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MEDET EXPERIMENT ON-BOARD ISS: PRELIMINARY RESULTS OF MATERIALS DEGRADATION ON THE SPECTROMETER SUB-UNIT

V. Rejsek-Riba [1], V. Inguimbert [1], M.Dinguirard[1], S. Duzellier[1], C. Pons[1], M. Crepel[1], D. Falguère[1], A.P. Tighe[2], M.R.J. van Eesbeek[2]

[1] ONERA/DESP, 2 Av. E. Belin 31055 Toulouse Cedex 4, France
e-mail : virginie.rejsek@onera.fr, Phone N°: (33) 562 25 27 41, Fax N°: (33) 562 25 25 69

[2] Materials, Physics and Chemistry Section, ESA ESTEC, Keplerlaan 1, 2200 AG Noordwijk, NL

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ABSTRACT

The MEDET experiment on-board ISS since February 2008, is an opportunity to follow in real time the phenomena degradation of a large set of materials used normally for space applications (20 different materials). The thermo-optical properties studied by our team during the mission were measured by UV and IR spectrometers.
An UV spectra preliminary data enabled to highlight the two degradation phenomena expected for a such mission namely : yellowing of some materials due to UV exposure and the decrease of thicknesses for others like polymeric films due to AO erosion.

1. INTRODUCTION

MEDET is a material experiment on board EuTEF/Columbus. It is a fruitful collaboration between ONERA, ESA, CNES and the University of Southampton. It combines seven sub-experiments devoted to the combined measure of the radiative space environment in low earth orbit (LEO) and its associated effects on materials. It allows for real time characterization of the local ISS environment and material degradation such as thermal coatings, polymers for inflatable structures....
The spectrometer experiment is part of MEDET and has been designed for surveying the degradation with time of several types of optical windows (including synthetic ultra-pure SiO2 and other radiation stable materials). The optical spectral transmission of the samples are measured by a system involving quartz optical fibres, two miniature spectrometer modules (to cover the solar spectrum from 200 to 1000 nm) and two illumination sensors. The materials to be tested are placed on a rotating wheel, containing 24 apertures and the sun is used as the light source, so that measurements are allowed only when the light detected by the illumination sensors is in a ± 40° acceptance angle (see figure1 and figure 2). An encoder system, consisting of photodiodes positioned under the wheel, is used to identify the location of the wheel with respect to the fibre optic at any given instant (see Fig. 1). Sun light passing through the sample is collected by a quartz diffuser to illuminate as homogeneously as possible a fibre optics strand. Half of the collected light is sent by UV fibres to a so-called "UV spectrometer" sensitive to the range 200-700 nm and the second half to a so-called "IR spectrometer" sensitive in the range 400-1000 nm.
Each miniature spectrometer modules consists of a grating, optics and a CCD detector. These are all rigidly housed within the same case. The module is based on a standard laboratory device (Zeiss MMS) which has been adapted for space flight use. However, in order to be operated safely on MEDET, which is an external payload, the miniature spectrometer modules are mounted in a pressurized cylinder. For redundancy, 2 sets of pressurized cylinders (with 2 spectrometers in each) are mounted behind the filter wheel. Both are operating simultaneously allowing each sample to be measured twice at each wheel rotation.

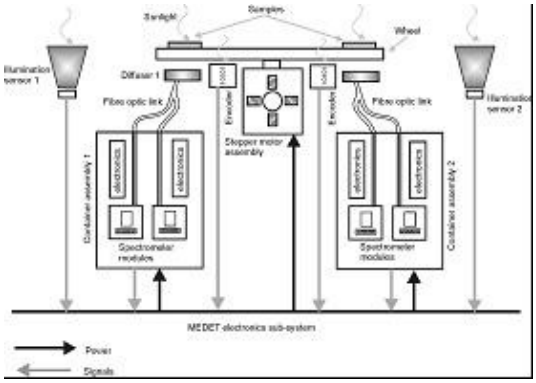


Fig.1. Schematic of Spectrometer experiment

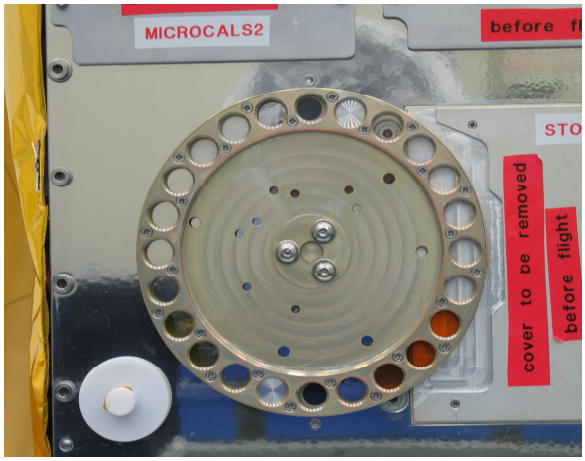


Fig.2. Sample flight wheel

This is the first time such a diverse collection of materials degradation experiments (see table 1) have been measured in real time in LEO.

