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# DURABILITY OF REFLECTOR MATERIALS IN THE SPACE ENVIRONMENT

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## ABSTRACT

Various reflector configurations were flown as part of the Long Duration Exposure Facility (LDEF) A0171 experiment. These reflectors consisted of nickel substrates with aluminum, enhanced aluminum (multiple layers of aluminum and silver), silver, and silver alloy coatings with glassy ceramic overcoatings. These samples have been evaluated for changes in reflectance due to 5.8 years in the space environment. The reflector materials have also been evaluated using angstrometer, Rutherford backscattering (RBS), and electron spectroscopy for chemical analysis (ESCA) techniques.

## INTRODUCTION

Multi-layered reflector materials, such as the samples flown on the LDEF satellite, have been tailored for highly efficient reflectance in the near-ultraviolet through visible and near-infrared wavelengths (approximately 350 nm - 1400 nm). Spacecraft applications include solar concentrators and energy dispersion systems where reflection of solar radiation is required. Analysis of the protective glassy ceramic layers utilized on these samples may have transferrable applications in other areas, such as solar cell covers.

## EXPERIMENT DESCRIPTION

The Solar Array Materials Passive LDEF Experiment (SAMPLE), A0171, was exposed to the low Earth orbit space environment on the LDEF satellite. The duration of this exposure to atomic oxygen, ultraviolet radiation, thermal cycling, meteoroid/debris impacts, and particulate radiation was approximately 69 months. The experiment was positioned 38 degrees off the RAM direction at its location on Row 8 position A. The exposure conditions are given in Table 1.

The SAMPLE experiment tray is shown in Figure 1. Solar reflector materials were included in the 300 flight specimen complement. Approximately half of the reflector samples received full surface exposure while the remainder were configured with a mask that covered half the surface area. These half-covered samples are hereafter referred to as half-moon samples. These samples flew on Plate IV of the A0171 experiment and are shown in Figure 2.

Nickel substrates were coated with either silver or aluminum then a glassy ceramic protective layer. The samples exposed on LDEF were:

- Silver coated with $SiO_2$
- Silver coated with $MgF_2$ and sapphire
- Silver alloy coated with a multi-layer dielectric of $MgF_2$ and $Al_2O_3$
- Aluminum coated with $SiO_2$
- Aluminum coated with SiO
- Enhanced aluminum coated with SiO and $SiO_2$
- Enhanced aluminum coated with $MgF_2$ and sapphire

These flight samples and the control samples which were stored in the laboratory for the duration of the mission have been evaluated for changes in visual and microscopic appearance, optical properties, surface roughness, and layer thickness. Changes in the film morphology were determined by precision angstrometer traces, ellipsometry measurements, and Rutherford backscattering analysis. ESCA analysis was also performed to determine composition and extent of contamination, if any.

Table 1. Experiment A0171 Exposure Conditions

| | |
|---|---|
| High Vacuum | - $10^{-5}$ - $10^{-7}$ Torr (Estimated) |
| UV Radiation | - 10,041 ESH |
| Proton Fluence | - $10^9$ p+/cm$^2$ (0.05 - 200 MeV) |
| Electron Fluence | - ($10^{18}$ - $10^{12}$) e-/cm$^2$ (0.05 - 3.0 MeV) |
| Atomic Oxygen | - 7.15 x $10^{21}$ atoms/cm$^2$ |
| Micrometeoroid/ Space Debris | - 2 to 5 impacts per 25 cm$^2$, < 1mm |
| Thermal Cycles | - ~32,000 cycles (temp. unknown) |

## VISUAL OBSERVATIONS

Visual observations of the reflector materials post-flight revealed few changes. The most significant changes occurred in the reflector samples of silver overcoated with magnesium fluoride and sapphire. These samples were more diffuse due to space environment exposure. The exposed areas appeared scratchy and non-reflective. One sample in particular (fig. 3) was darker, with increased oxidation in atmosphere. The flight silver samples with silicon dioxide coatings had a few pits but remained reflective. The silver alloy reflectors overcoated with a dielectric multilayer suffered some scratches but also remained reflective. The aluminum reflectors with silicon dioxide or silicon monoxide coatings appeared unchanged (fig. 4), as did the enhanced aluminum reflectors. Black light observations did not reveal any obvious molecular contaminant deposition.

