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# ANALYTICAL ELECTRON MICROSCOPY OF LDEF IMPACTOR RESIDUES

Ronald P. Bernhard & Ruth A. Barrett Lockheed ESC, C-23 Johnson Space Center Houston, Texas 77058 Michael E. Zolensky Solar System Exploration Division NASA Johnson Space Center Houston, Texas 77058 S 26 - 9D

### ABSTRACT

The LDEF contained 57 individual experiment trays or tray portions specifically designed to characterize critical aspects of meteoroid and debris environment in low-Earth orbit (LEO). However, it was realized from the beginning that the most efficient use of the satellite would be to characterize impact features from the entire surface of the LDEF. With this in mind particular interest has focused on common materials facing in all 26 LDEF facing directions; among the most important of these materials has been the tray clamps. Therefore, in an effort to better understand the nature and flux of particulates in LEO, and their effects on spacecraft hardware, we are analyzing residues found in impact features on LDEF tray clamp surfaces. This paper summarizes all data from 79 clamps located on Bay A & B of the LDEF.

We also describe current efforts to characterize impactor residues recovered from the impact craters, and we have found that a low, but significant, fraction of these residues have survived in a largely unmelted state. These residues can be characterized sufficiently to permit resolution of the impactor origin. We have concentrated on the residue from chondritic interplanetary dust particles (micrometeoroids), as these represent the harshest test of our analytical capabilities.

### INTRODUCTION

LDEF experiment trays were held in place by a series of chromic-anodized aluminum (6061-T6) clamps (Figure 1); eight clamps were used to attach the experiment trays on each of the 12 sides of LDEF, while experiment trays on the Earth and space ends were held in place by 12 clamps. Each clamp was fastened to the spacecraft frame using three stainless steel hex bolts. Clamps exposed an





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approximately 58 cm<sup>2</sup> each (4.8 cm x 12.7 cm x 0.45 cm thick, minus the bolt coverage). All 774 LDEF clamps were surveyed for impact features greater than 0.5 mm in diameter during experiment tray deintegration at the Kennedy Space Center. Some 337 out of 774 LDEF tray clamps have been archived by the M&D SIG in the Curatorial Facility at JSC and are available for scientific examination by qualified individuals.

LDEF affords the opportunity to obtain information about the directionality of the meteoroid and debris fluxes. This data can then be related to the sources of meteoroids and orbital debris only if the progenitor particulate can be identified for each (or a representative population) of impact features. With this goal in mind we are characterizing the bulk chemistry of a large number of impactor residues, and the detailed mineralogy of a fraction of these. This information is needed to deduce the asteroidal versus cometary abundance of impacting meteoroids, and source of spacecraft debris particles.

# RESULTS

#### **Clamp Survey**

A clamp numbering scheme was devised which would provide hardware location information with respect to its position within a particular bay (Figure 1). From the labeling scheme, it can be seen that a clamp occupying position 1 of Bay B02 would be identified by the label B02-C01, with B02 indicating the experiment location of Bay "B" and Row "02", and C01 interpreted as "C" for clamp and "01" being the clamp number. Each clamp uses a Cartesian coordinate system to reference impact locations on exposed surfaces. The X and Y coordinates were measured in millimeters using a standard origin assigned by the M&D SIG at the lower-left corner of each clamp (Figure 1).

Optical scanning of clamps, starting with Bay A Row 01 and working through the entire satellite, is being conducted in the Facility for Optical Inspection of Large Surfaces (FOILS) at JSC to locate and document impact features as small as 30 microns. These impacts are then examined by Scanning Electron Microscopy/Energy Dispersive X-ray Analysis (SEM/EDXA) to further characterize those features which contain appreciable impactor residue. Based upon the bulk composition of these residues, and using criteria developed at JSC (ref. 1), we have made a preliminary discrimination between micrometeoroid-and space debris-containing impact features. These data are then published in a catalog format which includes: (1) an optical photograph of each clamp, (2) a secondary electron image of the impact, (3) associated parameters such as impact feature size, (4) an EDXA plot of the residue, (5) impactor origin (if applicable), and (6) a curatorial number which will facilitate requests for specific impact features by interested investigators (ref. 2). An example of a page from one of these catalogs is given in Figure 2. All





Figure 2. An example page from NASA TM #104759. In these catalogs each crater is documented and a general X-ray spectra of associated impactor residue is illustrated.

results are being input into the M&D SIG computerized database, which documents all LDEF meteoroid and debris results, and is accessible to investigators via Internet or by modem (ref. 1).