Table 1: Selection of flight samples

Use	Material
Flexible and rigid thermal control coatings	Ex : RSF, RSR
Reference for coatings	Quartz
Solar cell cover glass adhesives	Ex : RTV-S690 and S695
Thermal control foils	Ex : Upilex S, Kapton HN
Atomic oxygen protection coatings	Kapton HN + different kinds of MAPATOX
Multilayer polymeric film for inflatable structures	Ex : KF01C01
Polymeric film for inflatable structures	PEN

IR and UV spectrometers have been acquiring in-orbit data of each material once or twice per day (depending on illumination conditions) since MEDET commissioning (i.e. 18 months in LEO exposure), except for some small data gaps due to power outages at MEDET/EUTEF levels. The synergetic effects of Ultraviolet and Atomic Oxygen degradations will be evaluated for each material in real time.
This work was focused on the evolution of UV spectra of the different materials in order to highlight degradation phenomena like yellowing or erosion.
IR analysis will be proceeded later.

2. EXPERIMENTAL DATA

At the beginning, the main part of the study was to sort the huge quantity of data from spectrometers in order to compare and highlight the spectra evolution of the different materials investigated versus condition exposure.

Nowadays the data obtained are “raw” in nature compared to data provided by a classic spectrometer where spectra are calculated accounting for a reference sample.
However two open positions in the wheel have been chosen to be measured as reference (no sample i.e. measurements at these positions do not evolve with time). The first idea was to divide sample spectra by open position ones to obtain relative and normalized data. Because of the measurement facility complexity, the correlation method to transform raw data in relative data versus a reference is not straightforward (different illumination conditions and light incidences). For this reason, in a first stage the data and observations reported here mainly deal with “raw” data spectra.

A second way to compare material spectra at MEDET level is based on the values measured by the 2 illumination sensors (IS). Indeed, for each measurement carried out corresponds a value of level of illumination. It means that for a same illumination sensor value and for the same illumination exposure, open position spectra should be identical. A part of this work consisted in sorting data with the same value of IS. This second approach also appeared difficult to apply because of light reflexions on MEDET surfaces (or ISS) and the presence of shadowing due to nearby EuTEF equipment (handle). However at that time, several configurations and periods of acquisition were determined for which open position spectra are perfectly identical for a same value of IS (Fig. 3). We will focus here on two dates with comparable spectra of the different materials;: 05/01/2008 and 06/25/2009.

Since MEDET commissioning, accumulation time of spectrometer has been changed by telecommands between 256 ms and 60ms, because of saturation of the signals met by polymeric thin film samples.

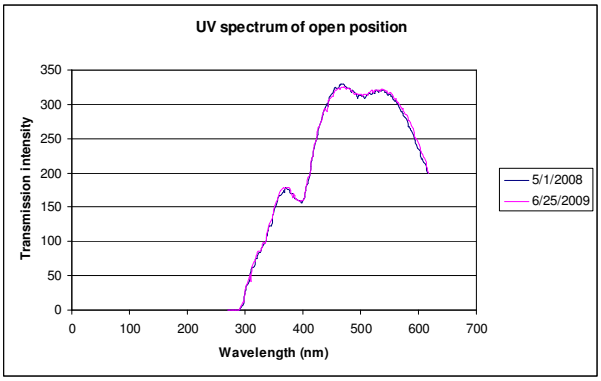


Fig.3. UV spectrum of open position for the same IS (IS=8.62V) in May 2008 and June 2009.

Some UV analyses have been performed on ground before the mission onto the different materials.
The measurement system is composed of a spectrophotometer Perkin Elmer Lambda 900 connected to an integrating sphere in air (at ONERA/DESP). The calibration curve was made with Spectralon SRS-99-010 standard. Transmission spectra in the range 250-2500 nm were performed.
Parameters : Data interval = 1nm, Lamp change = 319.2 nm.

Lastly, it is important to mention here that MEDET experiments (and then Spectrometer) are mostly oriented in RAM on the ISS orbit.

3. TEST RESULTS AND DISCUSSION

Sample results are disclosed below sorted by material nature (Table 2).

Table 2: Selection of flight samples studied.

