminate from the glass and form agglomerates of no more than a few micrometers in diameter. The appearance of these agglomerates is not unlike that observed in heat treated silver films (provided the films are heated in air). Formation of these agglomerates reduces the specularity of the reflection surface. Although the mechanism leading to the formation of the agglomerates is not fully understood at present, the similarity between this effect and that produced by moderate thermal treatments suggests a common cause, such as surface enhanced diffusion of silver.

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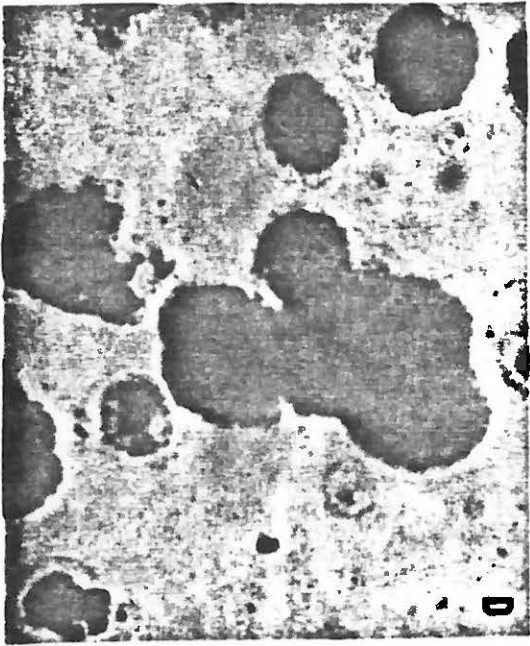
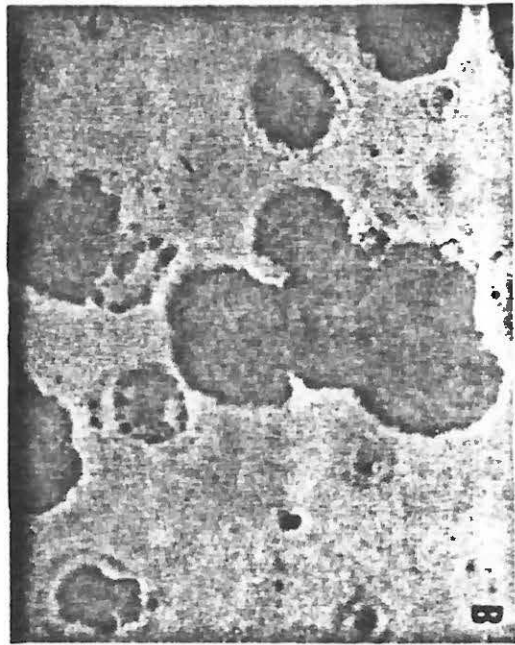
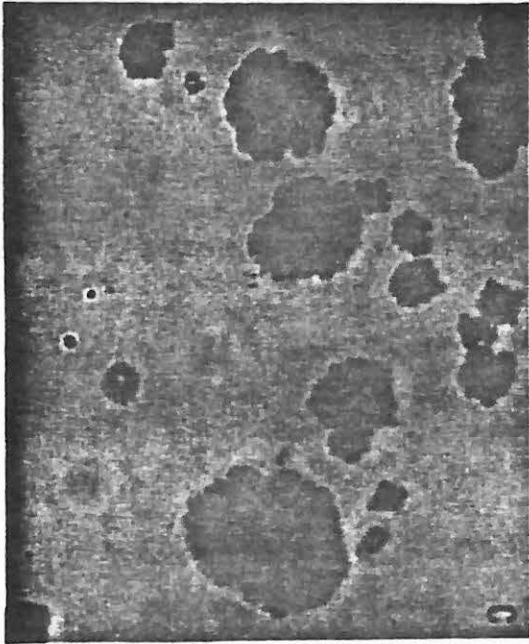
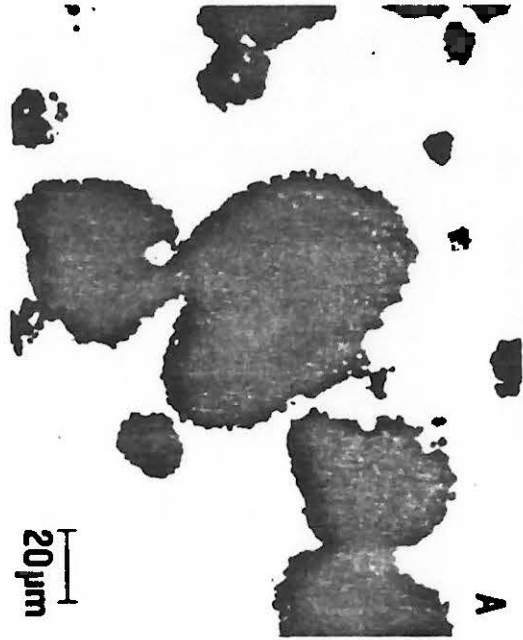
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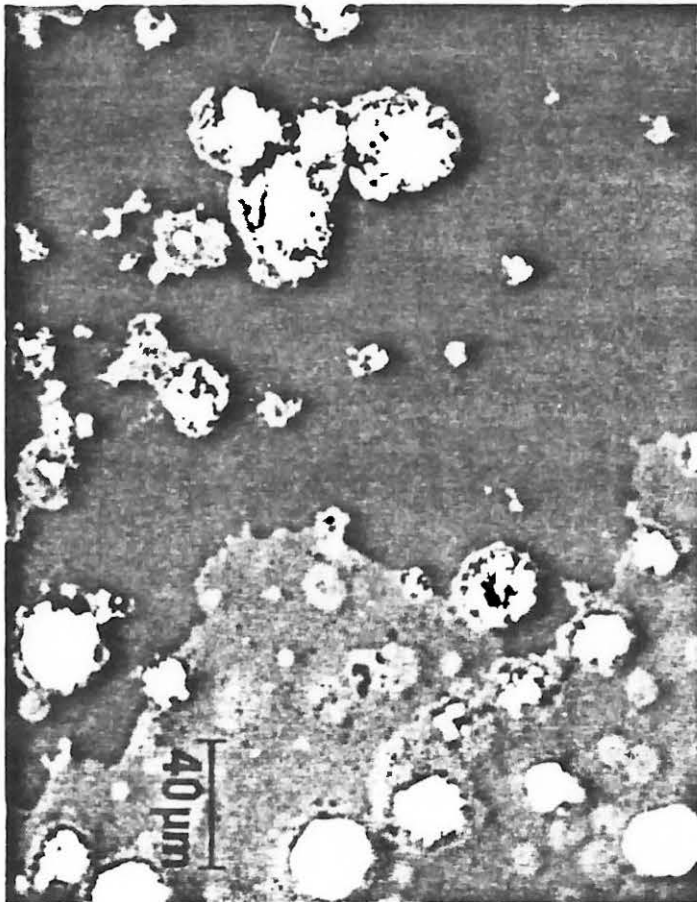
FIGURE CAPTIONS

