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Preliminary results from Infrared Multilayer Filters and Materials exposed to the space environment on the NASA LDEF mission

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ABSTRACT

With continually increasing demands for improvements to atmospheric and planetary remote-sensing instrumentation, for both high optical system performance and extended operational lifetimes, an investigation to assess the effects of prolonged exposure of the space environment to a series of infrared interference filters and optical materials was promoted on the NASA LDEF mission.(2)

The NASA Long Duration Exposure Facility (LDEF) was launched by the Space Shuttle to transport various science and technology experiments both to and from space, providing investigators with the opportunity to study the effects of the space environment on materials and systems used in space-flight applications.(4)

Preliminary results to be discussed consist of transmission measurements obtained and processed from an infrared spectrophotometer both before (1983) and after (1990) exposure compared with unexposed control specimens, together with results of detailed microscopic and general visual examinations performed on the experiment.

The principal lead telluride (PbTe) and zinc sulphide (ZnS) based multilayer filters selected for this preliminary investigation consist of : an 8-12 μm low pass edge filter, a 10.6μm 2.5% half bandwidth (HBW) double half-wave bandpass filter, and a 10% HBW triple half-wave bandpass filter at 15 μm. Optical substrates of MgF_2 and KRS-5 (TlBrI) will also be discussed.

1. INTRODUCTION

The Long Duration Exposure Facility (LDEF) was designed as a reusable vehicle for transporting experiments to and from earth orbit using the two way transport capability provided by the Space Shuttle. It is a free flying twelve sided cylindrical structure 9.1 metres long by 4.3 metres diameter containing a total of eighty-six self contained experiment trays mounted on the exterior. Each tray measures 86.4 cm x 127 cm which can be sub-divided into smaller units accommodating different sized experiments.(4)

The 9709 kg Facility was launched in April 1984, being released from the shuttle using a remote manipulator system in the payload bay and placed in an orbit of 556 km at an orbital inclination between 28.5° and 57° relative to the equator. The structure is three-axis gravity-gradient stabilized to maintain its correct orientation in space, assisted by a viscous magnetic damper mounted internally to remove residual rotation of the spacecraft whilst in orbit.(1)

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Duration of the exposure was originally anticipated to last for a period of 6-12 months, however due to a number of operational reasons and the Challenger tragedy of January 1986, the experiment was eventually recovered in January 1990. The recovery was made urgent by the onset of orbital decay exacerbated by the then highly active sunspot cycle which would have resulted in re-entry and loss of the vehicle.

LDEF is an international facility with the mission involving investigators from ten different countries, each investigator designing and building their own experiment tray to ensure mechanical and thermal integrity, depending on the nature of the experiment.

The principal selection of flight specimens for the Reading University experiment (#A0056) comprised spectrally selective filters from the then current atmospheric remote-sensing and weather forecasting satellite programmes, Viz: NIMBUS 4,5,6,7, ITOS, TIROS-N and planetary probes from the PIONEER and GALILEO research programmes.(2)

The principal objectives of the experiment were to ;

(i) Investigate the effects of the space environment on the spectral and mechanical stability of the sample set,
(ii) Assess the useful lifetime of the samples in an orbital environment,
(iii) Using post-flight investigations, determine the degradation (if any) and mechanisms affecting the optical system performance.

The high performance infrared filters, coatings and materials were exposed to the complete diversity of space radiations and temperature excursions provided by two locations on the vehicle. The level of exposure exceeds that anticipated for the use of these types of components and so would provide a better indication of the long term performance and stability of the filters. One location was continuously facing earth (G12) whilst the other location continuously faced space on the leading edge (B8) of the vehicle.

Samples exposed on the LDEF provided a unique opportunity to investigate the effects of the space environment on crucial radiometer filters and materials being used in space-flight instrumentation. By direct testing in space, more detailed information on the long-term performance of optical system components could be obtained.

By understanding the mechanisms involved with the current experiment, the aim is to develop and produce improved optics with long-term stability and greater resistance to the space environment.