Material nature	thickness	Sample nature
Polyimide (Kapton® HN)	75 µm	Film Sheldhal
Polyimide with AO protection (Kapton® HN + Mapatox K)	95µm	Film Sheldhal
Polyimide (Upilex® S)	25µm	Film
Polyethylene naphthalate (PEN)	2µm +2mm	2µm film between two quartz
Quartz	2mm	quartz
Thermal control coating on quartz (silicone resin based on polydimethylsiloxane)	2.12mm	Quartz with coating

In the following we focus on the main striking results. A deeper analysis is necessary to obtain meaningful results on the evolution of degradation of every materials.

3.1 Kapton HN film without atomic oxygen protection

The evolution of UV spectra between the May 2008 and June 2009 reveals the presence of degradation on Kapton HN (Fig.4).

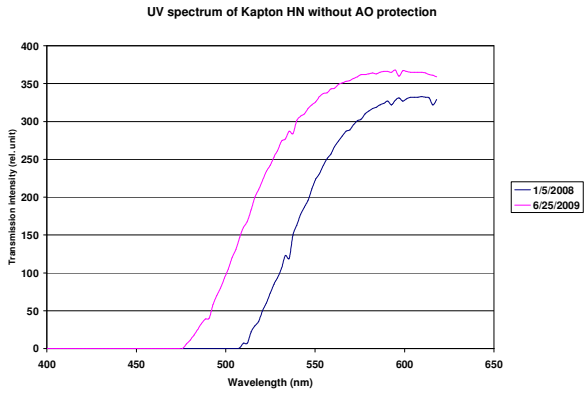


Fig.4. Data in Flight - UV spectra of Kapton HN without AO protection.

The shift of UV cut off towards smaller wavelengths and the increase of the overall transmission intensity characterize a decrease of the thickness of the material. After 15 months in LEO environment, Kapton HN film suffered of AO erosion principally.
The values of UV cut off presented in Table 3 have a part of incertitude because of the use of raw data for the calculation.

Table 3: Cut off data for Kapton HN in flight between 05/01/2008 and 06/25/2009.

Date in flight	Cut off (at half intensity)
05/01/2008	540µm
06/25/2009	514µm

To determine the decrease of the film thickness, an UV spectrophotometry study was performed on ground onto the Kapton HN film of different thicknesses.

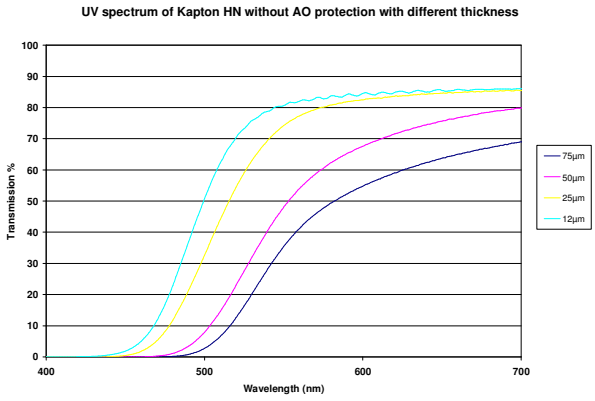


Fig.5. Data in ground - UV spectra of Kapton HN without AO protection (Perkin Elmer Lambda 900).

The evolution of the cut off level versus thickness film was calculated from ground data (Table 4).

Table 4: Data of cut off for different Kapton HN thicknesses (ground measurements).

Kapton thickness	Cut off (at half intensity)
75 µm	558 nm
50 µm	543 nm
25 µm	509 nm
12 µm	494 nm

The first in-flight exploitable data for this sample are given for 05/01/2008 (no information was available from the beginning of the mission March 2008). Consequently the interpretation concerning the kinetic of thickness decrease is still difficult and incomplete.
Referring to our data of cut off at ground and in flight conditions, the current thickness of Kapton HN can be assumed to be around 25µm. The loss in thickness would be of 50µm caused by AO erosion in 15 months flight in RAM position. Dever[1] and al and De Groh[2] and al determined the thickness erosion of Kapton HN at 60µm/year on passive samples on MISSE 2. Our data are in agreement with this result.
Moreover the preliminary results obtained by Tighe and al[3] onto carbon coated QCM sensors (Quartz Crystal Micro-balances MEDET experiment), enabled to calculate an AO fluence in agreement with our result.
The UV effect can not be estimated and however it must be taken in consideration.

3.2 Kapton HN film with atomic oxygen protection

UV spectra of Kapton HN with Atomic Oxygen protection is given Fig.6. Kapton HN protected spectra reveals a stability of UV cut off data compared to neat Kapton HN. The material is not degraded by AO erosion. A decrease of transmission intensity is noticed but not explained at this time.