- Figure 1 Optical micrographs taken by reflected light of a corroded mirror as viewed through the glass. Specimen conditions are (A) untreated, (b) mechanically stripped, (C) chemically stripped, and (D) mechanically stripped and wiped with a paper tissue. Note that B and D are the identical area of the specimen. All micrographs are at the same scale, as indicated in A.
- Figure 2 Optical micrograph taken using both reflected and transmitted light of a corroded mirror as viewed from the film side. The paint was mechanically stripped from the left portion of the sample and chemically stripped from the right portion of the sample.
- Figure 3 Scanning electron micrograph of a chemically stripped corroded mirror. EDX spectra for the points indicated on the micrograph are shown below the micrograph.
- Figure 4 Scanning electron micrographs of a mechanically stripped corroded mirror. The micrograph on the right is an enlargement of the central area of that on the left. EDX spectra for the indicated points are given below the micrographs.
- Figure 5 Effect of thermal treatment on the morphology of uncorroded mirrors heated in air for one hour at the indicated temperature. Micrographs were taken with transmitted light.

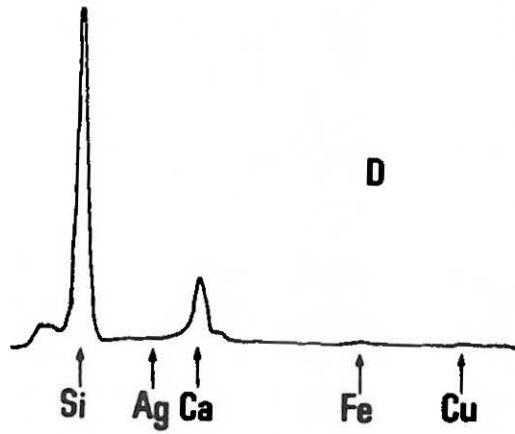
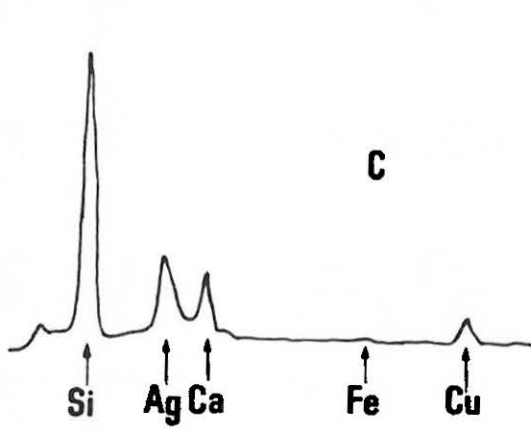
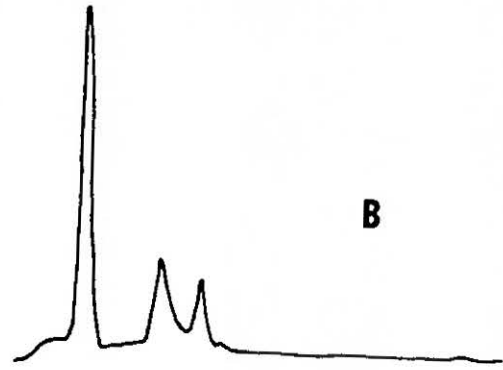
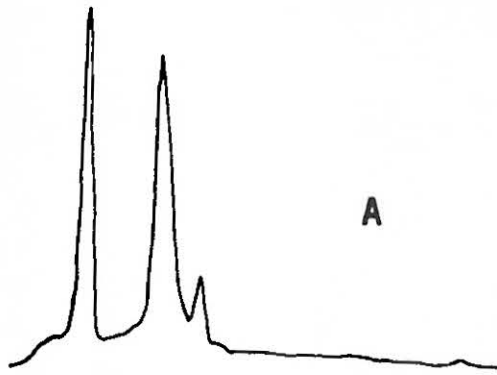
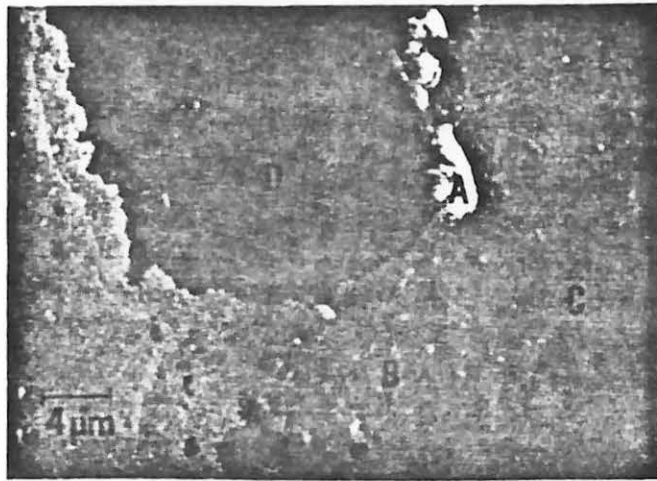
- Figure 6 A comparison of the morphology of an (A) corroded mirror and (B) a heat treated uncorroded mirror.
- Figure 7 Optical properties of uncorroded (solid lines), outdoor exposed (CM1 and CM2, dashed and chain-dashed lines, respectively) and accelerated aged (CM3, dotted lines) mirrors. (A) Specular reflectance of the mirrors. (B) Transmittance spectra of the glass superstrates. (C) Specular reflectance of the silver-glass interface. (D) Difference spectra, obtained by subtracting the reflectance of the corroded interface from that of the uncorroded interface.
- Figure 8 Transmittance spectra of a corroded mirror (CM1b) immediately after mechanical stripping (curve A) and after rinsing the stripped sample in running water and wiping dry with a paper tissue (curve B).
- Figure 9 Micrographs of the samples used to obtain the spectra shown in Fig. 8.
- Figure 10 Raman spectra of heliostats. The heat treated spectrum was obtained from a stripped mirror after removal of the copper layer and subsequent exposure to wet air at 300°C (the index mark at left is the zero level). The two lower curves were obtained from an intact mirror exhibiting corrosion spots, and represent spectra from corroded and uncorroded areas on the reflective silver surface. The vertical scale and zero level for these two curves are the same, but unrelated to the top curve.