## OPTICAL PROPERTY MEASUREMENTS

A variety of instruments are available for optical measurements in the Engineering Physics Division of the Materials and Processes Laboratory at the Marshall Space Flight Center. Post-flight reflectance measurements were performed on both control and flight samples with several instruments as follows - a Varian Cary model 2300 spectrophotometer, a Beckman model DK-2A spectrophotometer, a Perkin-Elmer model Lambda 19 spectrometer, and an AZ Technology Laboratory Portable Spectroreflectometer (LPSR). Thermal emittance measurements were made with a Gier-Dunkel DB 100 portable infrared reflectometer.

Reflectance and thermal emittance measurements for selected samples are given in Table 2. The reflectance measurement given for each material is the integrated reflectance from 350 nm to 1400 nm. The exposed reflectance measurements shown are for the worst case (highest $\Delta R_s$) in each sample group. The range of reflectance change for each sample group is also given. Small decreases in reflectance were noted for the majority of exposed samples. Changes in reflectance were consistent within each sample group with the exception of the silver samples coated with magnesium fluoride and sapphire. These reflectance changes are in agreement with the visual observations, with the most obviously degraded sample experiencing a 17% drop in solar reflectance (fig. 5). Thermal emittance measurements on all samples showed no significant changes due to space exposure.

## CHANGES IN SURFACE MORPHOLOGY

Precision angstrometer traces were made on all the samples using a Wyko Topo-3D surface profiler. A decrease in film height was noted in the exposed areas of the flight samples. A typical trace of a half-moon sample is shown in fig. 6. The changes in thickness of the exposed materials are given in Table 3 and range from 25 up to 160 Å. For applications of these materials where the total coating thickness is 1000 to 2000 Å, the percentage change is considerable, and the effect can be substantial for space optics. If these changes in thickness are assumed to be the result of atomic oxygen interaction, then the calculated reaction efficiencies, based on the angstrometer measurements of a change in layer height, range from 0.4 to 2.3 x $10^{-28}$ $cm^3$/atom.

Roughness of the samples was measured using a TMA Technologies μScan instrument. RMS roughness measurements are given in Table 3. Measurements of unexposed areas of the half-moon samples agreed well with control surface measurements. These roughness measurements also agree with visual observations, particularly of the oxidized silver/$MgF_2$-sapphire samples.

Ellipsometry measurements were made using a Gaertner Waferskan model L115B. For given N, the refractive index, and K, the extinction coefficient, calculations based on the Fresnel equations indicate a reflectance equal to the measured reflectance for an aluminum reflector coated with 1300 Å of silicon monoxide. The top layer thickness measurements are nominally in agreement with the measurements indicated by RBS analysis, as discussed in the next section.

## RUTHERFORD BACKSCATTERING ANALYSIS

Rutherford Backscattering (RBS) analysis was performed on a number of the half-moon reflector samples. RBS is based on the concepts of coulomb scattering and heavy ion energy loss in matter. In most applications, alpha particles are used as the incident beam. A collimated beam of mono-energetic alpha particles impinges on a target, and a small fraction of these particles is scattered due to coulomb interactions with atomic nuclei in the target. Scattering may occur at the front surface of the target or at depths within the target. If the alpha particle penetrates the target instead of scattering from the front surface, the alpha particle loses energy continuously during the penetration due to coulomb interactions with atomic electrons. This measured energy loss produces information related to the depth within the sample at which a scattering event takes place.

During this investigation, a 2 MeV beam energy, a 0.015" beam diameter, and 2 microcoulomb total charge to the sample were used. The incident alpha particles impinged normal to the sample, with the scattered alpha particles measured through an angle of 160 degrees. RBS spectra were obtained from both the exposed and unexposed areas of the reflector samples.

The nickel substrate, being massive relative to the coating materials, dominates the RBS spectrum up to approximately 1200 KeV. The presence of this nickel signal complicates the analysis with respect to the lighter elements.

Results from the RBS analysis are given in Table 4. A typical RBS spectra of the exposed and unexposed areas of an apparently unaffected silver/silicon dioxide reflector is shown in Figure 7. Slight diffusion was apparent at the silicon dioxide-silver and silver-nickel substrate interfaces in both spectra, but the thickness of the $SiO_2$ layer was not significantly changed. Enhanced aluminum with magnesium fluoride-sapphire overcoating showed the most significant change in RBS spectra. Figure 8 shows the diffusion of the silver in the enhanced aluminum into the nickel substrate and into the coating layer. However, this observation was not consistent among the samples. This diffusion was observed in sample #51 but not #60. These samples received the same amount of space exposure; therefore the cause of this variation is not obvious. Analysis of these samples is continuing.