2. DEGRADATION MECHANISMS

Following the retrieval of the facility, a detailed inspection of the complete structure was performed in the clean room conditions of the Safety and Escapulation Facility (SAEF2) at Kennedy Space Centre, Florida. NASA engineers reported the structure to be in a satisfactory condition with no unanticipated phenomena observed. However, general damage to a number of experiments containing thin films, coatings and thermal blanket materials had been sustained on the leading edge and space facing end of the vehicle. No major impact events occurred on the vehicle, the majority of smaller impact craters being detected on the leading edge, though craters were reported over the complete structure. Radiation measurements performed on the facility did show a measurable amount of induced radiation. This amount however was not considered to present any possible dosage threat to personnel in contact with the vehicle.

To study the degradation mechanisms and environmental effects on experiments attached to the facility, NASA has initiated various Special Investigation Groups (SIG's) to analyse the structure and report on the space environment to which LDEF was exposed.

2.1 Meteoroid and debris

The meteoroid and debris investigation intends to catalogue the location, size and characteristics of all impact craters on the vehicle greater than 0.5 mm diameter, this is expected to exceed 5000 entries. From this data it is anticipated the meteoroid impact flux/mass distribution together with directional distribution information of materials debris from low earth orbit will be determined. Chemical and isotopic analysis will be performed on an even distribution of impact craters from the complete vehicle, this will be used to characterize the cratering phenomena of the space-craft and investigate any exobiology information.

2.2 Thermal properties

The thermal properties of the LDEF environment were recorded for the first year of the spacecraft life. A thermal investigation group intend to develop and produce an end-of-mission thermal model by comparing data obtained from the first year's flight with the physical effects of temperature experienced by degradation of various coating materials over time and temperature distribution. By using details from the orbital parameters, altitude, and coatings from pre to post flight, a model will be produced recording the complete in-flight thermal environment of the Facility. An approximate 10°C rise in temperature was monitored as LDEF returned to earth.

2.3. Atomic oxygen (AO)

A flux of atomic oxygen (monatomic oxygen) was known to bombard the LDEF structure at the leading edge locations 7, 8, and 9 with an average energy of ~4.5 eV. The total velocity of atomic oxygen incident to the surface of the experiment is defined as the summation of the orbital RAM velocity, the atmospheric rotational velocity and the maxwellian velocity distribution.

The effects of atomic oxygen bombardment on a uniform polymer film or soft elastic material produces a heavily textured, rough, cloudy appearance across the surface. An organic film coated with a thin metallic layer experiences the impact of atomic oxygen by producing pinholes through the metal coating. Due to the high energy state of atomic oxygen, multiple internal reflections generate a cavity below the surface of the metal film. Recording the relative orientation of each sample and its experimental position was therefore essential to characterize the directional distribution and nature of the bombardment received.

Further information on radiation dosages, meteoroid and debris impacts, thermal properties and atomic oxygen bombardment will be presented in a more detailed paper at the conclusion of this investigation.

3. EXPERIMENT CONSTRUCTION

The exposed sample set, consisting of 46 components, were housed in two chromically anodised aluminium base plates; each sample being held in individual aluminium holders, designed for mechanical stability and intimate thermal contact to ensure a uniform temperature distribution across the exposed aperture of the substrate (Figure 1). Both the samples and holders were retained using disc springs and circlips, thermal contact was ensured by using lead washers and backing pieces located behind the substrate.

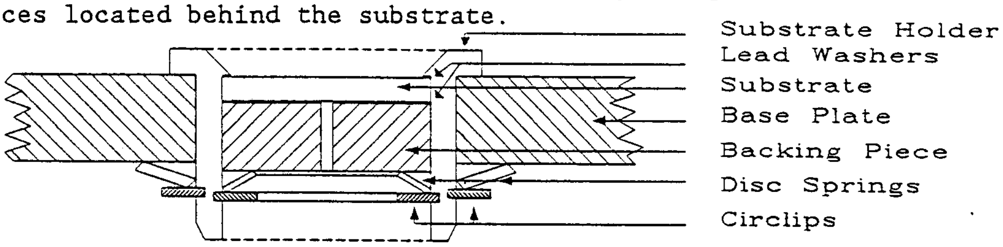

Figure 1. Mechanical arrangement of LDEF sample holders.

