Dimensional Stability of Hot-embossed Polyimide at Elevated Temperatures

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ABSTRACT

Following the very successful mission to Venus, ESA (European Space Agency) is currently planning further missions to explore our inner solar system. As the environment closer to the sun will be harsher in terms of solar radiation (particle, UV etc.) and heat input such missions set a major challenge to materials and processes (M&P) and will require new and innovative design solutions. For instance, ESA’s fifth cornerstone mission, named Bepi Colombo, scheduled for launch to Mercury around 2016 will face about 14 kW/m² of solar irradiation at Mercury perihelion together with a high IR load coming from the planet. To support the mission for critical issues, ESA has initiated the so called CMT (Critical Materials Technology) Programme in 2001. Recently this programme addressed the challenge of a suitable high temperature spacer material which acts behind the outermost layers as a thermal shield and must keep its functional properties during the mission lifetime. One such material of choice is Polyimide film.

This paper outlines results of recent dimensional/mechanical testing in which the stability of a high temperature hot-embossed polyimide was assessed and is a follow on from previous investigations into similar thermal control materials where thermal analysis was used to assess the stability of the materials under high temperatures conditions [1, 2].

Testing has been carried out in a purged nitrogen atmosphere in the temperature range 275°C to 365°C for up to several hundreds of hours. Various materials analytical tools were used to assess the dimensional stability of the process and to understand the materials relaxation kinetics by examining the height of the polyimide materials with varying temperature. This was achieved using both contact height measurements (using vernier calipers) as well as non-contact analytical techniques (using white light chromatic profiling) to assess the influence of temperature on the physical/dimensional stability of polyimide and also to give predictions regarding the long term behaviour of these materials.

1. INTRODUCTION

The high temperature multi layer insulation (MLI) to be used for Bepi Colombo will experience service temperatures of up to approximately 350°C. The baseline design is to first use some ceramic fabrics, then a few layers of a metallic foil (titanium) followed by Upilex S foils (ultra high resistant polyimide foil from UBE Industries Ltd), coated on both sides with aluminium (using VDA process).

For separation of consecutive layers a thermally stable spacer material is needed. It is suggested to emboss the Upilex layers for separation. Standard embossing of Upilex was shown not to withstand high temperatures.

A new procedure called ‘hot-embossing’ was introduced by the manufacturer. Initial screening tests carried out have shown that this new hot-embossed material is dimensionally stable at higher temperatures compared to the standard embossing material.

The success criterion for dimensional stability has been defined as a height reduction of not more than 20%.

2. MATERIALS

The following materials were used for dimensional stability testing (using calipers) of multi layer insulation (MLI) materials:

20 layers of 7.5 μm VDA (Vapour deposited aluminium)/Upilex S / VDA hot-embossed interspersed with 21 layers of 25 μm Upilex S / VDA

The sample dimensions used were 125 mm X 125 mm

For measurements using White Light surface Profiling single foil samples of 7.5 μm VDA (Vapour deposited aluminium)/Upilex S / VDA hot-embossed were used.
3. EXPERIMENTAL

3.1 Procedure for Dimensional Stability Testing

The following procedure outlines the test set-up used for contact dimensional stability testing following Isothermal testing at 275°C, 300°C and 365°C respectively. Two PT100 probes were used in the oven to monitor temperature. The temperature deviation was +/− 5°C. Samples were thermally aged in a convection oven (type Nabertherm N60/65SHA) with a continuous dry-nitrogen gas purge.

To examine the effects of load, the samples have been tested either with or without a load (aluminium plate) on top of the samples during Isothermal exposure and also during material dimensional measurements.

The overall sample heights were measured prior to and after each thermal cycle using a level rule and a calipers with a resolution of 0.01 mm (see Fig. 2). Each measurement is the average of 4 orientations of each sample, which is rotated 90° between the measurements.

For thermal cycles at temperatures of 275°C and 300°C four MLI samples have been used. Two samples (sample 7 and 8) were initially used at the lower test temperature, whilst samples 2 and 6 were used as reference samples.

Following this all four samples were tested at the higher temperature of 300°C (see Fig. 1 and Table 1 for details). Four Isothermal cycles of 1, 10, 48 and 158 hours have been used resulting in an accumulated ageing duration of 0, 1, 11, 59 and 217 hours respectively.