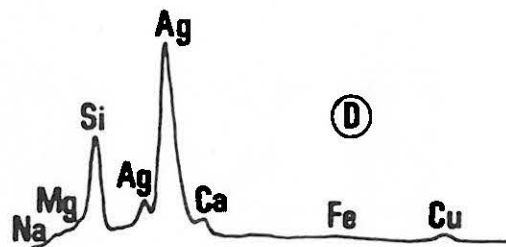
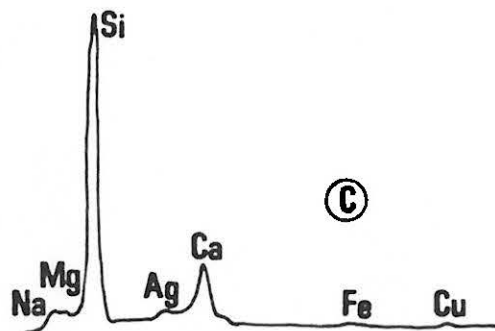
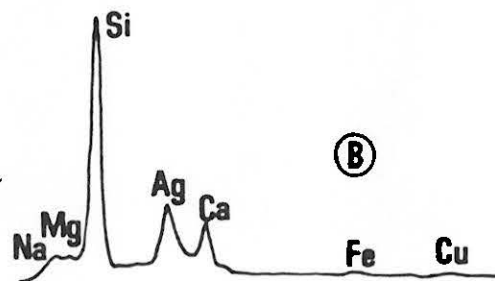
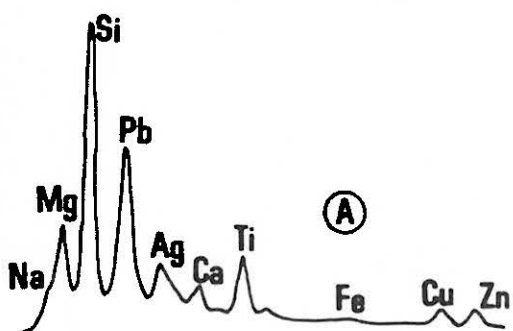
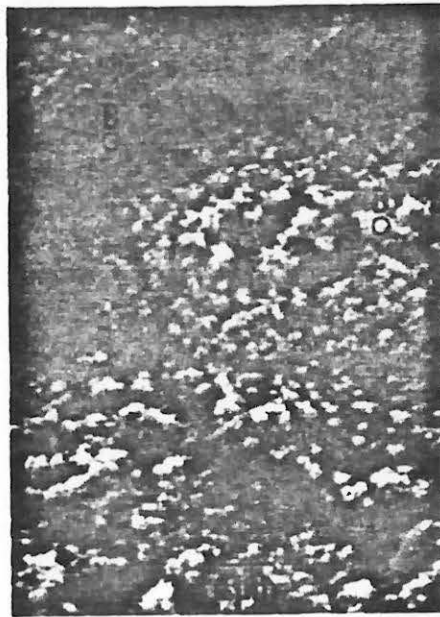
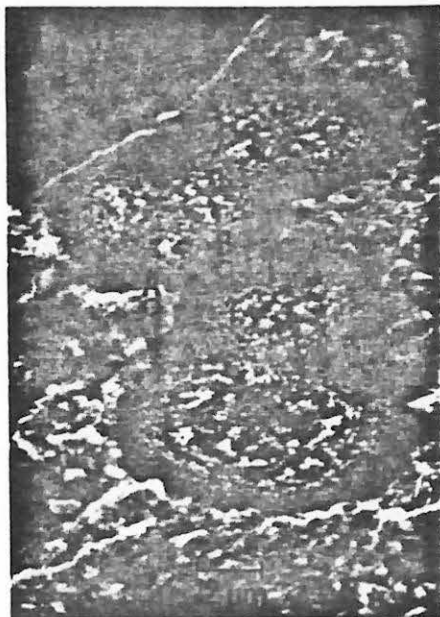


