

CHARACTERISATION OF TEFLON FEP (HST, LDEF) FOLLOWING LONG TERM EXPOSURE TO LEO

M. Fink^{a,*}, E. Semerad^a, G. Mozdzen^a, J. Wendrinsky^a,
G. Hulla^b, R. Kolm^b, I. Gebeshuber^b, H. Störi^b,
B. Dunn^c, M. v. Eesbeek^c, Th. Rohr^c, M. Moser^c

^a ARC Seibersdorf research GmbH, 2444 Seibersdorf, Austria

^b Vienna University of Technology, Wiedner Hauptstr. 8-10, 1040 Wien, Austria

^c ESA / ESTEC, 2200 AG Noordwijk, The Netherlands

ABSTRACT

The aging of Teflon[®] FEP (flourinated ethylene - propylene), an often used exterior spacecraft layer for thermal control in the low Earth orbit (LEO) environment, was determined. Therefore, changes of the morphology of the surface (light microscope), the topography of the surface (3d-profilometry, AFM), mechanical properties (AFM and micro – hardness) as well as the surface composition (ESCA, FTIR and XRD) with samples mounted on the Long Duration Exposure Facility (LDEF, 5.8 years in space) and with foils brought back to earth from the Hubble Space Telescope (HST, 3.6 and 8.25 years in space resp.) were performed.

The findings indicate that the chemical change during space exposure is marginal and for this reason hard to detect, but strong enough to cause mechanical embrittlement.

1 INTRODUCTION

The objective was to investigate the participation on the ageing of Teflon[®] FEP during space exposure.

Specifically following materials properties were characterised:

- variation of surface roughness,
- identification of surface species (contaminants, deposits etc.),
- identification of changes in general material properties between surface and bulk,
- investigation on embrittlement of materials,
- fractographic investigations on cracked or fractured surfaces,
- identification of chemical changes in materials down to the atomic bond.

Keywords: HST, LDEF, Teflon FEP, space weathering, material characterisation

* Corresponding author. Tel.: +43 50550 3384;
fax: +43 50550 3366; E-mail address:
markus.fink@arcs.ac.at

The **Long Duration Exposure Facility (LDEF)** was a cylindrically shaped free-flying long-term space exposure satellite. LDEF had 12 sides (rows 1-12) and space as well as earth facing ends (Fig. 1). LDEF was launched in April 1984 and retrieved in January, 1990 after 5.8 years in space.



Fig. 1. The Long Duration Exposure Facility (LDEF).

The general setup of the LDEF is shown in Fig. 2. Row 4 experienced the lowest AO fluence (9.32×10^4 atoms/cm²). Row 10 were placed beside the RAM direction and received an AO fluence of 8.17×10^{21} atoms/cm² [1].

The samples delivered from ESA were “LDEF Row10”, “LDEF Row4” and “LDEF Unexposed” which served as ground control sample for comparison.

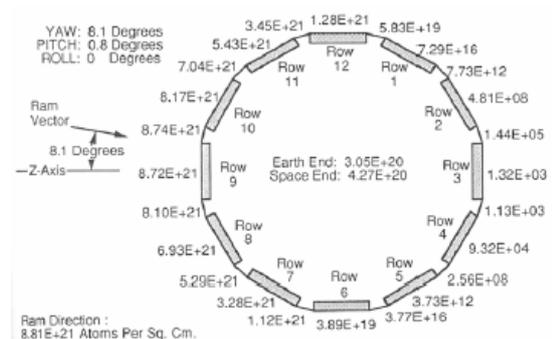


Fig. 2. AO – fluence on LDEF [1].

The LDEF foils consist of 127 μm FEP with on the rear side an Ag layer protected by Inconel and black Chemglaze 306. The Inconel/Adhesive coating of all specimens was removed before measurements. In the following the side facing to the Inconel/Adhesive is called “back side” of the specimens. The so called “front side” is the space facing side of the samples.

The **HST (Hubble Space Telescope)** (Fig. 3) was launched in April 1990. The telescope was designed to be serviced in space.

The first servicing mission was in December 1993. During this servicing mission the solar arrays were replaced with new arrays. One of the two replaced arrays was brought back to Earth. From this array the sample “STSA1 MLI” was taken (from the $-V2$ direction, s. yellow ring in Fig. 3, estimated equivalent sun hours are 20.056 and 6.26 solar facing and anti solar facing resp.) [2, 6].

In March 2002, the servicing mission “SM3B” took place. The samples “STSA2 MLI” (cracked sample, from the $-V2$ direction, s. red ring in Fig. 3, estimated equivalent sun hours: 45.95 and 14.33 solar and anti solar facing resp.) and “STSA2 Bellows” (blue ring in Fig. 3) were taken [6].