For thermal cycles at 365°C four new samples were tested. Three samples (sample 3, 4 and 5) were thermally exposed whilst one sample (sample 2) has been used as a reference.

Similar to lower temperature testing four Isothermal cycles of 1, 10, 48 and 158 hours have been used resulting in an accumulated ageing duration of 0, 1, 11, 59 and 217 hours respectively.

Two samples (samples 3 and 5) were aged free from stress. One sample (sample 4) was aged with a load of 70 grams equally distributed over an area of 125 mm x 125 mm (see table 2). The height of that sample was also determined with the same load of 70 grams.

The height of the other two samples (samples 3 and 5) were determined with a small load of 40 grams (sample 3) and with a higher load of 150 grams (sample 5). The lower load of 40 grams was to reduce sample curling due to internal stresses (see Table 2 for test overview of samples).

<table>
<thead>
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<tbody>
<tr>
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<tr>
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<td>40</td>
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<td>70</td>
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Fig. 1 Test sample set-up for samples tested at 275°C and 300°C. Clockwise from top left: sample 2, sample 6, sample 8 and sample 7.

Fig. 2 Caliper sample measurement set-up
3.2 White Light Surface Profiling Measurements

In order to further measure the dimensional changes of this hot embossed polyimide material, white light chromatic surface profiling and dimensional changes were recorded.

Surface profiling was carried out using a Scantron Proscan 2000 white light chromatic profilometer (Scantron Industrial Products Ltd. - see Fig. 3). Dimensional changes were recorded prior to and after thermal exposure in order to further assess the degree of stress relaxation in this material.

This system used a Chromatic lens configuration which focuses a narrow beam of light onto a moving platform. For measurement purposes a chromatic sensor S38/3 (resolution 0.1 μm, 3mm measuring range and 10 μm nominal spot size) was chosen due to greater accuracy and greater measuring range. Due to the reflective nature of the samples a frequency of 1000 Hz was used.

In total nine (9) different hot-embossed dimpled areas were scanned using this technique prior to and following thermal exposure. For profiling measurements only single foil 7.5μm thick VDA Hot Embossed Upilex was used unlike the MLI stacking arrangement from the previous exposures.

The single Upilex foils were placed in aluminium holders during exposure and subsequently removed to the X-Y stage for profiling following thermal exposure.

Samples were held in place on a moving platform whilst the sensor head recorded the height variation measurements over the dimpled hot-embossed surface area. The system uses a scan step and raster motion to scan the profile area of 4 X 4 mm.

Markers were used on the samples such that the same dimples could be identified and compared with each other in order to determine the initial height, aspect ratio and dimple profile.

It is thought that after heat treatment a stress relaxation or expansion may occur which would change the initial shape of the dimpled hot embossed surface – resulting in a smoother and more flattened profile.

3.3 Height Profiling Measurements

During height and dimensional measurements using the ProScan 2000 White Light Profilometer the following terminology was used (see Fig. 11 and Fig. 12 for details):

- **Base Height** = Average Height of Base Plane of Embossed Foil
- **Centre Height** = Average Height of Centre Plane of Dimpled Structure
- **Dimple Height** = Average Height of dimple (Top) Formed at the top of Dimpled Structure
- **Dimple Diameter** = Diameter of dimple at base plane
- **Surface Roughness** = Average Linear Surface Roughness of Basal Plane
- **Surface Roughness** = Average Linear Surface Roughness of Top Plane
“Dimple height” was measured by subtracting the average “base height” from the average “dimple height”. Where a “dimple” feature is present on the sample, the dimple height was calculated by subtracting the average “base plane height” from the average “dimple height”.

4. RESULTS AND DISCUSSION

Fig. 4 and Fig. 5 show the relative height reductions for samples after 217 hours at 275°C and 300°C respectively. The effects of sample loading are seen as well as a maximum height reduction up to 11 hours exposure after which time no significant height reduction is found. At no stage did any average height measurements taken differ by more than 20% (success criteria) from the original value taken.

Fig. 4 Relative height reduction versus ageing time for MLI samples after exposure at 275°C

Fig. 5 Relative height reduction versus ageing time for MLI samples after exposure at 300°C

Fig. 6 shows the percentage relative height of samples 7 and 8 after exposure at 275°C and 300°C respectively. It can be seen that the relative height reduces from 100% to 92% after exposure at 275°C and from 91% to 85% following the complete exposure for sample 7 at 300°C. Sample 8 shows a greater % height reduction from 100% to 84% and from 83% to 69% for the second phase of the thermal exposure at 300°C for a further 217hrs.

