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SEM-EDS ANALYSES OF SMALL CRATERS IN STARDUST ALUMINIUM FOILS: IMPLICATIONS FOR THE WILD-2 DUST DISTRIBUTION. J. Borg¹, F. Hörz², J.C. Bridges³, M.J. Burchell⁴, Z. Djouadi¹, C. Floss⁵, G.A. Graham⁶, S.F. Green³, P.R. Heck⁷, P. Hoppe⁷, J. Huth⁷, A. Kearsley⁸, H. Leroux⁹, K. Marhas⁵, F.J. Stadermann⁵ and N. Teslich⁶. ¹Institut d'Astrophysique Spatiale, CNRS, Université Paris-Sud, UMR8617, F-91405 Orsay-Cedex, France, janet.borg@ias.u-psud.fr, ²KT NASA Johnson Space Center, Houston, TX 77058, USA, ³PSSRI, Open University, Milton Keynes, MK7 6AA, UK, ⁴School of Physical Sciences, University of Kent, Canterbury, Kent CT2 7NH, UK, ⁵Laboratory for Space Sciences and Physics Department, Washington University, St. Louis MO 63130, USA, ⁶IGPP, Lawrence Livermore National Laboratory, 7000 East Avenue, L413, Livermore, CA 94550, USA, ⁷Max-Planck-Institute for Chemistry, P.O. Box 3060, 55020 Mainz, Germany, ⁸IARC, Department of Mineralogy, The Natural History Museum, London SW7 5BD, UK, ⁹Laboratoire de Structure et Propriétés de l'Etat Solide, UMR 8008, Université des Sciences et Technologies de Lille F59655 Villeneuve d'Ascq, France.

Introduction: Aluminium foils were used on Stardust to stabilize the aerogel specimens in the modular collector tray. Part of these foils were fully exposed to the flux of cometary grains emanating from Wild 2. Because the exposed part of these foils had to be harvested before extraction of the aerogel, numerous foil strips some 1.7 mm wide and 13 or 33 mm long were generated during Stardust's Preliminary Examination (PE). These strips are readily accommodated in their entirety in the sample chambers of modern SEMs, thus providing the opportunity to characterize in situ the size distribution and residue composition - employing EDS methods - of statistically more significant numbers of cometary dust particles compared to aerogel, the latter mandating extensive sample preparation [1]. We describe here the analysis of nearly 300 impact craters and their implications for Wild 2 dust.

Experimental set-ups: During PE, some 25 foils were analysed by SEM/EDS techniques in a variety of laboratories, substantially following [e.g.2,3]. In our laboratories, secondary electron imaging was carried out at 5 or 20 kV, while EDS was performed at 20 kV for heavy elements investigation and at 5 or 7 kV for light elements. Spatial resolution varied, and ranged from low resolutions of the entire foil strip, overlapping with optical microscope surveys of craters > 20 µm in size, to high resolution scans at crater sizes of 100 nm.

The goals of this investigation were to: i) carefully locate all the impact events to determine their spatial distribution, ii) characterize the morphologies (diameter, depth, surface roughness) of the impact craters and relate them to the physical properties of the incident particles (size, density) based on dedicated calibration experiments [4,5] at Stardust's constant encounter velocity of 6.1 km/s and iii) obtain compositional information on the melted residues on the crater bottoms, walls and rims, which are in part intimately mixed with molten Al.

Calibrations performed prior to Stardust return with sodalime glass beads impacted at ~ 6 km/s gave a ratio of 4.6 between the lip-to-lip diameter of the crater

and the mean diameter of the incident grain [5]. Additional shots with various materials of different densities and/or porosities indicate that this ratio does not vary greatly for silicate impactors, while the depth of the crater and the rugosity of the crater walls and bottom can be affected [6]. Concerning the chemical analysis, laboratory simulations performed with various minerals (olivine, diopside, plagioclase feldspar, pyrrhotite, etc.) indicate that the residue thickness may be only a few tens of nanometers, when the incident particles are in the micron size range, as is the case for most of the Wild-2 grains (see paragraph on "Results"). Such a thickness is much smaller than the depth of the primary electron beam interaction volume and complex absorption effects then take place, that cannot be quantified, so only qualitative identification of the major elements is possible and accurate determination of elemental ratios is not feasible in situ [6].

Results: During PE, 25 strips of foils were thoroughly scanned by the various members of the PE, allowing the characterization of 292 craters, in terms of localization, size and composition, on a total area of ~ 10 cm² of foil surveyed.

Clustering. A large variation in the crater distribution was found at the mm² scale, with a range from 0 to more than 50 craters identified per foil [1]. Three foils (C2008N, C2020W and C2044N) are heavily clustered, holding about one third of all the craters analysed by SEM/EDS in this study; furthermore, five foils (C2037N, C2044W, C2052N, C2055N and C2068W) each contain more than 10 craters, so that the eight "crater - clustered foils" contain ~ 85% of the total number of craters, in less than one third of the scanned area. This clustering of impacts and the strong variation in the spatial distribution of impact features is discussed in [7] and is not yet fully understood.

