THE RAPID IDENTIFICATION OF IMPACT RESIDUES IN THE SOLAR ARRAY PANELS OF THE HST BY DIGITISED BACK-SCATTERED ELECTRON & X-RAY ELEMENTAL IMAGING

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ABSTRACT

Spacecraft in low Earth Orbit are prone to hypervelocity impact damage from micrometeoroids and space debris. Preliminary optical surveys using a petrographic microscope and then a detailed scanning electron microscopy study using digitised back-scattered electron imaging and X-ray emission elemental mapping has lead to a simple and rapid identification method for impact residues. The methodology was developed using nine solar cell samples removed from the returned V-2 Solar Array Panel of the Hubble Space Telescope after 3.62 years of space exposure. The results suggest that maybe the majority of small impact craters (< 100μm in diameter) are a consequence of micrometeoroids rather than space debris although a large data set would be required to substantiate this.

1. INTRODUCTION

Spacecraft which are in Low Earth Orbit (LEO) are particularly susceptible to impact damage from particles less than 1 mm in diameter travelling at speeds between 5-70 km/s (Ref.1). This hypervelocity material can be defined as either natural micrometeoroids (impact residues containing Mg, Fe, Ni, S etc.) or space debris, such as paint fragments or rocket propellant (impact residues containing Ti, Al etc.). Impact damage and ultimately the residue chemistry, can only be studied when LEO materials are returned to Earth and investigated as part of post-flight investigation. The scientific benefit of such studies was highlighted by the Long Duration Exposure Facility (LDEF) launched by NASA to study the effects of long term space exposure (LDEF was in a LEO for 5.7 years) (Ref.2). The Post-Flight investigation of LDEF included the effects of impact damage on all types of surfaces, including solar cells (Ref.2). The impact investigation primarily employed optical microscopy for sample surveys and crater measurements and then more advanced electron microscopy techniques (Ref.3), such as Field Emission Scanning Electron Microscopy (FESEM) (Ref.4). These techniques enabled the distinction between micrometeoroids and space debris and in some cases more detailed classifications of the original impactor (Ref.3). LDEF findings also highlighted the fact that surfaces which were previously thought only to be susceptible to micrometeoroid impacts were also subject to collision with space debris as well (Ref.2).

This increase could be directly attributable to the increase in the utilisation of LEO for communication satellites, etc (Ref.5).

The techniques and knowledge gained from LDEF and more recently post-flight studies of the ESA European Retrievable Carrier (EURECA) were used in an initial investigation of one of the retrieved solar array panels from the Hubble Space Telescope (HST) (Ref.7). Prior to retrieval, the array had been in a LEO (600km) for 1320 days (Ref.1). For the impact analysis, the array was cut up into individual solar cell samples and sent to several European institutes (Ref.7). The initial impact residue analyses of the solar cells suggested that the identification of extraneous impact material would be complicated by the complex nature of the solar cells (Ref.8). The collector cells are made of a top, protective layer (150μm thick) of CMX borosilicate glass, bonded by a layer of silicone resin (70μm thick) to the underlying silicon solar cell (250μm thick). This composite structure is supported by a fibre-glass backing tape, again held in place by resin (Fig.1) (Ref.9). The main chemical constituents of the cell are Si and Ca, elements which have in the past also been used as indicators of micrometeoroid residues, but which clearly cannot easily be reliably used to recognise impact residues in this context.

Figure 1. A cross-section through a typical solar cell and supporting resin stiffener.

Analysis was further complicated by the dilution of other potentially more reliable elemental “fingerprints” due to mixing during impact with the target cell.
composition. This analytical difficulty had previously resulted in several of the initial investigations yielding inconclusive evidence of natural impactors (i.e. micrometeoroids, Ref.8). Nevertheless, a number of small (1-5mm) craters were attributed to micrometeoroids (Ref.10). Space debris residues proved somewhat easier to recognise due to their distinctive chemical signature (e.g. substantially higher Ti and Al than in the cell). The majority of impacts were thus attributed to space debris although a substantial number were unclassified. This was also in part due to time constraints of the post-flight investigation (Refs.8, 10).

Here we report on the further development of the techniques applied in previous studies (Refs.7, 10) and upon their improved utilisation in location and identification of impact residues. Recent work (Ref.11) has confirmed the tentative suggestion of the post-flight investigation that the best preservation of extraneous material should occur in samples where only the top layers of the cell have been penetrated and the underlying layers have acted as a trapping medium. This observation has led to the selection criterion for the samples examined in the present study. Suitable craters are generally less than 100μm in diameter, smaller than many considered in earlier studies (e.g. diameters up to 1mm in Refs. 7, 8 & 10). Thus there is a samples bias towards small impactors and / or low impact velocities.