The following disposition of components was made between the two locations:

Uncoated substrate materials

Substrate	Exposed Diameter (mm)	Location (No. off)
CaF_2	27.0	B8(1)/G12(1)
MgF_2	27.0	B8(1)/G12(1)
Ge	23.0	B8(1)
Si	13.0	B8(1)
CdTe	13.0	B8(1)/G12(1)
Al_2O_3	21.0	B8(1)
Y-cut quartz	13.0	B8(1)
Z-cut quartz	13.0	B8(1)
KRS-5 (TlBrI)	27.0	B8(1)/G12(1)
KRS-6 (TlClBr)	27.0	B8(1)/G12(1)

Coatings and filter materials

Coating Material	Substrate	Exposed Diameter mm	Location (No. off)
PbTe/ZnS	Ge	21.0	B8(8)/G12(8)
PbTe/ZnSe	Ge	21.0	B8(2)/G12(2)
PbTe/ZnS/SiO	Si	21.0	G12(1)
SiO	Si	21.0	B8(1)
Ge/SiO	AL_2O_3	23.0	B8(2)
PbF_2	ZnSe	21.0	G12(1)
AS_2S_3/KRS-5	KRS-5	13.0	B8(1)/G12(1)
CdTe/KRS-5	KRS-5	13.0	B8(1)/G12(1)
ZnS/ZnSe/KRS-5	KRS-6	13.0	B8(1)/G12(1)

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The choice of both substrate and coating materials selected for the two locations in the experiment still provide a good cross-section of optical components currently being implemented in infrared radiometer instrumentation.

4. VISUAL EXAMINATIONS

4.1 General

A visual examination of the experiment trays was performed upon receipt of the LDEF in the SAEF2 clean-room at Kennedy Space Centre. The general appearance of the aluminium base plate for the leading edge tray B8 revealed an unevenly distributed brown discolouration of the chromic anodising over the complete surface. This effect was also present on the earth facing tray G12 but was restricted to various isolated locations distributed around the tray.

A general inspection of the experiment samples produced no evidence of coating failure or delamination, however noticeable colour changes had occurred on the uncoated KRS-5 and KRS-6 samples. Tray B8 had received considerably more impact damage on the leading edge than the earth facing tray G12. The uncoated Calcium Fluoride sample on B8 possessed major impact damage, cleaving the substrate in two directions at approximately 120 degree spacing from the point of impact.

4.2 Microscopic

Samples were removed from their assemblies at the SERC Rutherford Appleton Laboratory clean room where a microscopic examination of each sample was performed.

The following characteristics were observed for samples where specific changes had occurred in appearance :-

Sample No. (Type)	Comments/observations
Tray B8	
2 (CaF_2 substrate)	Major impact area, substrate cleaved in two directions approximately 120 degrees spacing from impact, no remaining debris retained in the impacted area.
3 (KRS-5 substrate)	Evenly discoloured orange-brown appearance across the full substrate aperture. Uniformly distributed number of small surface blisters visible.
4 (KRS-6 substrate)	Contamination produced a translucent brown film evenly distributed over the complete element aperture.

17 (KRS-6 substrate + ZnS/ZnSe/KRS-5 coating)	Surface coating crazed along stress lines and bowed out from the substrate surface at the centre of the aperture. A number of coating flakes have disappeared from the crazed area.
19 (KRS-5 substrate + CdTe/AS_2S_3/KRS-5 coating)	Large blister at the centre of the element, extending across the full element aperture. Substrate has been effectively destroyed (blackened). No evidence of impact craters.
37 (Ge substrate + PbTe/ZnS coating)	Large impact crater located at holder/ substrate interface (no remaining debris). Coating material delaminated around pheriphery of impacted area.
47 (Si substrate + SiO coating)	Major impact crater observed at the edge of the element mounting holder (no remaining debris), but aluminium material has sputtered off the mounting surface and back reflected across the surface of the substrate. Coating remains in good condition.

<u>Tray G12</u>

7 (KRS-5 substrate)	Element discolouration separated into two regions, the larger being translucent where a uniform grey contamination has formed on the element surface. The smaller region is more transparent where the original substrate colour is visible. Small periodic striations are visible producing localized colour changes.
8 (KRS-6 substrate)	Uniform brown discolouration across the full aperture of the element. Small localized area of surface blistering observed.
44 (ZnSe substrate + PbF_2 coating)	Opaque contamination material distributed in the bulk substrate over a localized region of the element producing colour discontinuity.