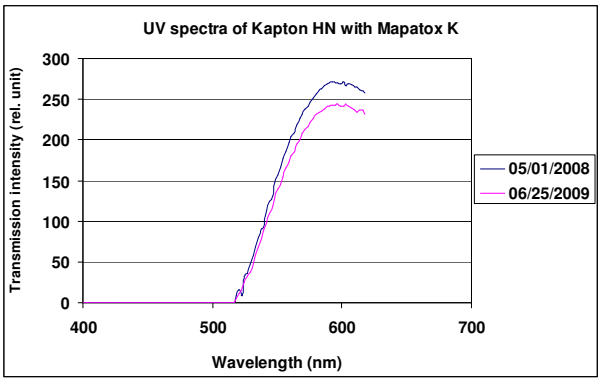


Fig.6. Data spectra of Kapton HN with Mapatox K.

The efficiency of the coating protection is highlighted by the analysis.

3.3 Upilex film without atomic oxygen protection : 25µm

The Upilex film is the thinner film studied here. The analysis of UV spectra between the two chosen dates reveals an evolution of the shape of the sample spectra (Fig.7). The "final" curve has the same shape as the open position spectrum.
Similarly to Kapton without protection, polyimide suffers from AO erosion. To date, the Upilex 25µm sample has been completely eroded by AO exposure.

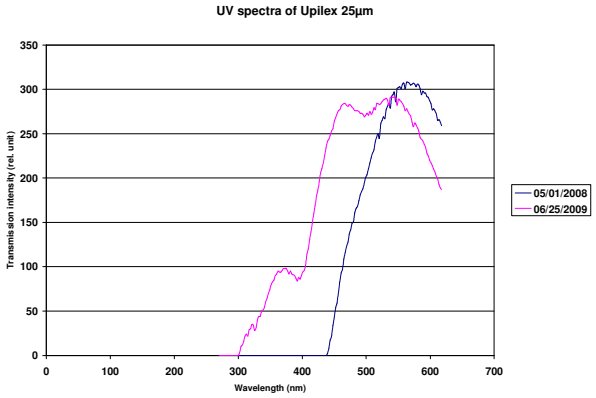
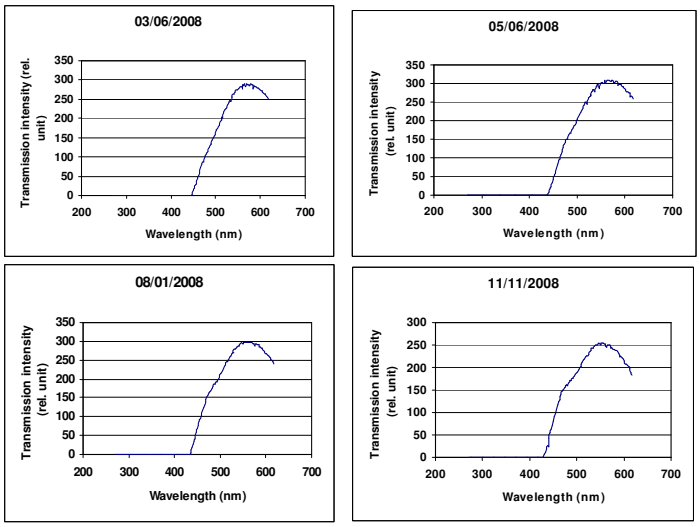


Fig.7. Data spectra of Upilex 25µm.

The evolution of the UV curves for Upilex was investigated in detail in order to determine kinetic of degradation. The spectra used are not comparable in intensity but they highlight the evolution of UV curves (Fig.8).



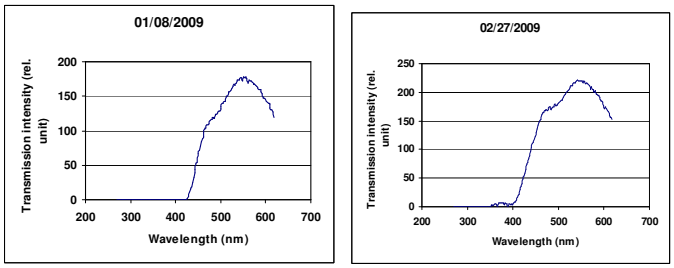


Fig.8. Data spectra of Upilex 25µm in real time.