2. ANALYTICAL PROCEDURES

The nine solar cells used in this study were removed from the upper blanket of the Solar Panel Assembly (SPA), section B of the V-2 array. The sample numbers of the cells are: S162, 163, 164, 169, 170, 175, 177, 275 and 276. Note that 275 and 276 were cut after the solar array had been removed from the clean room at ESTEC (Noordwijk) and therefore might be expected to show any effects of terrestrial contamination.

The samples were initially examined using a Zeiss-Axioplan Universal optical microscope. The optical survey was carried out at x10, x20, x40 magnification and was used to locate impact features. The samples were then carbon coated to prevent electrical charging during investigation by a JEOL JSM 840 SEM fitted with an Oxford Instruments e-XL X-ray Energy dispersive spectrometer (at Oxford Brookes University). The analytical work was carried out at an accelerating voltage of 20kV with a beam current of 2nA and a working distance of 32mm. The initial analysis involved the use of digitalised back-scattered imaging (BEI) of the impact crater at a low magnification (<x250) but at a high pixel resolution (512 x 512 point matrix with repeated Kalman frame averaging to increase signal to noise ratio). The impact pit was then examined at high magnification (<x750) using BEI, but this often did not clearly show the presence of residue. X-ray emission mapping was therefore employed for 20 characteristic X-ray energy intervals (Fig.2).

![Figure 2. An example of the ED spectral windows used for X-ray mapping.](image)

Areas of interest were located at low magnification and low resolution (128 pixels), then mapped at higher magnification and resolution and with repeated frame averaging to yield high contrast. The initial BEI and mapping investigations identified residues on a low detailed scheme (Ref.6) within 3 hours of analyse time. Selected spot micro-analyses of the located residue were then acquired and, after distinction of cell melt components by comparison with known cell composition, a classification based on elemental association in the impact was performed. The results are broadly comparable with the types of particle signature utilised in classification of LDEF impactors (Refs.3, 6), but we suggest that a more careful scrutiny of the elemental association and ratios may lead to a more specific evaluation of particle origins, whether natural or artificial.

3. RESULTS

The nine samples studied all contained small impact craters (diameter <100μm). A total of eleven impact craters was analysed (table. 1).

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Findings</th>
<th>Conclusion</th>
</tr>
</thead>
<tbody>
<tr>
<td>162</td>
<td>Mg, Fe, S</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>163</td>
<td>Spalled melt</td>
<td>Unknown</td>
</tr>
<tr>
<td>164</td>
<td>Fe, S, Ni, Mg</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>164</td>
<td>Fe, S</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>169</td>
<td>-</td>
<td>Unclassified</td>
</tr>
<tr>
<td>170</td>
<td>Fe, S</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>Stiffener (170)</td>
<td>Fe, Ni, Cr, Ti, Mn</td>
<td>Space Debris</td>
</tr>
<tr>
<td>175</td>
<td>Fe, Ni, S, Mg</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>177</td>
<td>Fe, Ni, Mn</td>
<td>Micrometeoroid</td>
</tr>
<tr>
<td>275</td>
<td>Na, Cl, C, N, O</td>
<td>Space Debris</td>
</tr>
<tr>
<td>276</td>
<td>Fe, S</td>
<td>Micrometeoroid</td>
</tr>
</tbody>
</table>

Table 1. Summary of impact residue results
Cells 164 and 170 both contained two impact craters, both of which were analysed, although it is not possible to attain whether these craters are associated with one or different impact events on the same cell. Although only the major impact features were studied, all of the samples contained evidence of multiple impact deformation to varying degrees (e.g. from shallow surface cracks on the CMX glass to 10μm or larger impact crater).

4. DISCUSSION

4.1 Methodology

The preliminary optical survey made it possible to select the most promising samples likely to contain residue material, for additional study by electron microscopy. At higher magnification (>×20) it was possible to locate debris and melt material within the crater pit, although it was not possible (using the optical microscope) to infer an origin for such material. It nevertheless suggested that the sample would be worth a detailed investigation. The material located by optical microscopy often appeared as black, metallic, fibrous particles above the underlying substrate of the solar cell. The repeated discovery of such material strongly suggests that partially penetrated cells offer the best preservation of impacting material (as suggested by previous work, Refs.10, 11).