To sum up the Rutherford backscattering analysis results, the silver and silver alloy samples, in general, revealed some diffusion between the silver-substrate and silver-protective coating interfaces for both exposed and unexposed material. Space environment effects on these materials, as indicated by surface profilometry and other measurements, were not obviously revealed by RBS analysis. The aluminum and enhanced aluminum samples with $SiO_x$ coatings appeared to be stable in the space environment. The enhanced aluminum with a magnesium fluoride-sapphire protective coating appeared to be affected by space exposure, but this observation was not consistent among the samples nor in agreement with angstrometer traces.

## ESCA ANALYSIS

An aluminum/$SiO_2$ reflector, a silver/$MgF_2$-sapphire reflector, and other material samples from this experiment plate were selected for ESCA analysis. The aluminum reflector was half-exposed to the environment, so three measurements were made on the unexposed and exposed areas. Only one of the three measurements on the exposed area has a significantly higher carbon content (C 1s peak) than that of the control area (see Table 5). The silicon to oxygen ratio is near 0.5, in agreement with the $SiO_2$ top layer chemistry.

The silver/$MgF_2$-sapphire reflector was fully exposed to LEO, therefore only exposed ESCA data is given in Table 5. Aluminum, from the sapphire layer, was found in atomic percentages of 12 and 16% in the two measured areas. Silver was also found in atomic percentages of 3 and 7%. Magnesium and fluoride peaks were not reported. The level of oxygen found is in agreement with the sapphire $Al_2O_3$ layer and formation of silver oxide and/or silicon oxide during the flight. This oxidation layer was not significant enough to appear on RBS spectra. For the silver reflector, carbon levels are typical of LDEF flight samples, though some of the carbon may have been deposited during post-flight storage before ESCA analysis was performed. From these measurements and results from other Plate IV materials, there is some variability in contaminant deposition in exposed sample areas. Visual inspection indicate these reflectors appear to be reasonably clean.

## CONCLUSIONS

Degradation of the solar reflectors, when it occurred, was not uniform across the material. When designing for the low Earth orbital environment, solar reflector materials require careful selection. The best performance of a solar reflector material in terms of property maintenance was obtained from a fully oxidized coating over aluminum. All of the coatings experienced densification, decreased thickness, and increased surface roughness, but the measured solar reflectivity values were better than expected based on visual observations. Intercoating diffusion was observed with Rutherford backscattering analysis.

## REFERENCES

Whitaker, A.F., Kamenetzky, R.R., Finckenor, M.M., and Norwood, J.K. Atomic Oxygen Effects on LDEF Experiment A0171. Second LDEF Post-Retrieval Symposium, San Diego, CA, June 1992.

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Table 2. Optical Property Changes of Solar Reflectors

| Solar Reflector/Coating | Control $R_S$ | Exposed $R_S$ | $\Delta$ $R_S$ (%) | Control $\epsilon_{IR}$ | Exposed $\epsilon_{IR}$ |
|---|---|---|---|---|---|
| Ag/$SiO_2$ | 0.94 | 0.93 | -1 | 0.03 | 0.03 |
| Ag/$MgF_2$-Sapphire | 0.93 | 0.76 | -4 to 17 | 0.02 | 0.02 |
| Ag Alloy/Dielectric | 0.92 | 0.90 | -1 to 2 | 0.02 | 0.02 |
| Al/$SiO_2$ | 0.87 | 0.87 | <-1 | 0.03 | 0.03 |
| Al/SiO | 0.87 | 0.85 | -2 | 0.02 | 0.02 |
| Enhanced Al/SiO-$SiO_2$ | 0.88 | 0.86 | -2 | 0.02 | 0.02 |
| Enhanced Al/$MgF_2$-Sapphire | 0.89 | 0.88 | <-1 | 0.05 | 0.05 |

Table 3. Changes in Film Morphology

| Solar Reflector/Coating | Decrease in Film Thickness (Å) | Control RMS Roughness (Å) | Exposed RMS Roughness (Å) |
|---|---|---|---|
| Ag/$SiO_2$ | 40 | 45 | 60 |
| Ag/$MgF_2$-Sapphire | 150 | 25 | 75 |
| Ag Alloy/Dielectric | 160 | 30 | 40 |
| Al/$SiO_2$ | 50 | 45 | 40 |
| Al/SiO | 150 | 30 | 35 |
| Enhanced Al/SiO-$SiO_2$ | 125 | 30 | 30 |
| Enhanced Al/$MgF_2$-Sapphire | 25 | 25 | 30 |