Each of the clamps is optically scanned; and all impact features greater than 40 microns (and some as small as 30 microns) are labeled and their position is documented. After scanning, an optical photograph is taken of the clamp illustrating each of the impact features located optically. SEM/EDXA is then conducted on each feature which has been identified optically. Not all features identified are high velocity impacts; in some cases, because of resolution limits during optical inspection, clamp manufacturing flaws, handling flaws, and contamination spots have been mistakenly identified as impact features. During SEM/EDXA analysis these features were properly identified and labeled as such. These features include craters and pits caused during manufacturing and handling of the clamps. Residual abrasive grit, from the polishing step of manufacture, could become trapped between the clamps when they were stacked one upon each other; inovement caused these grains to roll and leave tracks and pits (ref. 3).

Because the initial intent of this survey was to identify only those impacts which contained large amounts of micrometeoritic residue, a minimal amount of time has been spent analyzing for small or unobvious projectile remnants. Therefore, many of the impacts are classified as having no definite origin. Detectable residues were classified as either natural or man-made materials (Figure 3), and each of these two main populations may be further broken down into subgroups. Of the 425 craters examined to date, 136 contained residues categorized as natural, being from chondritic interplanetary dust particles. Monomineralic, mafic-silicate (olivines, pyroxenes and phyllosilicates) compositions and Fe-Ni sulfide particles were found to a lesser degree.

In some cases, large amounts of seemingly unmelted projectile fragments have been observed (Figure 4) in LDEF impacts, these projectile residues undergo further analytical processing. A detailed



compositional structural and analysis of several impactor residues was performed utilizing transmission electron microscopy (TEM), dispersive energy electron spectroscopy, and diffraction (e.g., ref. 4). Details of the procedures involved for the mineralogical characterization of impactor residues are given below.

Man-made debris compositions include spacecraft thermal paint rich in Zn, Ti, Cl, and Si (32 impacts); electrical components

Figure 3. Histogram illustrating the size-frequency distribution of the various particle types.





Figure 4. SEM images of chondritic projectile residues detected in crater 102. Portions of these residues were extracted from the crater and analyzed by TEM. (A) 210µm diameter crater, scale bar = 42µm. (B) Closeup of projectile residue, scale bar = 5µm.

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seen as Pb, Sn, and Ag (5 impacts); and spacecraft structural hardware consisting predominantly of Stainless Steel and Al/Mg alloys (18 impacts; not easily detected on Al surfaces).

The 234 clamp impact features in which we have found no detectable residues displayed only a composition typical of the clamp aluminum alloy. While we believe that many of these impacts were in fact caused by Al-dominated debris particulates, we also maintain that further, more detailed, analyses will undoubtedly uncover evidence of impactor residues in many of the presently unclassified impact craters, and the support of such subsequent analyses is a primary objective in publishing the catalog. A factor hindering our analyses is the fact that the clamps have all been anodized, a process which deposited a surface layer of Si, Mg, and S, all of which are important elements for the discrimination of natural from man-made materials. For this reason, we would like to discourage the use of anodized coatings in the future where the gain in thermal protection to the spacecraft will be negligible.

As mentioned we have published our data on all 425 impacts found on Bay A and Bay B of the LDEF (ref. 2). Subsequent catalogs will include clamps from succeeding bays of the satellite, and will be published as time and resources permit.

#### Mineralogy of Impactor Residues

LDEF impactor residues are being characterized to establish the nature and abundance of meteoritic and orbital debris materials in the LEO environment. Although our goal is to characterize residues from *both* orbital debris *and* meteoroids, we have concentrated our initial efforts on the more fragile chondritic meteoroids, since these represent the worst case for hypervelocity impactor residue preservation. If we can be successful with chondritic meteoroids we can surely succeed with the orbital debris residues.