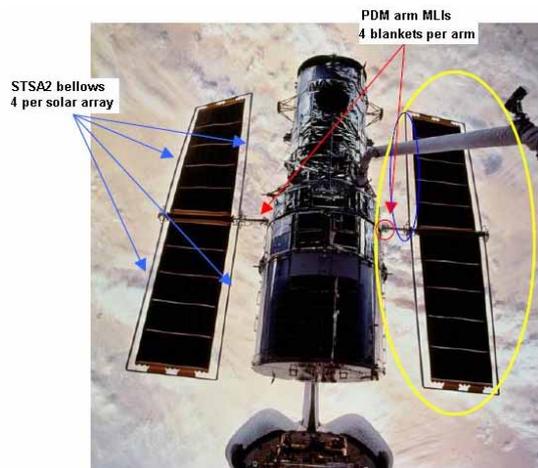


Fig. 3. The Hubble Space Telescope.

STSA MLI foils consist of 127 μm Teflon FEP, 40 μm Ag/Inconel, a glass fibre cloth impregnated with PTFE, 16 layers of double-sided aluminized kapton (50 μm) and another glass fibre cloth impregnated with PTFE [3]. From the 3 HST foils several specimens were taken from different areas. STSA2 Bellows has a thickness 52 μm .

2 EXPERIMENTAL DETAILS, RESULTS AND DISCUSSION

The tests included:

- Visual inspection (LO),
- Measurement of micro hardness,
- Topography by 3d profilometry,
- Surface analysis by AFM,

- Detailed surface analysis by ESCA,
- FTIR - analysis,
- Analysis of the crystallinity (XRD)

Micro - hardness and light microscope

The micro - hardness has been measured by ULTRA MICRO - DUROMAT 4000 (Ultra Micro - Hardness Tester with LM - MeF3) according to Vickers - Method (load: 1 pond, hold time: 20 s). The Ultra Micro - Hardness Tester permits the application in a force range of 0.05 p to 200 p.

As the applied test forces are extremely small the hardness testing of thin layers, fibres, sintered materials, as well as the non-destructive testing of integrated circuits can be determined by this device. The applied force was 1 pond (= HV0.001). The hardness tester has the shape of an objective and can be used with Reichert microscope MeF3.

Measurements have been performed on the front side of the specimens. As the used method is normally not applied for plastic materials, the obtained values shall be regarded only qualitatively, just for comparison of changes happened on the material surface during the exposure. Nevertheless, the indentation deepness (ca. 1/7 of the indentation diameter) could be inspected lower as for metallic and ceramic specimens.

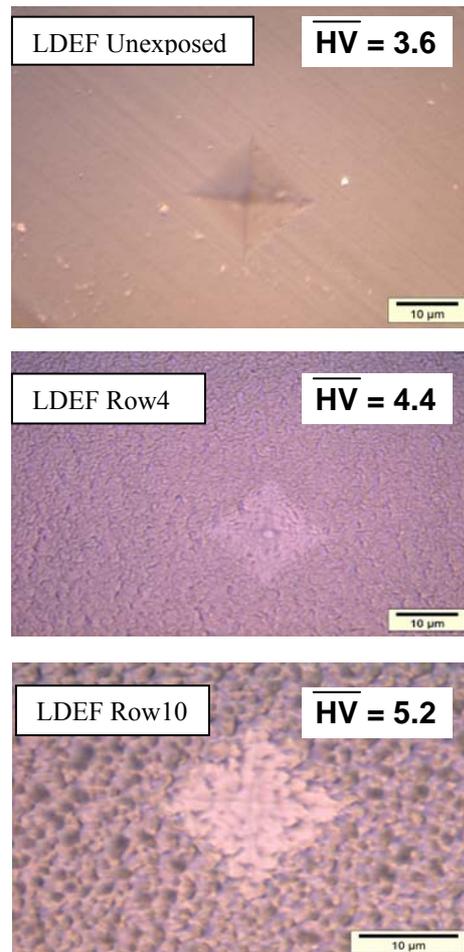


Fig. 4. Pictures and qualitative micro – hardness values of the LDEF – samples.

In the case of the LDEF – samples, a difference of ca. 30 % between the hardness of the unexposed and exposed specimens (Row10) could be noticed. The micro – hardness of Row4 is in between. With the light optical microscope it was found that the structure of the unexposed specimen is rather smooth, Row4 has a rougher and harder surface and Row10 has the roughest and hardest one (Fig. 4). A higher roughness of the surface causes a higher impreciseness when reading off the micro-hardness tester.