Table 4. Rutherford Backscattering Analysis Results

| Solar Reflector/Coating | Observations |
|---|---|
| Ag/$SiO_2$ | No appreciable change<br>Slight diffusion at interfaces |
| Ag/$MgF_2$-Sapphire | Visually apparent damage is not uniform enough to appear on RBS spectra |
| Ag Alloy/Dielectric | Slightly increased diffusion at layer interfaces where exposed to space environment |
| Al/$SiO_2$ | No appreciable change |
| Al/SiO | No appreciable change |
| Enhanced Al/SiO-$SiO_2$ | Little difference between exposed and unexposed spectra.<br>Protective layer appears to be mainly $SiO_2$ |
| Enhanced Al/$MgF_2$-Sapphire | Silver of enhanced aluminum material diffused into surrounding layers.<br>Results not consistent in sample group. |

Table 5. ESCA Survey Results

| Location | Si (at%) | O (at%) | Ratio Si/O | C (at%) |
|---|---|---|---|---|
| Aluminum/$SiO_2$ | | | | |
| Unexposed #1 | 32 | 54 | 0.59 | 14 |
| #2 | 33 | 51 | 0.65 | 16 |
| #3 | 31 | 53 | 0.58 | 16 |
| Exposed #1 | 27 | 47 | 0.57 | 26 |
| #2 | 31 | 50 | 0.62 | 19 |
| #3 | 30 | 55 | 0.55 | 15 |
| Silver/$MgF_2$-Sapphire | | | | |
| Exposed #1 | 15 | 50 | 0.30 | 20 |
| #2 | 14 | 41 | 0.34 | 22 |