When a solar panel experiences an impact event the cell itself undergoes extensive damage which often results in the partial detachment (i.e. spall zone) of the CMX glass and localised fusion of the underlying layers. The crater pits often exhibit melt features of both the cell and the impactor. This means that the preserved debris is often of complex chemical composition, e.g. the residue can consist of at least four components: impactor fragments and melt, impactor and cell melt (this can be composed of several different cell substrates), and cell fragments and melt.

The digitised BE images (BEI) show the compositional contrast between pixels and may define the chemical changes within the different layers of the cell and more importantly the compositional differences between the cell and any extraneous matter. Most impact residues under BEI have a distinctive, ropy vesicular appearance when compared to the surrounding host area (Fig.3). In some case these melts demonstrate possible volatile retention during rapid cooling. This is indicated by the structures in Fig.3: within the residue there are small areas (dark in the BEI) which could be gas bubbles.

Figure 3 A BEI of a vesicular residue within a melt.

In previous studies (Ref.8 & 10) impact features were investigated using secondary electron imaging (SEI), which produced highly detailed images of the impact crater. These were not always useful when attempting to locate residues, since SEI is primarily a topographic imaging method and in a number of cases the impactor and the cell have fused, resulting in a complex melt where the residue of the impactor maybe embedded within the layers of the cell leaving little or no surface trace. BEI can locate compositional differences between a impactor residue and the cell melt to a depth of about 1μm below the surface of the crater pit (Fig.4).

Figure 4. Comparison between SEI and BEI.

Previous work on the HST solar cells (Ref.10) had used BEI, but found it not to be particularly useful. It may be this was due to the samples being gold coated (instead of carbon). Although such a coat can be used for SEI work, it reduces the clarity of BEI and means that embedded features such as those in (Fig.4) would probably not be identified (Ref.12).

After the BEI has located the area containing the residue, the digitised X-ray maps distinguish between the elemental variations that occur within the melt and thus distinguish between the cell components and the impactor residue. It is possible from the maps to infer the origin of the impactor. For example Fig.5, the maps identify four different compositions within the residue: a Mg-silicate, Fe-Ni sulfide, Fe-sulfide and
possibly Fe-Ni metal. Thus the impactor was probably a polymineralic micrometeoroid and is similar to the types of chondritic residues identified in LDEF (Refs.2, 3).

![Figure 5. X-ray maps identifying the different elemental components within the residue.](image)

The X-ray maps can also be used to identify embedded features, the benefit they have over BEI is that they can show elemental variations up to a depth of about 5 μm beneath the melt surface.

Again the reason why the X-ray mapping appears to be so successful in the identification of residues compared to previous work (Ref.10) may also be due to sample coating (i.e., carbon versus gold). A gold coat on a sample will greatly affect the maps, because not only does it prevent beam penetration, but it will also mask several important elements used for residue identification purposes (e.g. S, P and possibly Ni) (Ref.12).

The combination of BEI and X-ray mapping have proved highly successful in the location and identification of residues within the solar cells studied. The small number of samples specially selected as being the most likely to contain residues does create a sample bias. To prove that the techniques can be used on other types of cratering, a sample which had been completely penetrated was selected. This impact crater was probably one of the most difficult to study because it was in the stiffener substrate and not the solar cell itself (Fig.1). The stiffener is composed of several silicone and sulfone resin layers and a woven glass fibres. The glass fibres complicate imaging of the crater because they do not allow a high quality carbon coating, resulting in charging during SEM analysis, which meant that it was almost impossible to obtain an SEI of the crater and very difficult for BEI. The digitised BEI did allow the affect of the charging to be reduced and a high resolution image was produced (Fig.6a). This image showed bright particles on both the crater lip and on the glass fibres within the crater. The image also confirmed that the impactor had completely penetrated the cell, reflected by extensive damage to the glass fibres. A higher magnification image of individual fibres indicated that residue had been fused on to them (Fig.6b). X-ray maps of the crater, and the individual fibres within, showed the different compositions of the stiffener layers and areas slightly enriched in Fe, Ni and Cr (Fig.6c & 6d). The elemental composition (especially the high Cr) would suggest an impactor of artificial origin, i.e. a metal alloy such as stainless steel. The size of the impact residues on the individual fibres (~1 μm) meant that even semi-quantitative analyses could not be carried out. The location and subsequent identification of a residue in such a complex substrate proved that the techniques employed in this study can be used on various types of substrate from the ‘simple composition’ of the solar cells to a composite glass-fibre stiffener.