We have developed simple techniques for the study of selected chondritic (containing Si, Mg, Fe, +/-Al, Ca, S, Mn, and Ni in appropriate amounts) impactor residues in shallow craters in gold plates, from the LDEF experiment A0178. A detailed structural and compositional analysis of several of these impactor residues was performed utilizing transmission electron microscopy, energy dispersive spectroscopy, and electron diffraction. The immediate goal of this continuing work has been to determine the shock effects exhibited by chondritic meteoroids (a.k.a. Interplanetary Dust Particles or IDPs), and to compare the impactor residues to chondritic IDPs collected from the stratosphere.

Residues from the interior of several meteoroid impact craters were removed with a tungsten needle, mounted in EMBED-812 epoxy, and ultramicrotomed into 90 nm thick sections. Observation of the sections on carbon-coated copper grids was done by transmission electron microscopic techniques using JEOL 100CX and 2000FX analytical electron microscopes. Chemical analyses were performed with a PGT System 4, and an energy dispersive X-ray spectrometer and reduced with the PGT dedicated software. The structural state of all analyzed materials was assessed by electron diffraction, which proved to be a critical step, considering the non-crystalline nature of many materials observed.

We examined the mineralogy of residues from three impact features: nos. 102, 121, and 295. Impact residue in 102 has abundant, very finely-divided, crystalline augite  $(En_{55-59}Wo_{36-40})$  and orthopyroxene  $(En_{84-96})$  showing abundant evidence of intense shock, these being planar deformation features, mosaicism (see Figure 5), and, in some instances, evidence of recrystallization (120° grain intersections). The matrix consists of frothy ferromagnesian glass. Spherical bodies of Fe-Ni metal and pyrrhotite abound locally, particularly at grain boundaries (see Figure 6). Some Fe-Ni grains are metallic glasses (J. Bradley, personal communication, 1993). Impact residue 121 contains fragmental grains of olivine (Fo<sub>57-67</sub>), orthopyroxene ( $En_{63-64}$ ), Fe-Ni metal, and abundant frothy glass (see Figure 7). The olivine and pyroxene grains show abundant evidence of shock (see above for criteria). Impact residue 295 contains shocked, fragmental olivine (Fo<sub>56-71</sub>) and orthopyroxene ( $En_{71}$ ), pyrrhotite, and glass.

The pyroxenes in residue 102 have Fe-poor, restricted compositions (see Figure 8). For comparison we show the observed compositional ranges of olivines and pyroxenes in chondritic IDPs in Figure 9. As can be seen in the latter figure, the compositional range of ferromagnesian minerals is considerably more restricted in the hydrous IDPs relative to the anhydrous ones. Comparison to the compositions of ferromagnesian minerals in residue 102 (Figure 8) suggests that impactor 102 was a hydrous IDP. The parent bodies for hydrous IDPs are believed to be main belt asteroids.

The compositions of olivines and orthopyroxenes in the other residues characterized in this study are equilibrated compare to anhydrous chondritic IDPs, and also Fe-rich compared to hydrous chondritic IDPs (see Figure 9). They are also Fe-rich as compared to ferromagnesian silicates from partially melted chondritic IDPs (see Figure 8), which are typically on the order of  $Fo_{90}$  and  $En_{90}$ . The presence of equilibrated and shocked ferromagnesian minerals, recrystallization textures, glass, and melted metal and sulfide bodies decorating grain boundaries, is indicative of varying degrees of shock metamorphism in all impact residues we have characterized. Our failure to locate any magnesian olivines or pyroxenes in these particular residues is illustrative of the pervasive shock metamorphism they experienced. We are continuing to characterize additional IDP impactor residues, including those from suspected orbital debris particulates.



Figure 5. A TEM image of a microtomed residue grain from impact 102. Pyroxene crystals in the field of view have been recrystallized, as indicated by 120° grain intersections.



Figure 6. A TEM image of a microtomed residue grain from impact 102. Spherical bodies of Fe-Ni metal and pyrrhotite abound locally, particularly at grain boundaries.



Figure 7. A TEM image of a microtomed residue grain from impact 102. Vesicular glass abounds in the matrix of impactor residue 102, probably formed during impact into LDEF.



Figure 8. Comparison of the compositional range of olivines and pyroxenes from IDPs which have melted during atmospheric entry (melted IDPs) and IDP residue grains from LDEF impact features.



# **ANHYDROUS IDPS**



Figure 9. The compositional range of olivines (Fo to Fa) and pyroxenes (upper quadrilateral diagrams) in hydrous and anhydrous chondritic micrometeoroids.

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