Size distribution. More than 90% of the identified craters are smaller than 5 µm in diameter, yet most are of submicron size. This implies that the size distribution of the Wild-2 dust grains is dominated by particles of submicron sizes, as small as a few tens of nanometers. Such small grains cannot easily be identified in the

aerogel collectors and have not been extracted to date. So the study of their impact features on foil remains the only possibility for characterizing the dominant population of Wild-2 dust. High resolution images of these craters show a non-uniform melting of their interior (fig 1) and even, in some cases, the presence of small crystalline residues, as confirmed by Focused Ion Beam – Transmission Electron Microscope (FIB-TEM) analyses [8]. Also, many of the craters, (e.g. figure 1) are clearly due to impact by composite grains (i.e. containing diverse internal components), as they present a markedly non-circular outline, not the simple circular bowl shapes of [6].

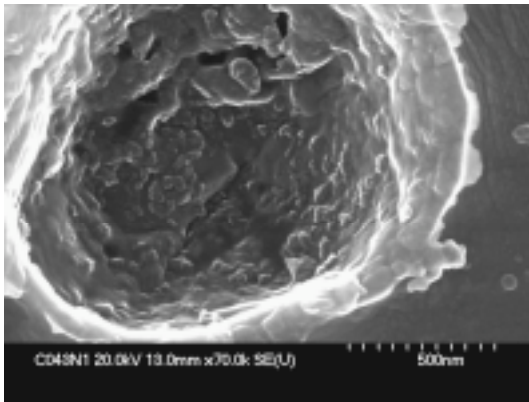


Figure 1 – High resolution SEM image of one crater on foil C2043N. Its interior is not fully melted and its shape is not round, both features characteristic of impact by a composite grain.

Chemical identification. EDS analysis was performed on 263 craters, all smaller than 5 μm . All the elemental signatures found are potentially extraterrestrial in origin; no man made components were identified and thus there was no evidence of secondary ejecta from impacts elsewhere on the Stardust spacecraft. Nearly 50% of the projectiles appear to be composite grains containing variable amounts of Mg, Fe silicates (probably olivines and/or pyroxenes) and Fe sulfides, while ~ 25% are Mg,Fe silicates alone and ~ 18% pure Fe sulfides alone (fig 2).

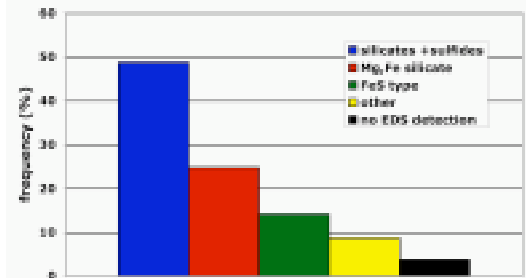


Figure 2 – Mineralogical distribution of residues in the craters.

The few remaining residues either show no EDS signature or dominance by other elements (Ca, Na, ...) in various proportions. C and O have not been considered in this statistical evaluation, as the analytical method varies from group to group. Figure 3 shows that the sub-micron particles also display the same domination by composites seen in the larger ones.

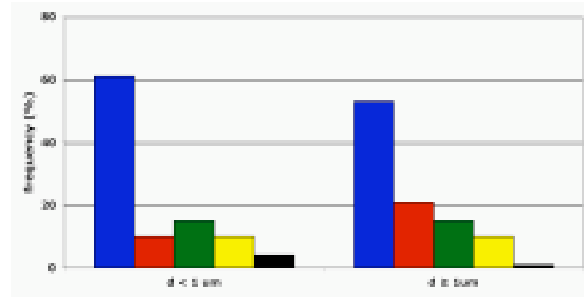


Figure 3 – Mineralogy distribution as a function of the size of the crater, clearly showing that the smallest impactors are mainly composite grains.

Conclusion: SEM/EDS, routinely used as an investigation tool of impact craters in metallic targets appears to be a very useful and sensitive tool to obtain statistical surveys of the Wild-2 dust. It permits for the characterization of the smallest size fraction of this dust, by far the most numerous, that cannot be studied currently when trapped in aerogel. It must be considered as a first tool to be followed by high resolution analytical techniques such as NanoSIMS [9], TOF-SIMS [10], FIB-SEM [11] or FIB-TEM [8], used during the PE on some craters, providing a more complete understanding in terms of chemical, mineralogical or isotopical dust compositions. In the future, SEM/EDS will continue as a survey tool to possibly identify compositionally rare and interesting particles as candidates for these more quantitative, high resolution characterizations of Wild-2 dust.

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