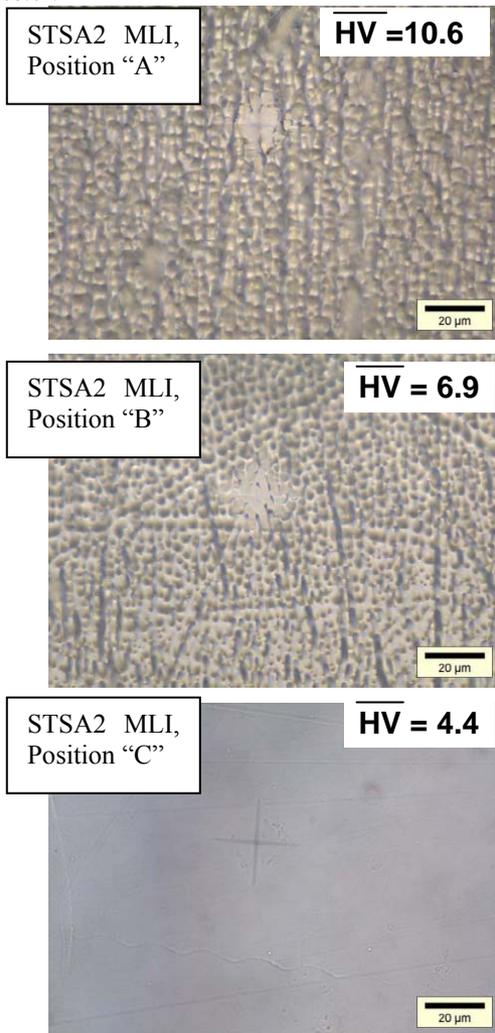


Fig. 5. Pictures and qualitative micro – hardness values of several positions of STSA2 MLI.

The micro – hardness of STSA Bellows wasn't detectable. The impression of the indenter couldn't be clearly identified on the rough surface of the thin specimen.

Comparing the micro – hardness of several positions of STSA1 MLI, nearly no variation could be detected (HV = 3.98 – 4.4).

In Fig. 5 pictures and micro-hardness values of the front side of the 3 positions of STSA2 MLI are shown. It varies very strong from one area to another. In "C", far away from the crack, the surface is very smooth and the micro – hardness is comparable with the values of STSA1 MLI. In

position "B", a few cm away from the cracked area, an increased micro – hardness can be noticed. Directly beside the cracked area ("A") the micro – hardness is dramatically increased (more than 200 % compared with "C"). In "B" and particularly in position "A" the surface is clearly rougher.

3d – profilometry

The roughness of the surface has been determined by "WYKO Surface Profilers" applied VSI-Mode. VSI (Verticals Scanning Interferometer) is a digital interferometer that vertically scans through focus. The fringe modulation corresponding to each plane of focus is recorded by the detector and transferred to the system's computer. The vertical resolution of the VDI-mode is better than 3 nm. The measurements have been performed by using an objective "x20" which allows scanning an area size of 309 µm x 235 µm. The roughness parameter Ra (roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane) of LDEF Unexposed is very low (Ra = 26 nm) what indicates very smooth surface. LDEF Row4 has been characterised by slightly higher roughness (Ra = 83 nm) than unexposed foil. Dramatically increasing of the roughness of LDEF Row10 (Ra = 470 nm) shows very clearly an effect of exposure on material surface (Fig. 6).

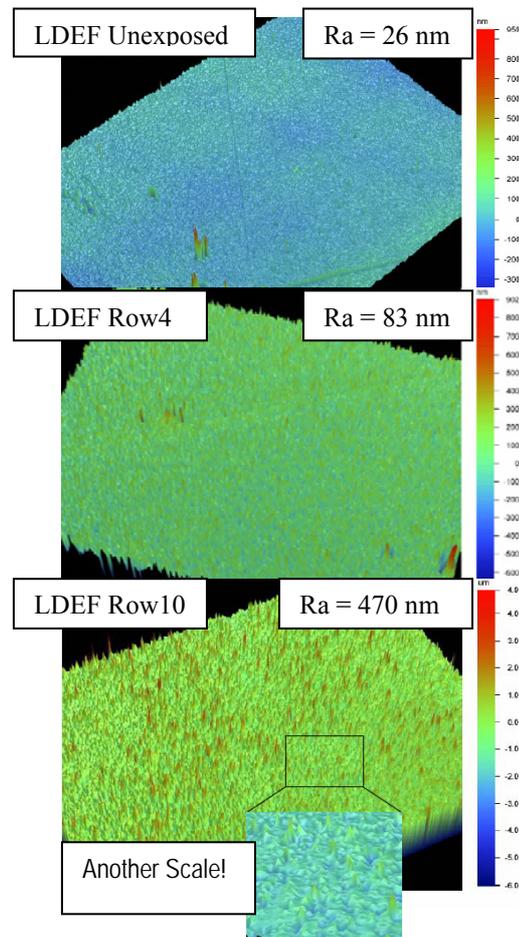


Fig. 6. Pictures and according roughness taken with the 3d profilometer.

This effect also can be seen with STSA2 Bellows. On its back side the roughness is 57 nm, on its front side it is 116 nm.

In the case of STSA1 MLI the roughness is about the same all over the foil.

STSA2 MLI shows a dramatically increased roughness in the near ($R_a = 555$ nm) and beside ($R_a = 635$ nm) the crack compared to an area far away from the disturbed zone ($R_a = 29$ nm).