**Figure 3**
Silver reflector with $MgF_2$-sapphire coating

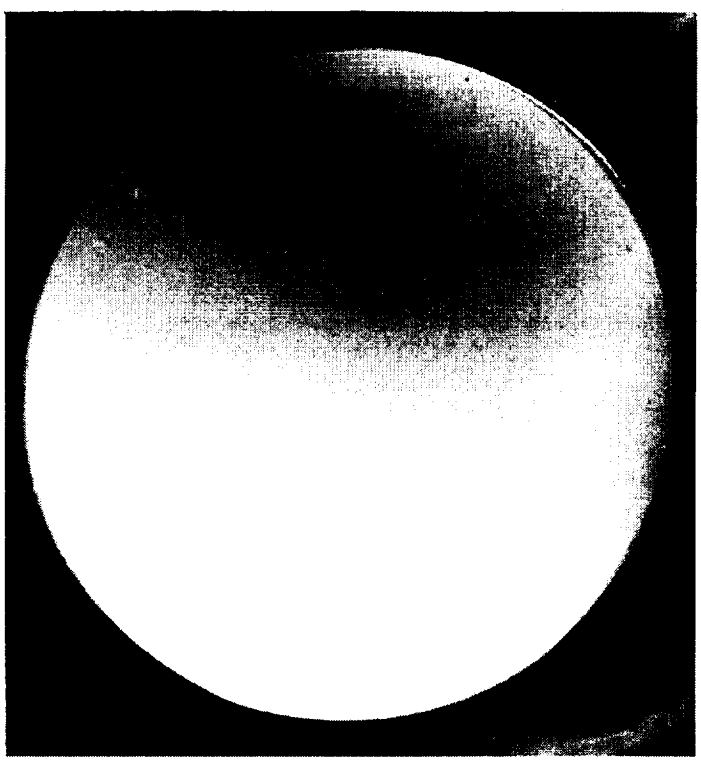

**Figure 4**
Aluminum reflector with $SiO_2$ coating



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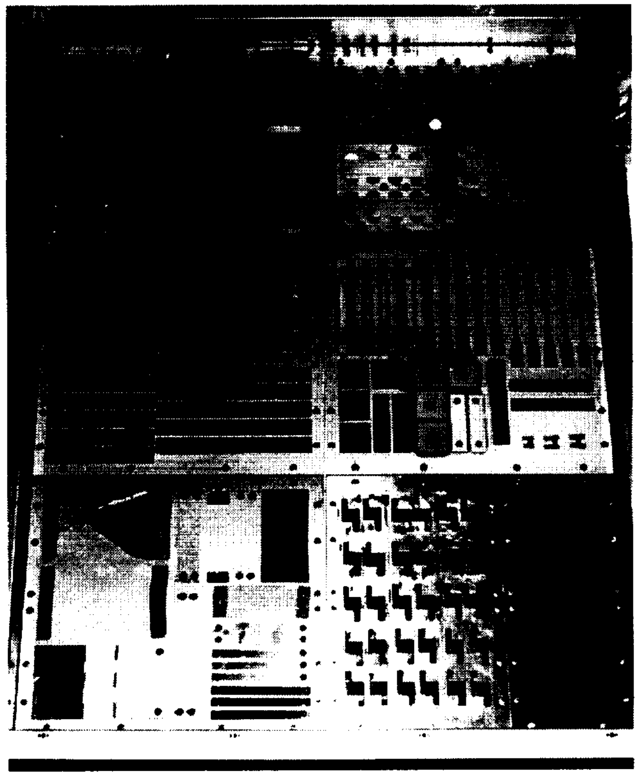