The visual inspection performed on the remaining uncoated (and harder) substrate and coated materials revealed various repeatedly-observed characteristics. Viz;

(i) A small quantity of particulate contamination material evenly distributed over a large number of samples producing a finely divided white speckled surface on many elements,

(ii) Various harder crystals had received tiny impacts chipping small amounts of material from local impact areas,

(iii) On many of the multilayer coated optics micropitting was evident in a small number of locations across the exposed aperture.

5. MULTILAYER DESIGN SELECTION

Three different types of multilayer design were selected for the preliminary spectral investigation. These were considered to represent a good cross-section of those exposed and would provide a good indication of any unanticipated changes which may have occurred.

(i) Sample B8/37 (Figures 2,3.) consisted of a Germanium substrate coated with a complete PbTe/ZnS 8-12μm fully blocked long-wave pass edge filter. The exposed surface carried a 19-layer principal stack, with its 50% edge located at ~1300 cm^{-1} (7.7μm). The unexposed surface carried a 17-layer subsidiary (and linking) stack to provide continuous short wavelength blocking and antireflection. This sample was selected to assess any changes occurring across a typically wide region of transparency.

(ii) Sample G12/40 (Figures 4,5.) comprised a Germanium substrate coated with a 13-layer PbTe/ZnS L-spaced triple half-wave 10% wide bandpass filter at 15μm. The core of the bandpass filter had the following structure:

Substrate (Ge) / X $\underline{LL}HLH\underline{LL}HLH\underline{LL}$ X / Air,

To this was added simulated Herpin matching (X) at either end of the stack to provide sufficient passband transparency. This sample was chosen with the matching being particularly spectrally sensitive to changes in thickness, index or absorption.

(iii) Sample B8/42 (Figures 6,7.) was a traditional 13-layer H-spaced PbTe/ZnS double half-wave bandpass filter, 2.5% wide at 10.6μm with the following structure:-

Substrate (Ge) / $LHL\underline{HH}LHLHL\underline{HH}LHL$ / Air

The choice of this sample was based on the sensitivity of the narrow peak to any selective graded absorption or changes of refractive index occurring throughout the structure.

6. PRE-FLIGHT MEASUREMENTS

Each of the control samples and those exposed on the LDEF was measured in transmission and reflection prior to the flight in 1983 using a Perkin-Elmer 457 grating infrared spectrophotometer. The measurements were performed between 2.5 and 40 μm and expanded in the localized passband region of interest.

The 457 is a double beam optical null spectrophotometer, recording linear transmittance verses linear wavenumber through an f/5 monochromator. The instrument is designed with a pre-sample chopping arrangement.

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The following measurement accuracy and repeatibility is quoted:(5)

Wavenumber accuracy	< ±4cm^{-1} between 4000-2000 cm^{-1}
	< ±2cm^{-1} between 2000-250 cm^{-1}
Wavenumber repeatability	2 cm^{-1} from 4000 to 2000 cm^{-1}
	1 cm^{-1} from 2000 to 250 cm^{-1}
Transmission accuracy	±1% of full scale
Transmission repeatability	within 1% of full scale

Variations in transmittance of ~1% full scale deflection are defined by the peak to peak noise value of the thermocouple detector (Johnson noise) and pre-amplifier.

A polystyrene calibration spectra produces a measured accuracy of approximately ±1cm^{-1} at various well defined absorption peaks.

7. POST-FLIGHT MEASUREMENTS

Post flight measurements conducted on both exposed and control samples were performed on a Perkin-Elmer 580A infrared spectrophotometer over the region 4000 cm^{-1} - 160 cm^{-1}. This equipment has been fitted with a data acquisition system comprising an IBM-clone microcomputer installed with an A/D converter. The system is operated by in-house software comprising both the necessary processing of 24000 12-bit data points, and multilayer coating design to produce overlays of the original calculated design spectra and actual measurements after fabrication.

The PE580A is a double beam grating spectrophotometer equipped with a dual post-sample chopping arrangement to eliminate measurement of re-radiation from the sample or reference beam. It contains an f/5.7 monochromater.