AFM

Deflection-distance curves were performed with an Asylum Research Molecular Force Probe ambient AFM (MFP-3D, Asylum Research, Santa Barbara, CA). The experiments took place in doubly distilled water, to minimize water meniscus forces. The AFM cantilever was pressed into the surface with a constant velocity of 5.95 micrometers/second until the deflection reached a few hundreds of nanometers (max. 1000 nm) (see red curve in Fig. 7). Then the AFM cantilever was lifted back. Sometimes, due to adhesive forces the cantilever gets caught and thus negative forces result until it detaches completely from the film (see blue curve in

Fig. 7). On the ordinate of Fig. 7 the deflection y from $F=k*y$ is diagrammed. F is the force and k the spring constant of the cantilever. The cantilevers used had spring constants of 42 N/m (for front-back measurements) and 0.03 N/m (for cross section measurements). The slope of the deflection-distance curve was used to derive information about the hardness of the samples (steeper slope means higher hardness). The differences in sample suppleness can readily be seen right after engaging of the cantilever: the harder the sample, the more the cantilever is bent immediately after contacting the sample.

As compared to the slope of the unexposed foil, the slope changes in LDEF Row4 (front side) by a factor of 1.69, and in its back side by a factor of 0.66.

As compared to the unexposed foil, the slope changes in LDEF Row10 (front side) by a factor of 1.32, and in its back side by a factor of 0.59.

So, a clear trend is increased hardness on the front side of LDEF Row4 and LDEF Row10, as compared to the back side of these films and as compared to LDEF unexposed. The back sides of LDEF Row4 and LDEF Row10 show decreased hardness as compared to LDEF Unexposed.

The results were confirmed by the cross-section profile deflection-distance curves which were obtained in steps of about 10 micrometers across the films. These results were reproducible throughout the samples.

The unexposed film shows in the cross-section profile the same hardness throughout the sample. Due to AFM measurements a damage of the foils can be located only in the first 10 μm of the front side of the samples.

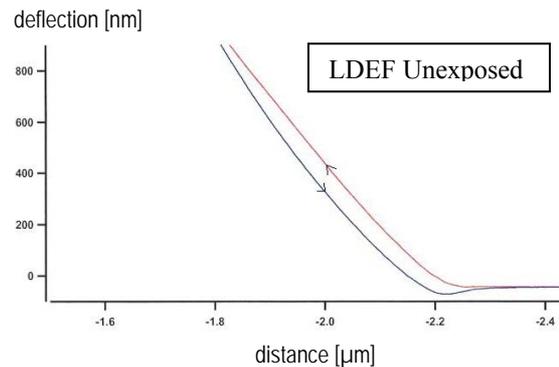


Fig. 7. Typical deflection – distance curve.

There are nearly no hardness differences between the front and back side of STSA2 Bellows. The reason could be the very low thickness of this film. STSA1 MLI showed on several positions increased hardness on the front side. However, the hardness differences depend very much on the site of measurement.

Also STSA2 MLI was softer on the back side than on the exposed side.

ESCA

Samples of 1 by 1 cm square were cut from the films and were fixed to a flat sample holder using flat springs. During the investigation, samples were excited with $\text{Mg-K}\alpha$ -radiation. The power of the x-ray tube was 270 W. No x-ray monochromator is present in the Microlab MK III system used. After introduction into the vacuum system a quick alignment of the samples had to be performed to make sure, the sample is illuminated by the x-ray source and is in the field of view of the detector. This was done by moving the sample to maximise the signal.

This explains the slightly different absolute intensities of different samples. In order to achieve high quality low noise spectra, at first long exposure times have been used. Later embrittlement of the films after some irradiation was observed, indicating severe radiation damage during analysis (Fig. 8).

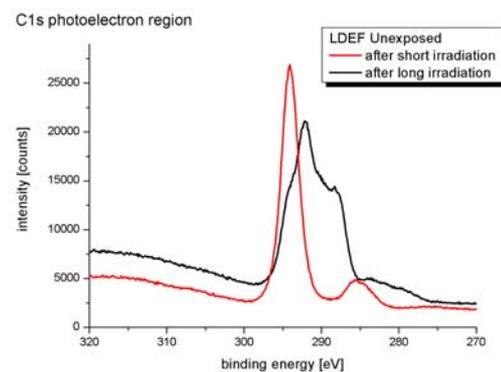


Fig. 8. ESCA carbon signal of LDEF Unexposed after a few minutes and after a few hours of x-ray irradiation resp.

To get a first understanding of this phenomenon, a LDEF Unexposed sample was exposed to the radiation of the x-ray source under normal analysis conditions. In a repeated cycle, the carbon spectrum was recorded for 3 minutes, thereafter the fluorine spectrum was recorded for 3 minutes and thereafter a waiting time of 4 minutes was introduced. Thus, the fluorine and carbon signals were recorded every 10 minutes under continuous irradiation. This was repeated for 20 cycles, covering 200 minutes. Afterwards the data reduction procedure described later was applied, fitting the carbon signal to 5 peaks and the fluorine signal to one.

Results were compared to Beamson and Briggs [4], where the degradation of a variety of polymer films under irradiation is compared. The relevant quantity is the ratio of the peak areas of fluorine to carbon. This ratio is normalised to the ratio obtained from the first measurement. The quantity plotted in Fig. 9 together with data from Beamson and Briggs is therefore $(F/C)/(F_0/C_0) \cdot 100$. F and C denote the respective peak areas, while the index 0 refers to the initial measurement. As Beamson and Briggs used a power of 1400 W for the x-ray source during their experiments, the time axis of the present data was multiplied by a factor of 270/1400. It has however to be noted, that the geometry of both systems might be different in a way, which cannot readily be accounted for. Therefore, we cannot conclude, that the LDEF film is vastly more sensitive to the x-rays than the polymers investigated by Beamson and Briggs.