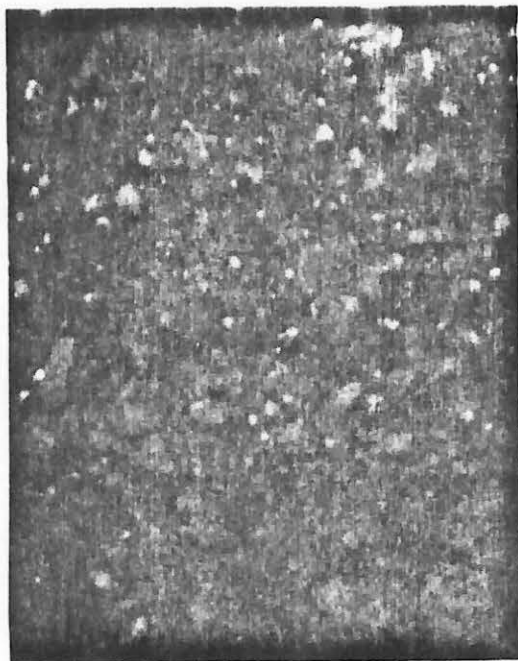
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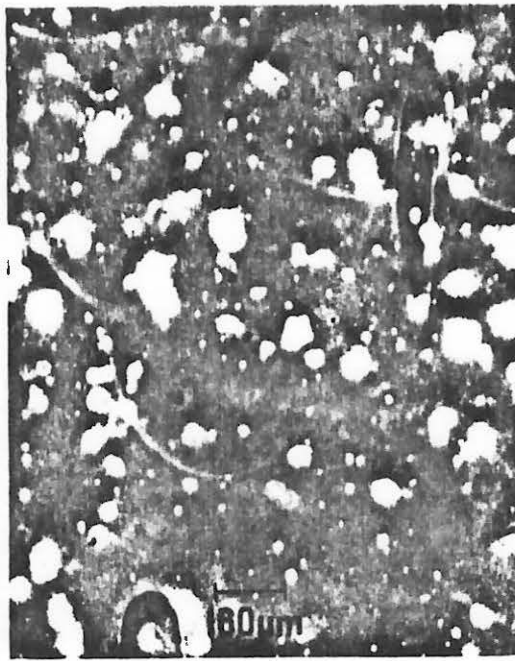