Measurement accuracy is quoted over the following wavenumber ranges for the instrument:(6)

1.5 cm^{-1} decreasing linearly to 1 cm^{-1} between 4000-3500 cm^{-1},

1 cm^{-1} between 3500-2000 cm^{-1},

1 cm^{-1} decreasing linearly to ±0.5 cm^{-1} between 2000-1450 cm^{-1}, and

0.5 cm^{-1} from 1450-180 cm^{-1}

Wavenumber repeatability is 0.5 cm^{-1} from 4000 cm^{-1} to 2000 cm^{-1} and 0.25 cm^{-1} from 2000 cm^{-1} to 180 cm^{-1}.

Transmittance accuracy from the data acquisition readout is:

±0.2% between 4000 cm^{-1} -700 cm^{-1} and

±0.5% between 700 cm^{-1} and 180 cm^{-1}

Transmittance repeatability errors are quoted as being better than the measured accuracy of the instrument.

8. PRE-POST FLIGHT COMPARISONS

Comparisons of the filter spectra measured on the two spectrophotometers, before and after exposure, is performed electronically and displayed as direct overlays permitting an immediate visual correlation between equivalent spectral features. To provide compatability, spectral characteristics measured on the PE457 spectrophotometer are optically digitized using a Hewlett-Packard Scanjet plus flat-bed scanner, and processed using an in-house spectral chart recording digitizer program specially developed for this application. The scanner produces a high quality (300 dpi) image of the spectra from which the trace is extracted, this is compatible with the converted A/D format from the PE580A workstation. Compensation for wavenumber accuracy is performed using the well defined absorption peaks of polystyrene. Correcting for variations in transmission (100%, 0%) before the sample is introduced is also done with the software, by calculating the new transmission levels of the spectra.

The comparative accuracy and repeatability between the two spectrophotometers provides a good indication of the validity. Any spectral changes falling close to, or outside of this general guideline can therefore be considered as arising from the exposure to space.

	PE457	PE580A	Total
Wavenumber accuracy (cm^{-1})			
4000-2000 cm^{-1}	4	1	±5 cm^{-1}
2000-250 cm^{-1}	2	0.5	±2.5 cm^{-1}
Wavenumber repeatibility (cm^{-1})			
4000-2000 cm^{-1}	2	0.5	2.5 cm^{-1}
2000-250 cm^{-1}	1	0.5	1.5 cm^{-1}
Transmission accuracy (%)			
4000-700 cm^{-1}	1	0.2	1.2%
700-250 cm^{-1}	1	0.5	1.5%

9. RESULTS

Spectral profile analysis performed on the selected filters (see Figures 4-7) produced the following results.

Edge filters

Sample	5% pt.	80% pt.	50% pt.	Edge Steepness (%)	Average Transmission (8-12.5μm)	Peak Transmission (~11μm)
C-6 Before	1349	1292	1307	4.38	87.24	93.5
C-6 After	1341	1287	1302	4.13	87.64	93.5
B8-37 Before	1341	1283	1297	4.50	87.89	94.5
B8-37 After	1335	1283	1297	3.98	85.36	92.0

Bandpass filters

Sample	Short wave 50% pt.	Long wave 50% pt.	Centre Wavenumber	HBW (%)	Peak Transmission
C-13 Before	684	616	650	10.45	89.3
C-13 After	685	618	652	10.25	88.5
G12/40 Before	684	616	650	10.48	88.7
G12/40 After	688	621	654	10.27	89.5
C-11 Before	959	933	946	2.71	91.4
C-11 After	958	933	945	2.60	90.1
B8/42 Before	957	932	944	2.62	91.9
B8/42 After	956	931	943	2.64	89.5

The most significant changes on-board occurred on the exposed edge filter (B8/37 - Figure 2), where the transmission reduced by ~2.5% across the full 8-12 μm passband. This absorption may be due to a surface effect in the outer layers of the structure as there has been no degradation in either transmission or shape in the most sensitive region, viz: at the top of the edge.

A difficulty arises in making comparisons beyond 20 μm due to an inherent lack of energy in the performance of the 457 spectrophotometer; leading to a poor pen response with its associated inaccuracies.