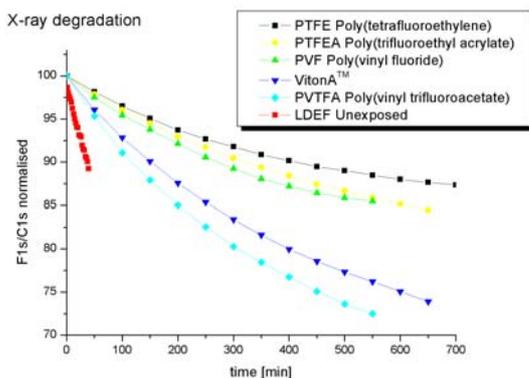


Fig. 9. Degradation of various fluorine containing polymers.

In order to characterise the radiation damage during analysis a peak fit by superposition of several Gauss-Lorentz peaks was made [5]. Peak 1 is the largest peak (the bigger one of the red curve in Fig. 8). As this peak always stays the biggest one, it was used as an energy reference to compensate for minor shifts of the energy scale. The coarse charging correction is performed by assuming an energy of 689.67 eV for the fluorine peak. According to the assumed teflon-like structure of the samples, it is assigned to carbon in CF₂. Peak 2

is shifted roughly 2 eV towards higher binding energies and is most likely carbon in CF₃. All other peaks are located on the lower binding energy side of peak 1. The locations are -2 eV for peak 3 (most likely carbon in CF), -4.5 eV for peak 4 (tentatively assigned to carbon with four fluorinated carbon neighbours) and -7.5 eV for peak 5. Peak 5 is assigned to hydrocarbon impurities on the surface, usually assigned an absolute binding energy of 285.0 eV. The binding energy of peak 1 is thus 292.0 eV. All the energies are in fair agreement with the literature [4]. Fig. 10 shows a typical peak fit with the different peaks as listed above. As the literature values are recorded on material different from the one investigated here, minor deviations between the energies mentioned and the literature values of [4] are to be expected.

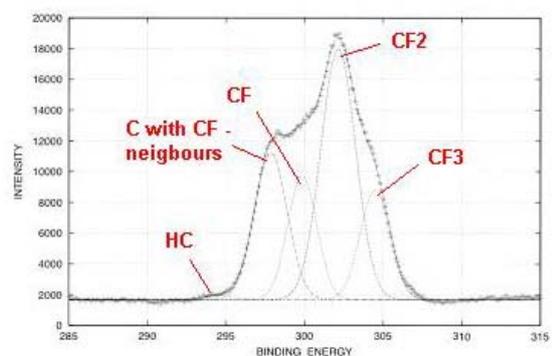


Fig. 10. Peak fit for the carbon peak of the LDEF-Unexposed sample. The largest peak is CF₂.

Fig. 11 gives an overview of the change of the shape of the carbon peak due to radiation damage. The quantities shown are the relative abundances of the different carbon peaks in the overall carbon peak. The data are from an experimental run equivalent to the one of Fig. 9.

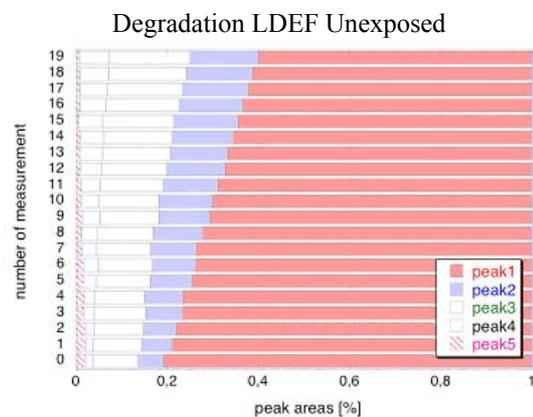


Fig. 11. Change of shape of the carbon peak with radiation damage increasing from bottom to top.

In general, only the element carbon, fluorine and oxygen were detected on the LDEF samples. Oxygen was in general only present in trace amounts. An exception from this statement is the sample Row 4 (front side), where there was a more intense oxygen signal and where also silicon was detected. On closer inspection, this was interpreted as SiO₂. This lead also to a peculiar shape of the carbon peak. Possibly the SiO – peaks are pointing to an oxidised siloxane contamination [7], but they are too small for detailed analyses.

With STSA samples generally the only elements detected were carbon and fluorine with traces of oxygen. There are however some exceptions. STSA1 MLI had a very thin film of SiO₂ on the surface. In this case the shape of the carbon peak was changed considerably and an additional carbon peak needed to be introduced into the peak fitting procedure. This peak was identified as carbon in SiC. Maybe this is also pointing to a siloxane contamination, like LDEF Row4 at its exposed side [7].

A typical overview spectrum is shown in Fig. 12.