Fig. 6 Relative height versus ageing time for MLI samples after exposure at 275°C and also 300°C

Fig. 7 shows the cumulative percentage relative height (relative to the initial sample height at 275°C) reductions for samples 7 (0g load) and 8 (70g load) after 275°C and 300°C respectively. It can be seen that prior thermal history does influence the overall height of the samples which result in samples reducing more than the 20% success criteria limit.

Fig. 7 Cumulative Relative height reduction versus ageing time for MLI samples after exposure at 275°C and also 300°C

Fig. 8 shows the percentage relative height reduction for samples 7 (0g load) and 8 (70g load) comparing exposure after phase 2 (275°C) and phase 3 (300°C) testing respectively.

The initial exposure at 275°C results in a relative height reduction of approximately 14% and 7.5%
(sample 8 and 7 respectively) and this reduction decreases to approximately 10% and 5% on further exposure at 300°C for 217hrs.

In order to better understand the relaxation kinetics behaviour of this new hot-embossed polyimide material, further testing was carried out at a higher temperature of 365°C. Table 3 shows the height measurements of the four MLI samples tested after exposure at 365°C (see table 2 for sample details), at the beginning of life (BOL) and after the 4 exposures.

It can be seen that the height of all three exposed samples reduced more than the success criterion of 20%. It may be argued that without any measurement weight, the criterion may not have been exceeded.

Table 3 Height measurements of all samples at 365°C and absolute and relative height changes at all inspection points. (Values always refer to the initial height; positive values indicate a decrease in height)

<table>
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<th>Sample ID</th>
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<th>exp1 1 h</th>
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<th>exp4 217 h</th>
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Fig. 9 shows the relative height change as a function of thermal ageing duration at 365°C. It can be seen that the height of all three samples reduced by more than the success criteria of 20% when tested at 365°C for 217hrs.

Fig. 10 shows the reduction of sample height as a function of the weight that is applied during the height measurement, which is 40 grams for sample 3 and 150 grams for sample 5.

The traces for the four exposures are extrapolated to zero weight. The figure shows that the 20% criterion is already exceeded after ageing for only 11 hours.
Surface Profiling and dimensional change measurements were taken on various samples. The results compared dimensional and stress relaxation phenomena in the Upilex S foil following long term thermal exposure at 365°C.

Table 4 and Table 5 below show the calibrated corrected dimple heights and % height change prior to testing and after 1 hour and 217 hours thermal exposure. The results show the overall average dimple height (i.e. height from average base plane to average top of dimple - see Fig. 11). The results show an overall reduction in the dimple height of the hot-embossed surface with thermal exposure duration.
As can be seen from Fig. 13, using white light profiling there is an overall reduction trend which can clearly be seen due to thermal exposure of the Upilex film. The overall height of the dimpled surface clearly reduces with time and thermal exposure leading to a more planar flattened surface. Considerable height reduction takes place after even short thermal exposure as seen after 1 hr. While values differ between those found using the mechanical calipers measuring technique, average values are somewhat similar in behaviour and the trends noticed are similar in appearance.

5. SUMMARY

Untreated samples pass the test criteria at 275°C

Untreated samples pass the test criteria at 300°C

Samples which had been previously exposed at 275°C pass (for samples with a 0g load during testing) when re-exposed at 300°C

Samples which had been previously exposed at 275°C fail (for samples with a 70g load during testing) when re-exposed at 300°C

Untreated samples fail the test criteria at 365°C

Using white light profiling to measure the variation in dimple height variation with temperature and time, it was found that height measurements on the single Upilex foil showed a significant height reduction after thermal exposure.

On average, shrinkage of up to 24% was found after 1 hr exposure and this increases to an average of 34% total shrinkage from the original height after 217 hours thermal exposure.

6. CONCLUSIONS

The use of hot embossed Upilex-S foils as a high temperature spacer for Bepi Colombo is deemed to be permissible to temperatures of about 275°C. For higher temperatures (300°C and above) thermo-mechanical relaxation is expected for longer durations and this may (especially above 300°C) lead to a shrinkage of more than 20%.

7. REFERENCES