Most other observed spectral variations fall within the error budget of the spectrophotometers.

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There were no changes observed for the MgF_2 crystal (Figure 8) exposed on the LDEF leading edge; however measurement of a KRS-5 sample (Figure 9) from the leading edge showed a considerably reduced transmission level and indicate possible "metallic"-like surface contamination. This same characteristic has also been observed for the KRS-6 (TlClBr) sample located on both sites of the experiment, to be investigated later.

10. CONCLUSION

The preliminary results discussed in this paper now establishes further confidence in the long term spectral and mechanical stability of infrared filters and materials exposed to the space environment.

The information is particularly welcome at this time, when the operational lifetimes for the next generation of remote-sensing radiometers are lengthening to between 10 and 15 years.

The investigation continues.

11. ACKNOWLEDGMENTS

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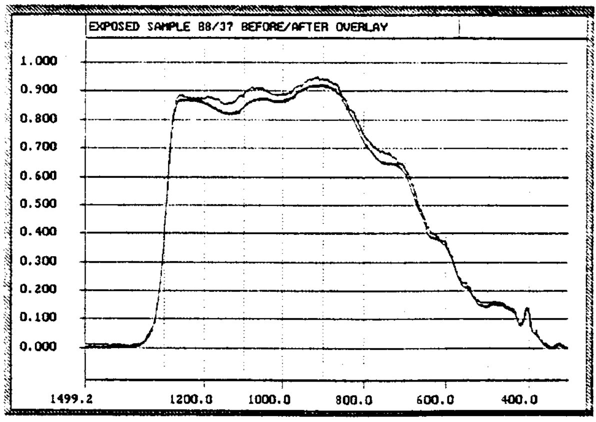

Fig. 2 8-12 μm low pass edge filter.
Overlay before/after exposure.

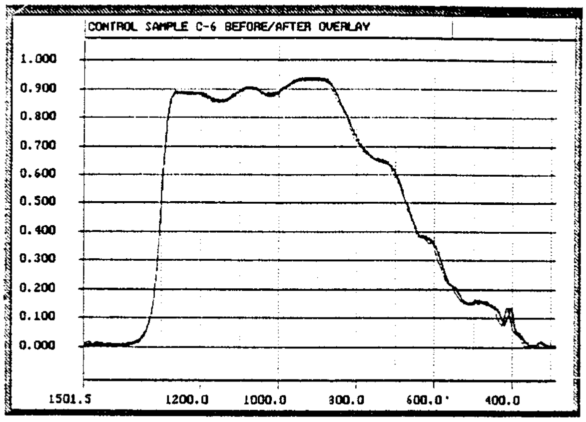

Fig. 3 8-12 μm low pass edge filter.
Control sample measurement.

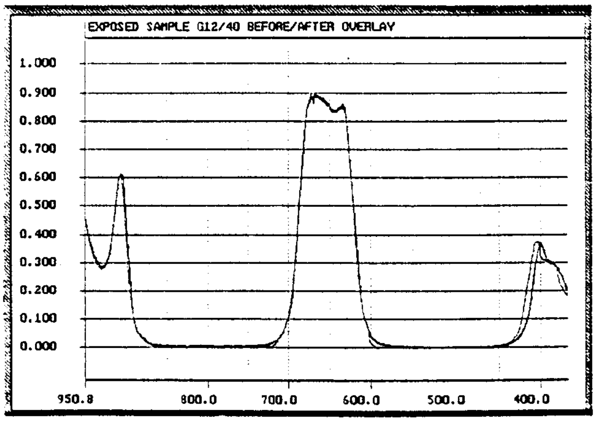

Fig. 4 15μm bandpass filter.
Overlay before/after exposure.

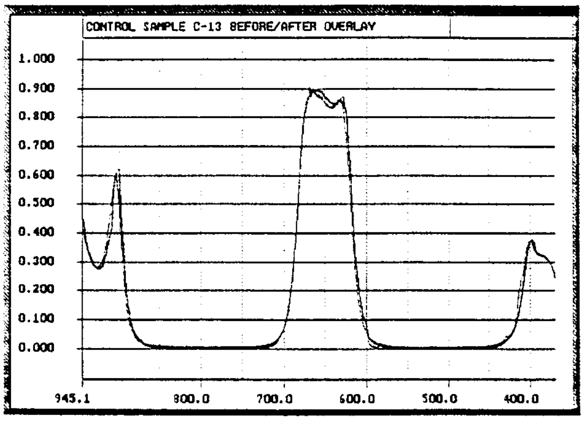

Fig. 5 15μm bandpass filter.
Control sample measurement.