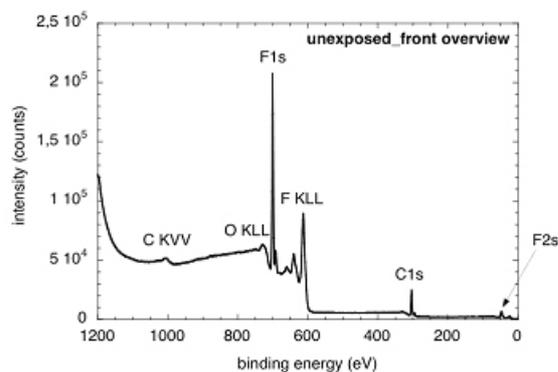


Fig. 12. Typical ESCA overview spectrum.

In addition to the overview spectra, detailed spectra have been recorded for the C1s region. The ratio of all subpeak areas of the C - peak to the peak area of CF₂ is a good standard to characterise the radiation damage.

The carbon peak was fitted as sum of the peaks described above. A 6th peak, accounting for SiC, was only used for the samples LDEF Row4 and STSA1 MLI. Fig. 13 and Fig. 14 show a graphical representation of fitting results.

In order to establish depth profiles of the films, the film samples were abraded using a Struers Accutom 50 precision grinding machine with a specially shaped grinding wheel covered with diamonds. Films have been attached to a glass slide using double sticky tape and mounted on the suction sample holder. The positioning accuracy of the sample holder with respect to the wheel is 5 µm. After the abrasion process, samples were rinsed with distilled water.

This method was tested with LDEF Unexposed. The sample was abraded and no changes could be

observed, specially no oxygen or diamond incorporation. Thus, it can be assumed that the usage of the grinding wheel hasn't an effect on the structure of the samples.

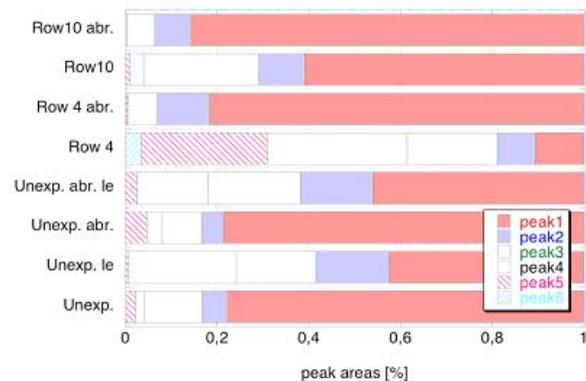


Fig. 13. LDEF-samples. Bargraphs for 10 µm abraded (“abr.”) and original samples, only front side. “le” means long exposure to x-rays. All other samples were exposed for a few minutes only during analysis.

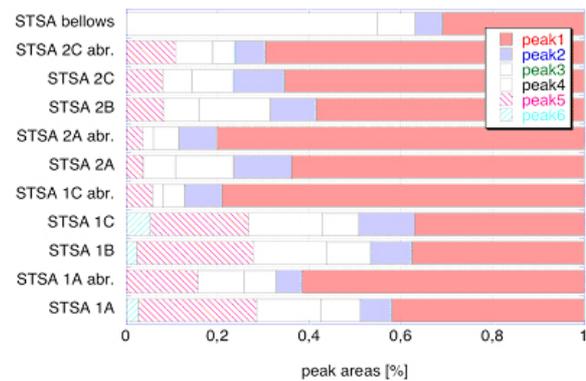


Fig. 14. STSA samples. Bargraphs for 10 µm abraded (“abr.”) and original samples on several positions (“A, B, C” of MLI samples), only front side.

As seen from abraded samples, the depth of the damage induced is always less than 10 µm. The changes of the structure of the samples induced by irradiation during prolonged analysis are in some cases much more severe than the changes induced by irradiation in space.

FTIR

The spectra were gained by a Bruker Equinox 55 FTIR – spectrometer equipped with a Golden Gate ATR. By this device the sample surface as the reflecting area is brought into a tight contact with the surface of a diamond crystal (2 x 2 mm²). The IR - light is passing through the diamond, reflected at the sample surface which thus is absorbing parts of the spectra resulting in bands related to certain chemical bonds.

With ATR a region of about 1 μm from the surface into the bulk could be investigated depending on the IR - transparency of the material.

The spectra gained are rather clear and showed some relationship with that of PTFE, based on the main grouping C-F bands according to FEP at 1202 and 1147 cm^{-1} , others being at 775, 638, 554 and 511 cm^{-1} , which can also be found with PTFE. More bands are found at 982, 749 and 720 cm^{-1} . When organic compounds are degraded by heat and/or atomic oxygen double bonds and carbon-oxygen compounds should be found. Therefore the bands related to C-O, C=O, C=C and O-H would be expected, C-O - bands being located at about 1200, C=O - bands at 1650 - 1800, with halogens at 1750, C=C at about 890 or about 1650 and O-H between 1000 and 1210, COH at 1000 - 1200 cm^{-1} , the definite position depending on the chemical environment. Among these bands the C=O - band (carbonyl group) is usually rather reliable.

There could be found 2 bands at 1735 and 1718 cm^{-1} resp., the heights of which were normalized by the height of the band at 1793 cm^{-1} to enable a comparison among the samples.