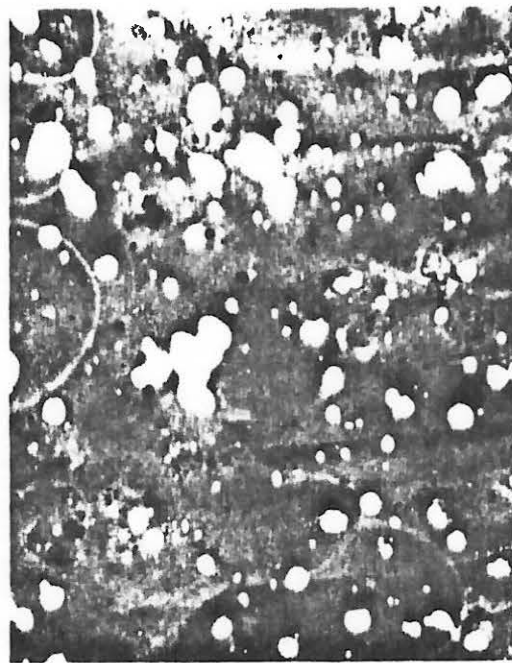
150°C

A



250°C

B



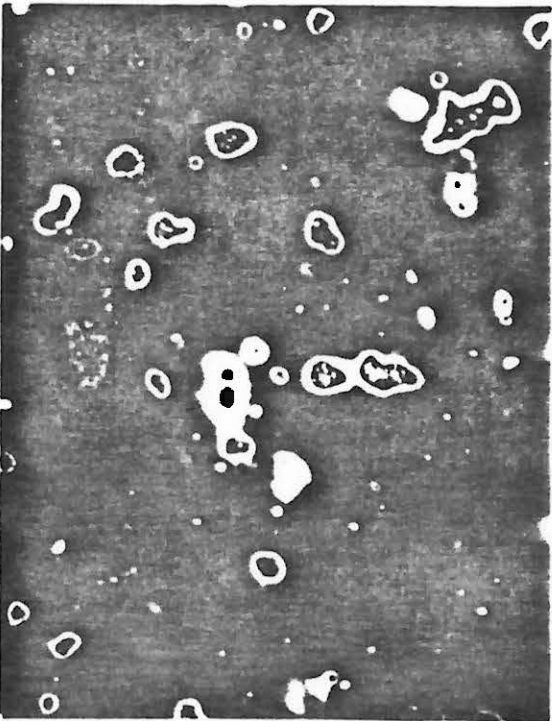
275°C

C

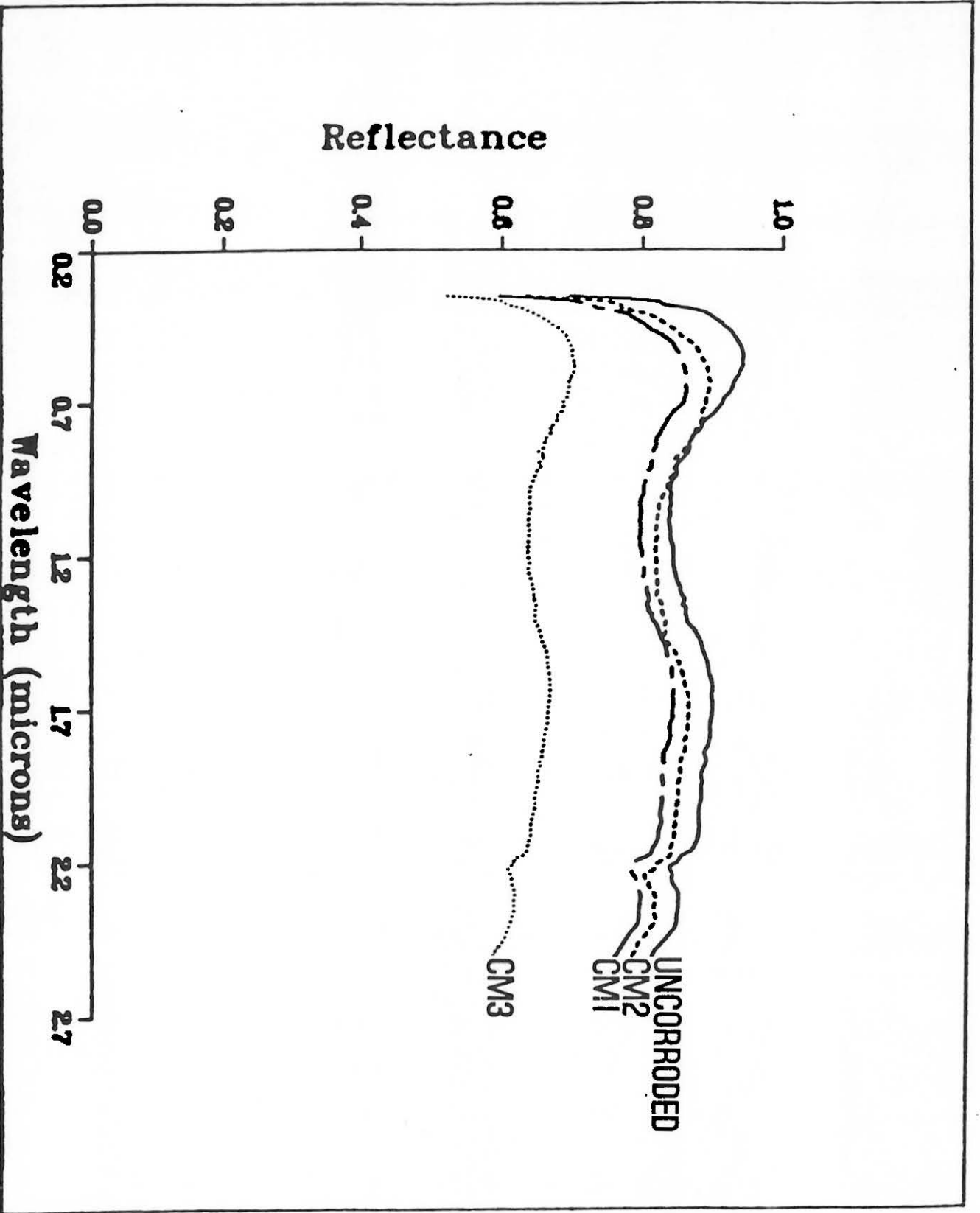