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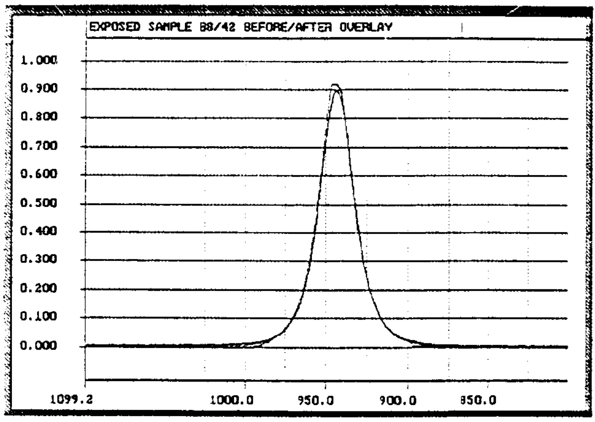

Fig. 6 10.6 μm bandpass filter.
Overlay before/after exposure.

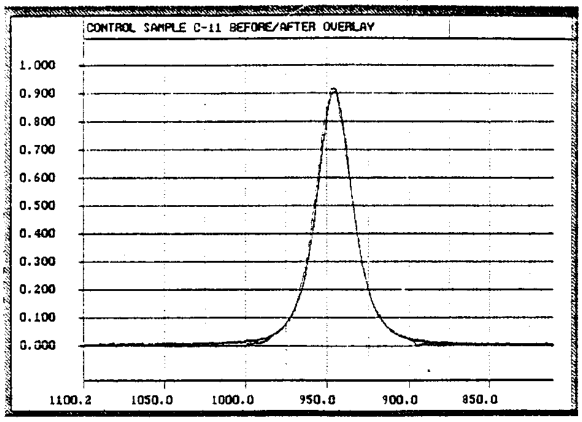

Fig. 7 10.6 μm bandpass filter.
Control sample measurement.

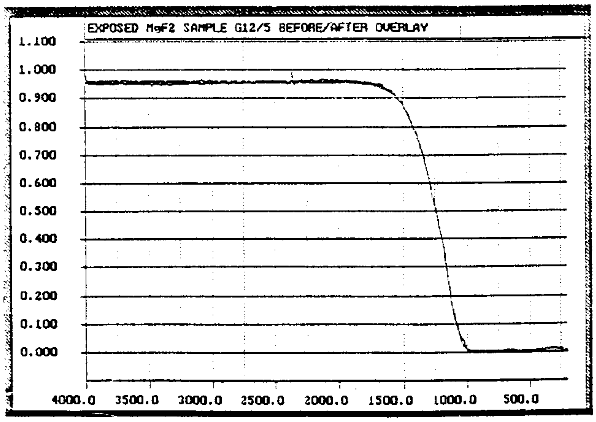

Fig. 8 Uncoated MgF_2 sample.
Overlay before/after exposure.

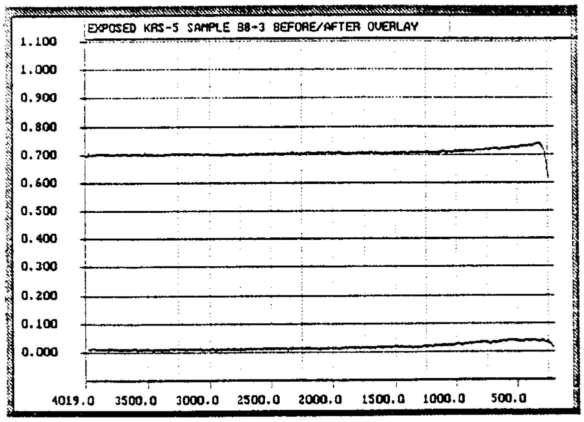

Fig. 9 Uncoated KRS-5 sample.
Overlay before/after exposure.