Another band – at 737 cm^{-1} – which might refer to a double bond, was also investigated with STSA - samples.

Investigation of three bands according to the C=O and C-O bonds with the LDEF – specimens indicate an increased amount of Carbonyl- and C-O – bonds and thus a stronger chemical degradation of the surface of the specimens LDEF Row4 and LDEF Row10. the so-called back-side of LDEF Row4 show an about fourfold amount of carbonyl bonds compared to the front side, but a much lower amount of C-O bonds (Tab. 1).

Sample	Normalized Height of Band (Ref. Band: 1793 cm^{-1})				Normalized Height of Band (Ref. Band: 983 cm^{-1})				Ref. Band 983 cm^{-1}	
	1735 cm^{-1} (C=O)		1718 cm^{-1} (C=O)		1735 cm^{-1} (C=O)		1718 cm^{-1} (C=O)		1030 cm^{-1} (C-O)	
	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2
Unexp. front:	6.16	7.14	7.86	7.54	0.46	0.59	0.58	0.62	0	0
Row4	11.7	17.43	13.03	16.2	0.85	3.08	0.94	2.86	0	4.45
Row10	14.7	12.89	12.87	10.1	1.23	1	1.07	0.79	0	0.98
back:	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2	Pos. 1	Pos. 2
Unexp. back:	6.92	7.94	8.31	9.88	0.58	0.67	0.7	0.83	0	0
Row4	49.78	48.27	54.85	51.76	3.65	4.01	4.02	4.3	0	0.43
Row10	17.36	23.91	13.85	16.76	1.62	1.36	1.3	0.96	0.2	0.11

Tab. 1. Normalized heights of bands of the LDEF - specimens.

In STSA1 MLI and STSA2 MLI, the peculiarity of the C=O - bands varies at several positions investigated. The C=O - bands of the specimen STSA2 Bellows reach about the same height as the bands of STSA2 MLI at the cracked position ("Pos. A"). All STSA - samples display much stronger C=O - bands than LDEF unexposed ("Unexp. front"). According to the band at 737 cm^{-1} STSA2

MLI display stronger bands than STSA1 MLI, the 737 cm^{-1} - band of the sample STSA2 Bellows is comparable to the values measured at the different positions (Pos. A, B, C) of the sample STSA1 MLI (Tab. 2).

Sample	Normalized Height of Band					
	Ref. Band: 1793 cm^{-1}			Ref. Band: 983 cm^{-1}		
	1735 cm^{-1}	1718 cm^{-1}	1735 cm^{-1}	1718 cm^{-1}	1030 cm^{-1}	737 cm^{-1}
	C=O	C=O	C=O	C=O	C-O	
factor	10	10	10	10	100	
STSA1 MLI						
Pos. A	2.66	2.27	1.12	0.96	3.19	5.33
Pos. B	6.54	5.89	2.59	2.33	6.2	6.54
Pos. C	2.41	1.68	1.23	0.86	0	2.98
STSA2 MLI						
Pos. A	3.03	2.58	1.2	1.02	3.05	8.9
Pos. B	4.04	3.44	1.48	1.26	3.95	11.12
Pos. C	2.05	1.47	1.18	0.85	0	2.52
STSA2 Bellows	3.13	2.27	1.72	1.25	4.99	6.73
Unexp. front	0.62	0.79	0.58	0.7	0	2.06

Tab. 2. Normalized heights of bands of the STSA – specimens at several positions.

Because of the results found with FTIR we have to follow that there are several chemical degradations on the foils.

XRD (Crystallinity)

FEP comprises a copolymer of PTFE with 50 to 90 % hexafluoropropylene. Its crystallinity can be adjusted by annealing processes (e.g. increased from 40 to 67 % by annealing at 210 $^{\circ}\text{C}$).

To decide if the differences concerning the crystallinity are significant XRD investigations were performed positioning 10 x 10 mm^2 specimens on a sample carrier of silicon.

The powder x-ray diffractometer applied was a Philips X'Pert PRO (XP-2) using $\text{CuK}\alpha$ - radiation, the voltage being 40 kV, the current 40 mA. The 2θ - range was between 2 - 40 $^{\circ}$ and 2 - 55 $^{\circ}$ resp.

All diffractogrammes display 3 diffraction maxima, one high - intensity one at 17.8 $^{\circ}$, and 2 small at 13.2 $^{\circ}$ and 36.1 $^{\circ}$. Additionally all diffractogrammes show a broad peak between 10 and 20 $^{\circ}$. This range between 8 and 22 $^{\circ}$ which is assumed to contain an amorphous and a crystalline maximum is evaluated by a simulation / integration method to gain the areas listed in Tab. 3.

LDEF Sample	Amorphous counts	Crystalline counts	Ratio crystalline / amorphous
front:			
Unexposed	19132	12126	0.634
Row4	19163	13194	0.689
Row10	18938	12083	0.638
back:			
Unexposed	19608	12246	0.625
Row4	19542	13261	0.679
Row10	19620	12344	0.629

Tab. 3. XRD - peaks integrated in the 8 - 22 $^{\circ}$ range.