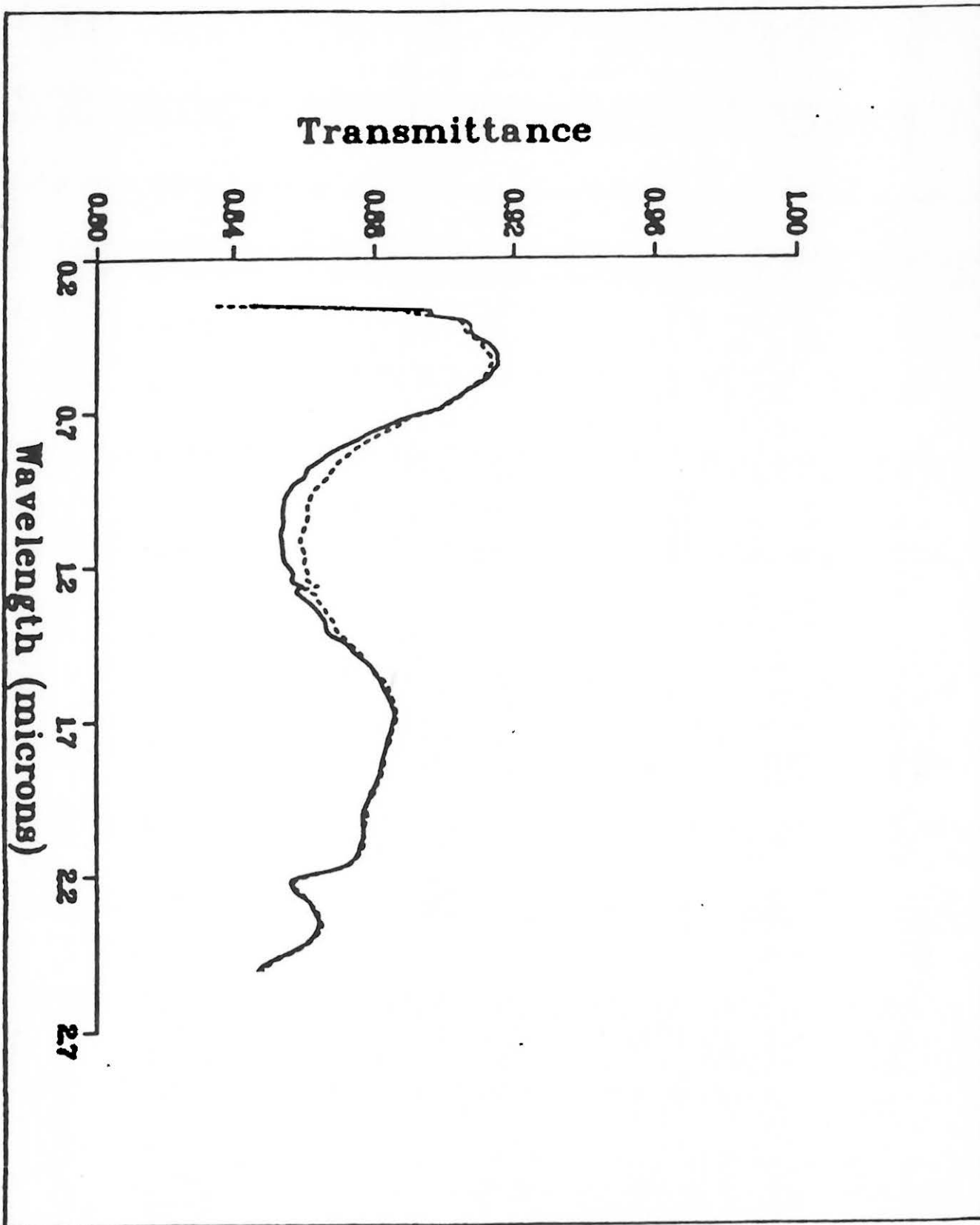
A. CORRODED

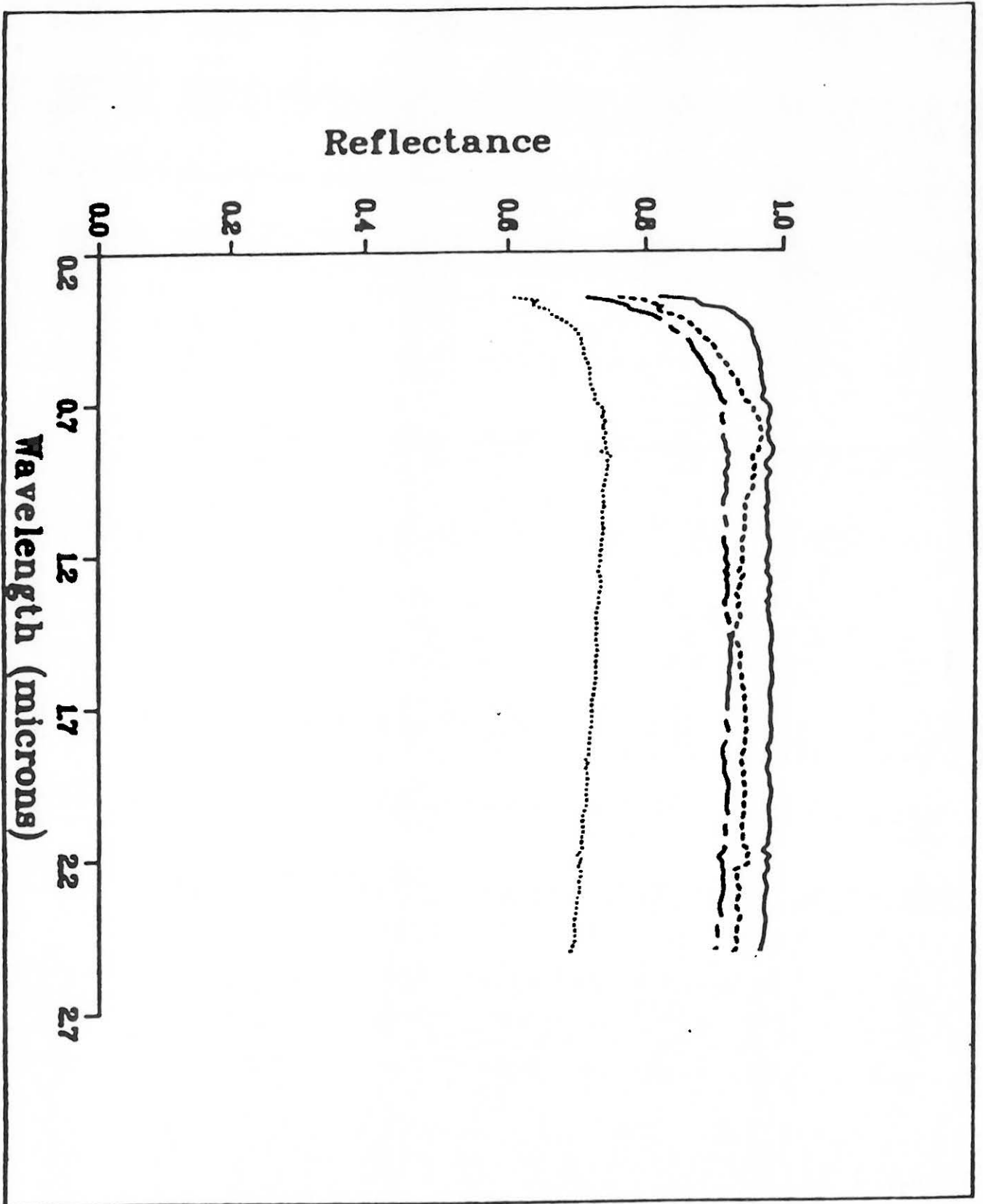
100µm