Plate IV

**Figure 1**
LDEF Experiment A0171 (SAMPLE)

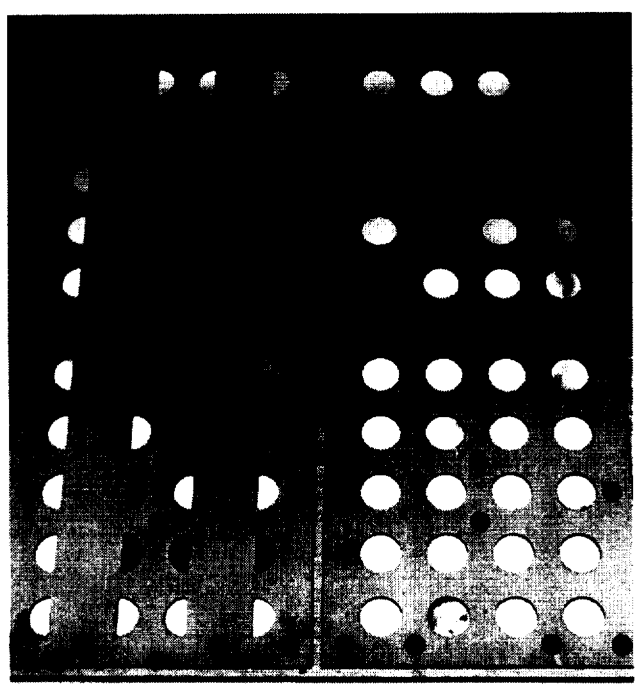

**Figure 2**
A0171 Plate IV - Thermal Control Coatings

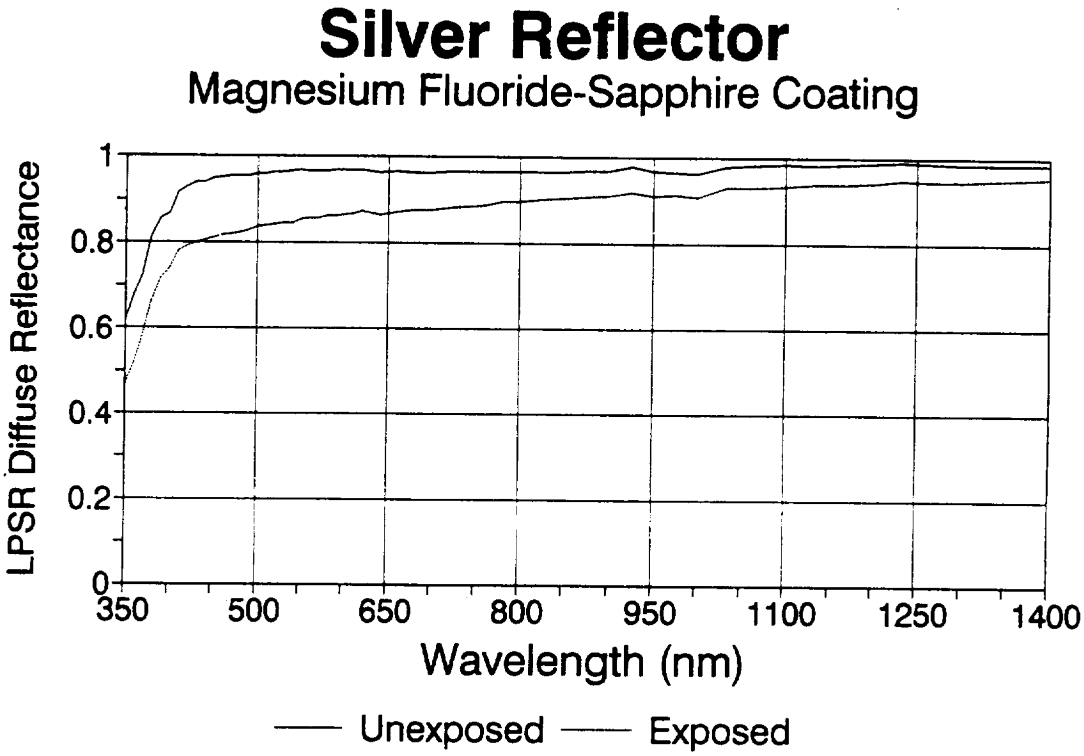


Figure 5

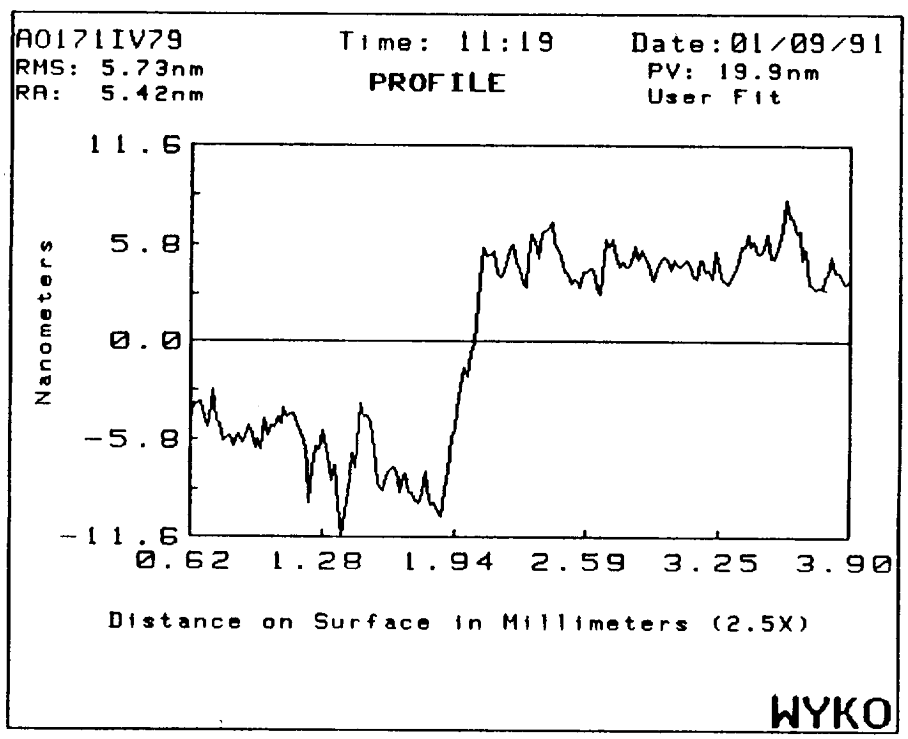


Figure 6

Angstrometer trace of
silver reflector with $SiO_2$ coating
Transition between exposed/unexposed areas