The evolution of UV spectra shows that pronounced degradation occurred from August 2008 with the distortion of the curve. The spectrum obtained in February corresponds to the open position curve. In 11 months, the degradation of Upilex was complete and so the thickness loss is 25 µm. The works of Dever[1] and al and De Groh[2] and al confirm this result with a value of 18µm thickness loss per year, as well as Shimamura[4] who showed that in LEO Upilex thickness loss is around 28 µm/year.

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3.4 PEN film between two quartz

Investigations on a polymer film between two quartz allow for selecting the influence of one parameter namely UV exposure on the degradation of such a material. Quartz protects PEN against AO erosion.
The evolution of spectra between May 2008 and June 2009 (Fig.9) shows a shift of curves toward the higher wavelengths. The result is explained by a yellowing of the material under UV exposure. The absorptance value of the PEN film is now higher than at the beginning of the mission. Further analysis and correlation with ground are planned to quantify this effect.

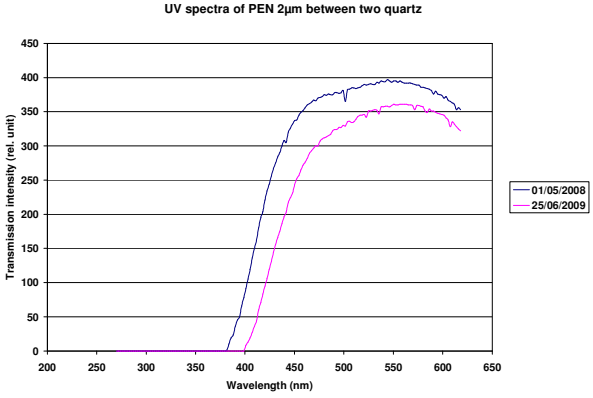

Fig.9. Data spectra of PEN film between two quartz.

3.5 Quartz

The stability of uncoated quartz under radiation is well-known[5]. The non-evolution of UV spectra here between the beginning and the end of the mission is expected.
However as shown on Fig. 10, a slight decrease is observed for the spectra amplitude. This decrease in transmission of the uncoated quartz could be attributed to contamination.

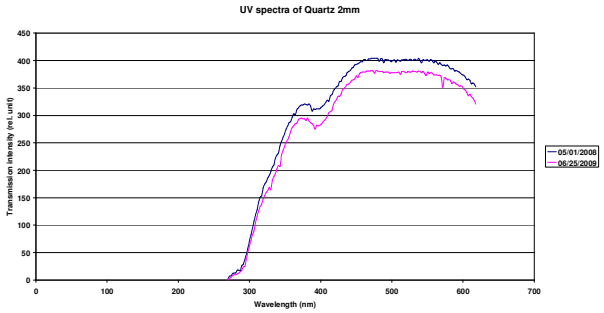

Fig.10. Data spectra of quartz 2mm.

At the return of MEDET and the samples, an UV analysis before and after cleaning quartz is planned to confirm our hypothesis.

3.6 Quartz with silicone resin

Different thermal control coatings based on silicone resin have been investigated in the mission. Only one result is presented in Fig. 11 because of the similar shape curves obtained for all of them.
A shift of UV spectrum toward higher wavelengths is noticed (Fig. 11). Compared to quartz results, we can conclude to coating degradation and more particularly to yellowing of the material.
The result confirms a predominance degradation due to UV but AO erosion occurs certainly at the same time.

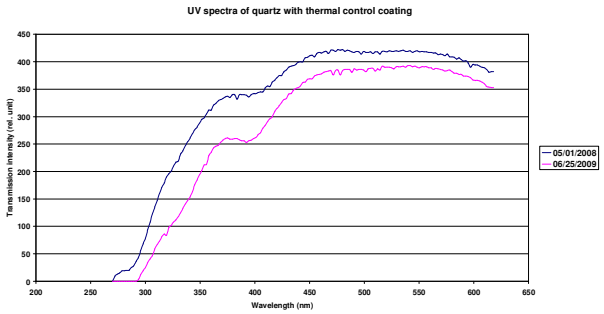

Fig.11. Data spectra of quartz with silicone resin thermal protection.

4. CONCLUSIONS

Preliminary results presented show a tendency of samples response evolution in real time for a part of materials studied. Erosion of polymeric film and yellowing of thermal coating have been observed and explained.
A deeper study of the UV and IR spectra obtained in flight conditions is necessary to propose a real quantification of the evolution of the degradation for our set of materials (per month for example.)

A further accomplishment will be the experiments' planned return to earth when the materials can be analyzed and a direct comparison can be made between the in-orbit results and the ground based results. Furthermore physico-chemical analyses will enable to assess in detail degradation phenomena.

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5. REFERENCES

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