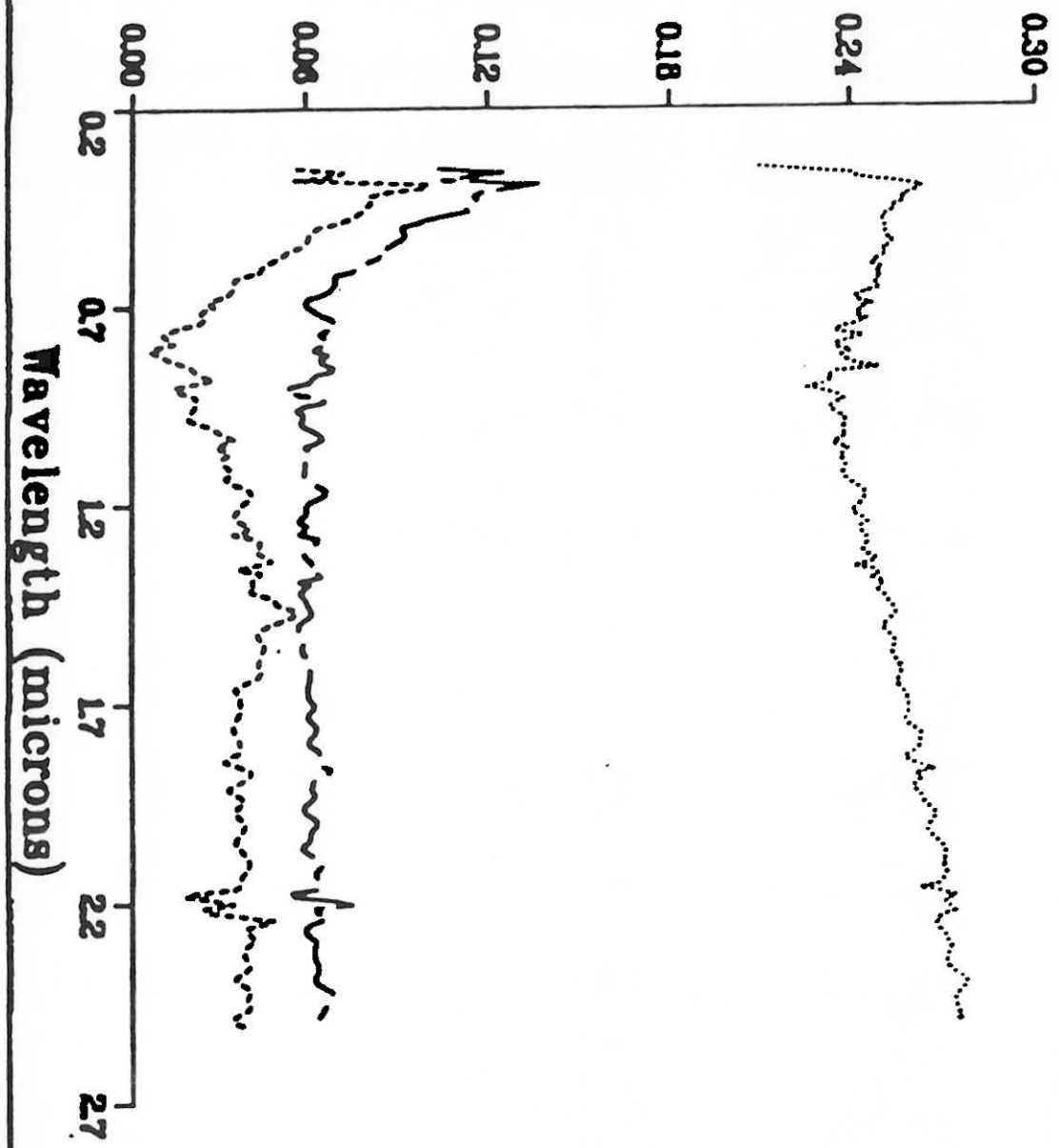
B. HEAT TREATED -- 275 °C

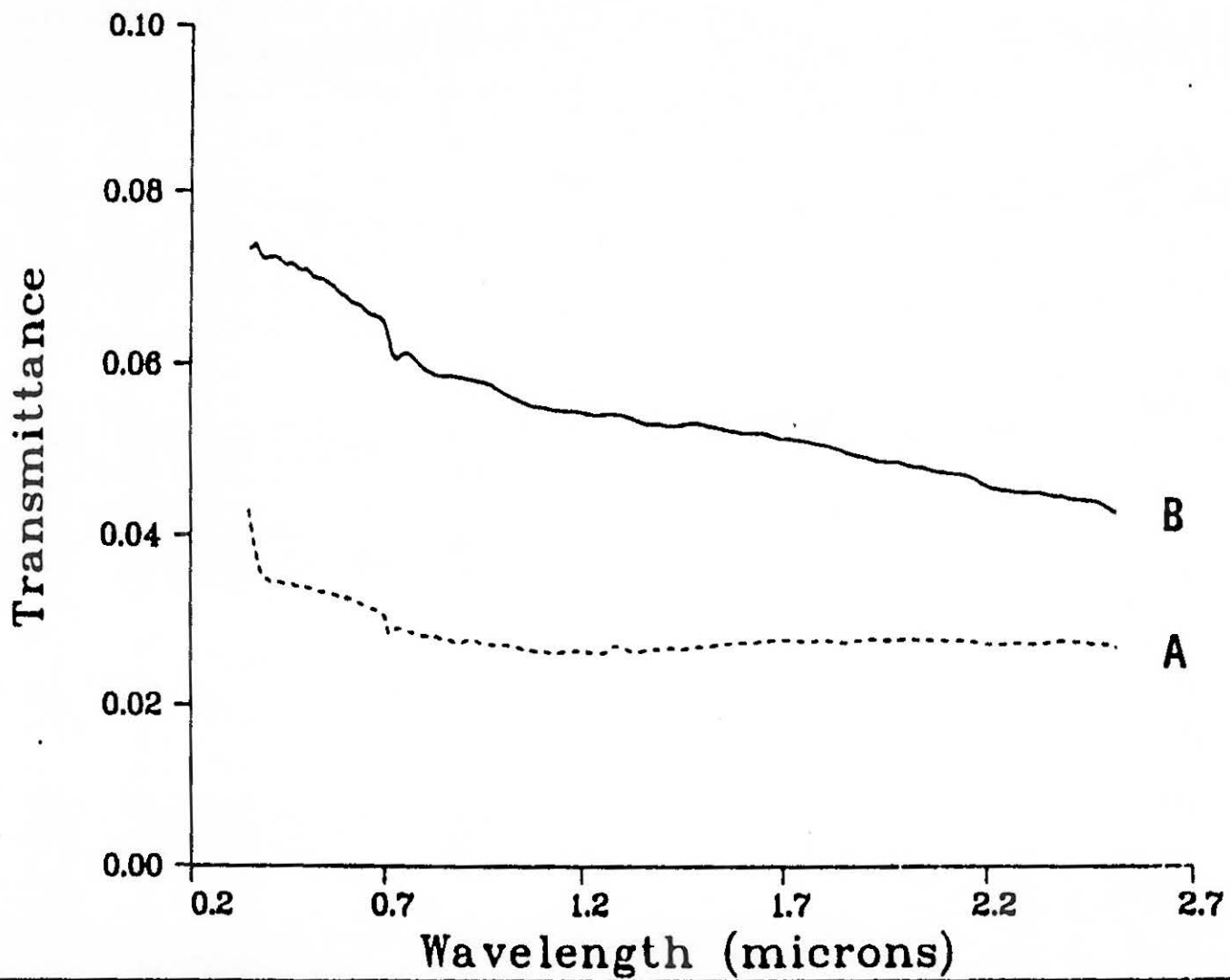


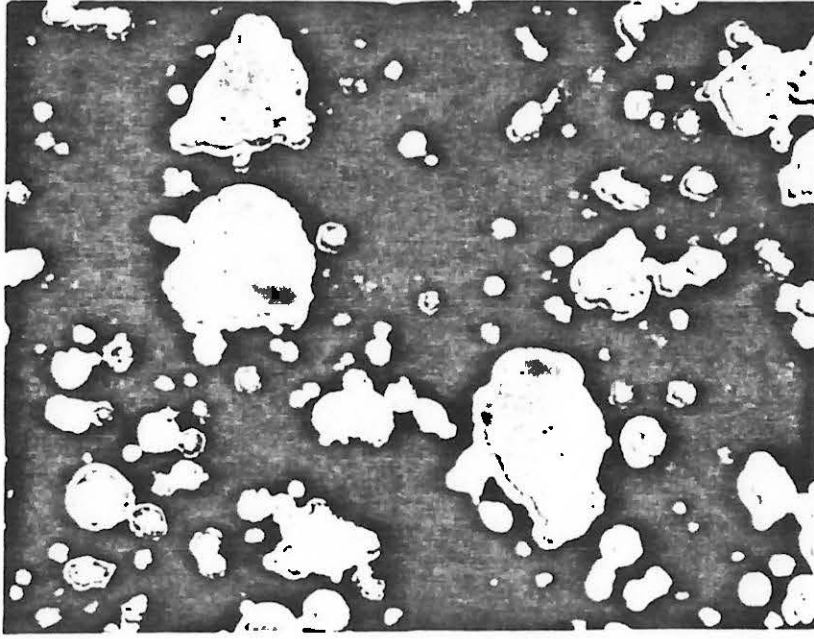