All back sides display slightly higher intensities, but it won't be serious to interpret it as being significant.

If the ratio crystalline / amorphous is calculated differences between the samples LDEF Unexposed and LDEF Row10 on the one hand and LDEF Row4 on the other hand can be found postulating a higher degree of crystallinity with LDEF Row4. A ratio of 0.689 is significantly different from 0.63 because the difference is about 10 p.c.

The STSA – specimens were investigated in the same manner as the LDEF – specimens.

All diffractogrammes display 3 diffraction maxima, one with high intensity at 17.9°, and two small maxima at 31.2° and 36.1°. Additionally all diffractogrammes show a broad peak between 10 - 20°. This range between 10 and 22° which is assumed to contain an amorphous and a crystalline maximum is evaluated by a simulation / integration method to gain the areas listed in Tab. 4.

Sample STSA1 MLI displays the highest total intensity (31760), followed by STSA2 Bellows (20850) and STSA2 MLI. The specimens STSA1 MLI and STSA2 MLI display the same ratio crystalline / amorphous phase, whereas the sample STSA2 Bellows contains a slightly bigger amount of crystalline phase.

The absolute values of the crystallinity cannot be derived from this results.

Sample	Amorphous counts	Crystalline counts	Ratio crystalline / amorphous
STSA1 MLI	18290	13470	0.74
STSA2 MLI	9330	6660	0.71
STSA2 Bellows	11420	9430	0.83

Tab. 4. XRD-peaks integrated in the 10 - 22° range.

3 CONCLUSIONS

The embrittlement of FEP – foils mounted on HST and LDEF resp. during long term exposure in LEO was determined. Therefore, changes of the morphology of the surface (optical microscope), the topography of the surface (3d-profilometry, AFM), mechanical properties (AFM and micro-hardness) as well as the surface composition (ESCA, XRD, FTIR) were investigated.

With LDEF – specimens, a discrepancy between mechanical and chemical measurements could be stated. The sample LDEF Row10 which was mounted in RAM direction had the highest micro-hardness value and the highest roughness, followed by the WAKE direction mounted LDEF Row4 foil and LDEF Unexposed, which served as reference. The degradation of the carbon peak measured with ESCA, the ratio crystallinity/amorphous (XRD) as well as the content of carbonyl group of the foils (FTIR) pointed to another ranking of degradation. LDEF Row4 shows the strongest chemical degradation, followed by LDEF Row10 and LDEF

Unexposed. This discrepancy between the rankings can be explained as follows: Samples mounted in RAM – direction (LDEF Row10) had a permanent high number of reactants (AO: 8.17×10^{21} atoms/cm²), whereas WAKE – direction mounted samples (LDEF Row4) sustained fewer reactants (AO: 9.32×10^4 atoms/cm²). Sometimes low AO – flux can be more hazardous than high flux. For polymers an interplay between AO – induced contamination and AO – induced “cleaning” controls the level of contamination. At low AO fluxes, this interplay may lead to a maximal steady-state level of contamination. [7]

Generally, the depth of embrittlement is less than 10 µm.

Measurements taken with ESCA and XRD should be handled with care because the degradation induced by irradiation during prolonged analysis are in some cases much more severe than the changes induced by irradiation in space.

In general, the tested HST materials (3.6 and 8.25 years in space, resp.) are much more embrittled than the tested LDEF specimens (5.8 years in space).

With mechanical experiments, tests of morphology and topography, a very strong degradation of STSA2 Bellows and STSA2 MLI in the cracked area could be stated. Far away from the cracked position of STSA2 MLI, the embrittlement is comparable with STSA1 MLI. Testing the surface composition, this trend couldn't be clearly ascribed to chemical changes during space exposure. The chemical degradation of STSA2 Bellows is the highest one. STSA1 MLI and STSA2 MLI also show an advanced degradation.

The chemical change during space exposure is marginal and for this reason hard to detect, but strong enough to cause extensive mechanical embrittlement.

4 REFERENCES

- [1] LDEF – 69 Month in Space, NASA Conference Publication 3134 Part1, Kissimmee, Florida, 1991
- [2] Investigation of Teflon FEP Embrittlement on Spacecraft in Low Earth Orbit, K. de Groh and D. Smith, NASA Technical Memorandum 113153, 1997
- [3] Hubble Space Telescope Solar-Array Workshop, ESA Proceedings WPP-77, ESTEC, 1995
- [4] High Resolution XPS of Organic Polymers, Beamson and Briggs, the Scienta ESCA300 Database, 1992
- [5] W.S.M. Werner, Surf. Interf. Anal. 31(2001)141
- [6] M. Moser, Memo QM105-19/MM, Feb. 2005
- [7] Y. Haruvy, “Low Flux Atomic Oxygen: Can it be More Hazardous than High Flux? A Risk Assessment Study“, Ch. 1 in “Protection of Materials and Structures from the Low Earth Orbit Space Environment”, J.C. Kleiman, R.C. Tennyson, Eds., pp. 1-7, Kluwer (1999).