![Figure 6a. The BEI of the impact into cell stiffener, 6b. The BEI of an individual glass fibre, 6c & 6d. EDS of the bright particles and residue identified in the crater](image)

### 4.2 Micrometeoroid Impactors

Seven of the eleven impact craters studied contained natural micrometeoroid residues (Table.1). These were classified as: Fe-Ni sulfides or mafic silicates (e.g., olivines and pyroxenes). The mafic silicates which represent chondritic impactors are typical of those detected on LDEF [Refs.2-4]. The Fe-Ni metal residue, on the other hand may represent a micrometeoroid class not frequently identified in the collections of cosmic dust [Ref.13].

The fact that the majority of the impacts appear to be a consequence of micrometeoroids rather than space debris is interesting and worthy of further comment. Firstly, the HST solar arrays prior to removal were in a sun facing orbit 60% of the time (Ref.8) and may not, therefore present an unbiased random sample of impactors. Secondly, the low incidence of space debris could be associated with the orbital altitude of the HST (~600 km). Compared to both EURECA (500km) and LDEF (320-450 km) the HST was in a higher orbit,
although the results for LDEF (Ref.3) are similar to the findings here.

The mafic silicates included the probable identification of an Mg-olivine, which LDEF studies [Ref.3] failed to observe. The frequent discovery of sulfides in residues highlights the fact that our technique is able to characterise residues which contain volatile elements which were thought to vapourise during the violent impact process. The reason why such volatile elements are not lost could be related to the type of impacts, i.e. because they have not completely penetrated the cell, there is an efficient trapping mechanism. Some volatiles including gases may also be retained in the melt glass because of rapid cooling.

Although it is possible to identify the natural micrometeoroids as extraterrestrial, it is not easy to classify them in terms of meteorite class using the present schemes (Refs.3, 6). These previous schemes have classified residues as “chondritic”, but the use of the term is ambiguous. Herein a number of Fe-Ni sulfides has been detected, which could be residues from chondritic particles. Alternatively, however, they may also arise from meteorites analogous to irons or stony-irons. It is therefore preferable to specify likely mineralogy rather than attempt detailed classification to meteorite type.

4.3 Space Debris Impactors

Only two of the impact residues studied (Table.1) were identified as space debris. These were classified as S-bearing organic matter and salt and a residue of Fe with accessory Ni and Cr. The presence of Cr implies that the impactor was some type of stainless steel (which has numerous space applications). Solar cell 162 contained impact crater caused by a natural impactor also contained a Ti-rich particle in a matrix rich in C, N and O, suggestive of a paint fragment and associated bonding polymer. This fragment was fused to the rim of the crater (Fig.7).

This implies that it is possible for cells to collect low velocity “additions” which may or may not be the cause of the crater formation. This finding shows a complication to post-flight investigations, it is quite possible to wrongly identify which impactor generated the crater!

4.4 Further Applications

The X-ray maps can rapidly identify minor elemental variations across the surface solar cell substrate at low magnification (<x50). This could be used to locate rapidly the number of impact craters present on a sample. The best method of carrying out this type of work is to use a feature which was identified in previous studies (e.g. Ref.8), that the solar cells are coated with an ultra-thin MgF$_2$ layer. When a cell has experienced an impact event this layer is removed, thus by mapping for either Mg or F, it is possible to locate area with low concentrations in these elements and thus locate craters (Fig.8).

Figure 8 Elemental map for F over crater area.

The characteristic X-ray map showing the loss of an F-rich surface (Fig.8) where a impact has occurred, also clearly defines the extent of damage. Thus these maps could be used for the crater measurements carried out as apart of a post-flight investigation (Ref.7).

5. CONCLUSIONS

There is no simple technique which enables comprehensive identification of residues in very small impact craters on space hardware. However the combination of BEI and X-ray mapping appears to offer a systematic method by which residues can be located, identified and where possible classified by micro-spot analyses. These techniques could be employed in identifying new features when future samples (e.g. from EURECA II, or space station materials) are returned to Earth, as apart of the Post-Flight Investigations.
The results of this study indicate that 11 non-penetrating impacts (i.e. craters <100μm in diameter) in HST solar cells are a consequence of natural micrometeoroids and not space debris. Although a larger data set is required to substantiate whether this is a general feature on all impacted space hardware, it does support the findings of LDEF [Ref.3] and previous HST impact residue studies [Ref.10].

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REFERENCES