Difference Spectrum

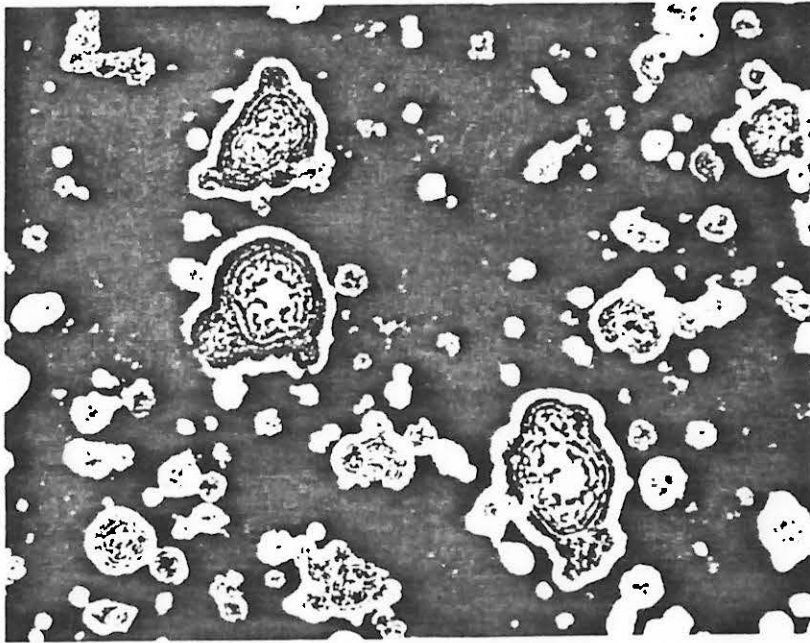






B. WATER RINSED

80µm



A. LN STRIPPED

INTENSITY (arbitrary units)