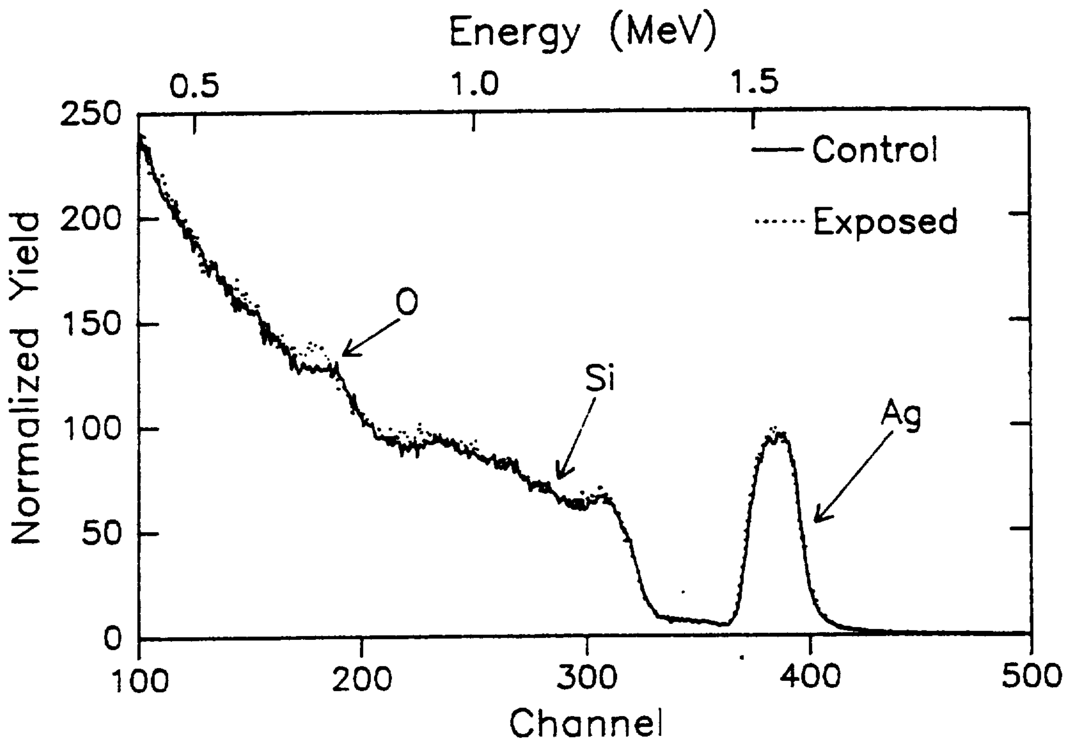


**Figure 7: RBS Spectra of $Ag/SiO_2$**

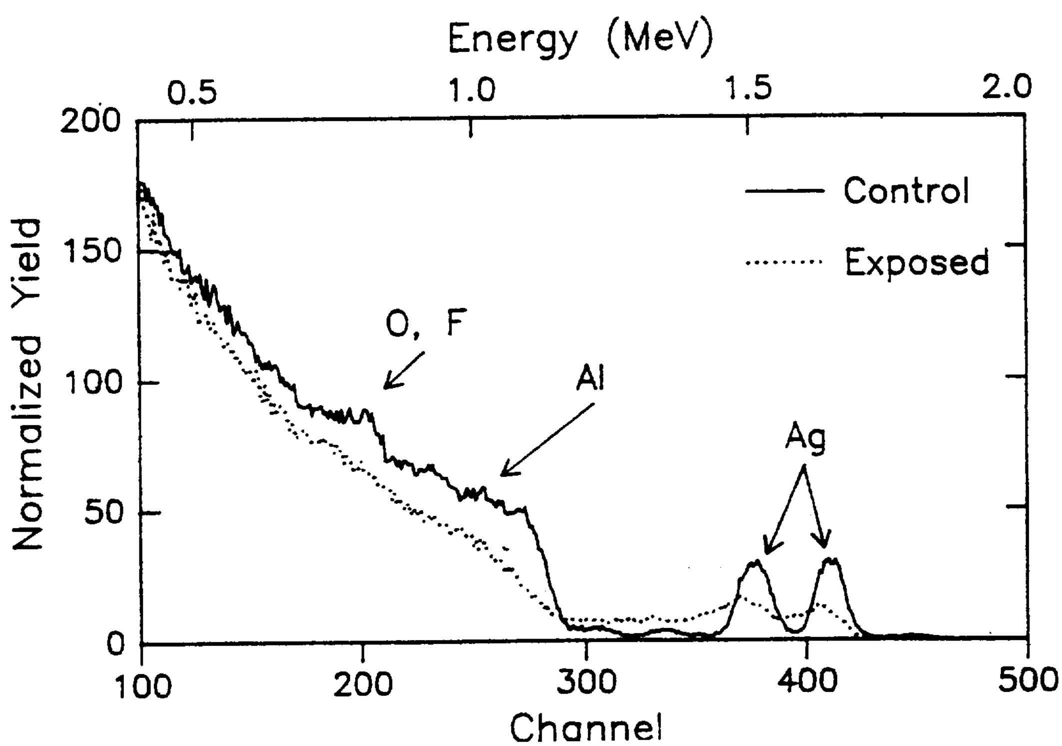


**Figure 8: RBS Spectra of Enhanced $Al/MgF_2$-Sapphire Sample Indicating Interlayer Diffusion**