Progress report on TEM studies of Stardust samples

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September 9, 2006

1. Introduction

This report summarizes transmission electron microscopic (TEM) data obtained on three Stardust samples that have been provided by NASA to the University of Jena:

(1) C2054,0,35,32,1; 2nd extraction from cell 2054, giant track 1, particle #12
(2) C2054,0,35,45,1; epoxy embedded particle from track 35, grid #1, 70 nm x 4 sections on amorphous carbon
(3) C2115,24,22,1,1; extraction from cell 115; giant track 6, terminal particle Ga-rich

All three samples were captured in aerogel. Particles C2054,0,35,32,1 and C2054,0,35,45,1 were extracted by K. Messenger on 03/10/06 and 04/21/06, respectively. To ultramicrotom both samples they were embedded by K. Messenger in either Embed812 or sulfur. L. Keller measured FTIR spectra for C2054,0,35,32,1 and reports the presence of organic peaks. Particle C2115,24,22,1,1 was extracted by Chris at Berkeley on 04/05/06. It was then embedded in sulfur and ultramicrotomed by K. Messenger.

2. Methods

Energy-dispersive X-ray spectra (EDX), electron energy loss spectra (EELS), and conventional bright-field and dark-field as well as high-resolution TEM images have been acquired on two microscopes: (1) energy-filtered 200 kV ZEISS LEO922 TEM at the University of Jena and (2) 200 kV PHILIPS CM20 FEG STEM at the University of Bayreuth both equipped with ThermoNoran EDX detectors. For EELS measurements the ZEISS microscope has an integrated in-column Omega filter, whereas the PHILIPS microscope is equipped with a GATAN parallel electron energy loss spectrometer (PEELS) 666.

3. Results

3.1 Sample C2054,0,35,32,1

In order to obtain a first glance of the sample mineralogy, EDX-STEM element distribution maps of up to 16 elements were acquired. When interpreting these maps it has to be kept in mind that both continuum and characteristic X-rays are detected simultaneously and that a correction of thickness has not been applied. Neglecting ubiquitous oxygen Fig. 1 shows the most important 6 elements and the corresponding dark-field STEM image of the particle. The sample is clearly dominated by silica-rich grains but also contains titania (TiO_2) and iron sulfide as well as Mg-bearing glass areas (see also Fig. 2). The sulphide-bearing areas usually overlap with those of Mg-bearing glass.

Selected area electron diffraction (SAED) patterns have been taken to identify the crystal structures and origin of phases. SAED of the silica phase reveal that the elongated (about 1 µm long) crystals are quartz (Fig. 3). The diffraction pattern has been taken along the $[10\bar{1}]$ zone axis and shows reflections belonging to the $\{10\bar{1}0\}$ (4.25 Å) and $10\bar{1}1$ (3.34 Å) forms.

Inspection of the titania yielded two zone axis diffraction patterns that are unequivocally consistent with anatase being the TiO_2 polymorph. The diffraction patterns were taken along the $[0\bar{1}0]$ and $[1\bar{1}\bar{1}]$ zone axes (Fig. 3).

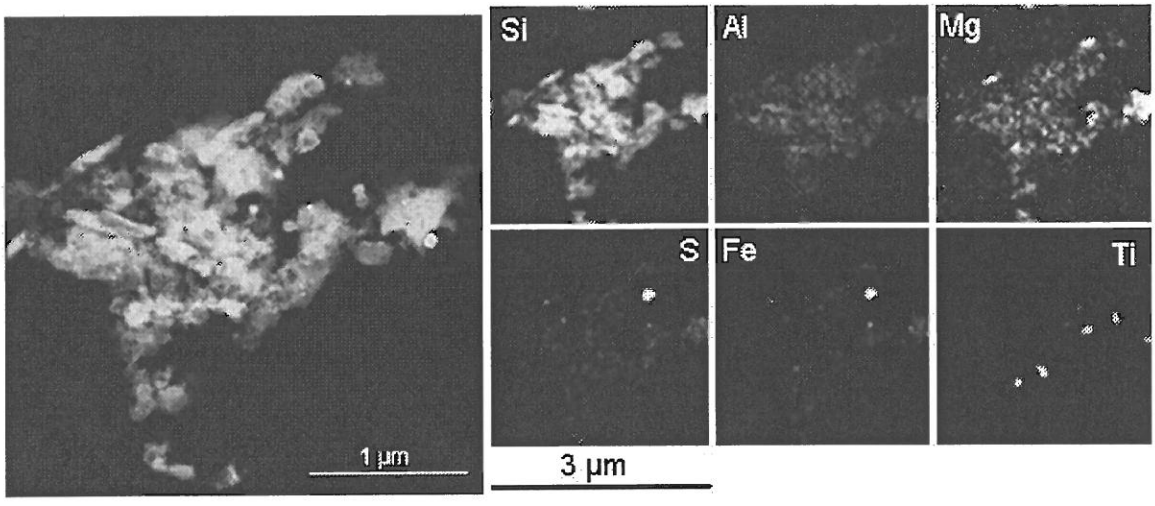

Fig. 1. Dark-field scanning TEM image (left) and corresponding element maps (right) of the overall particle C2054,0,35,32,1.

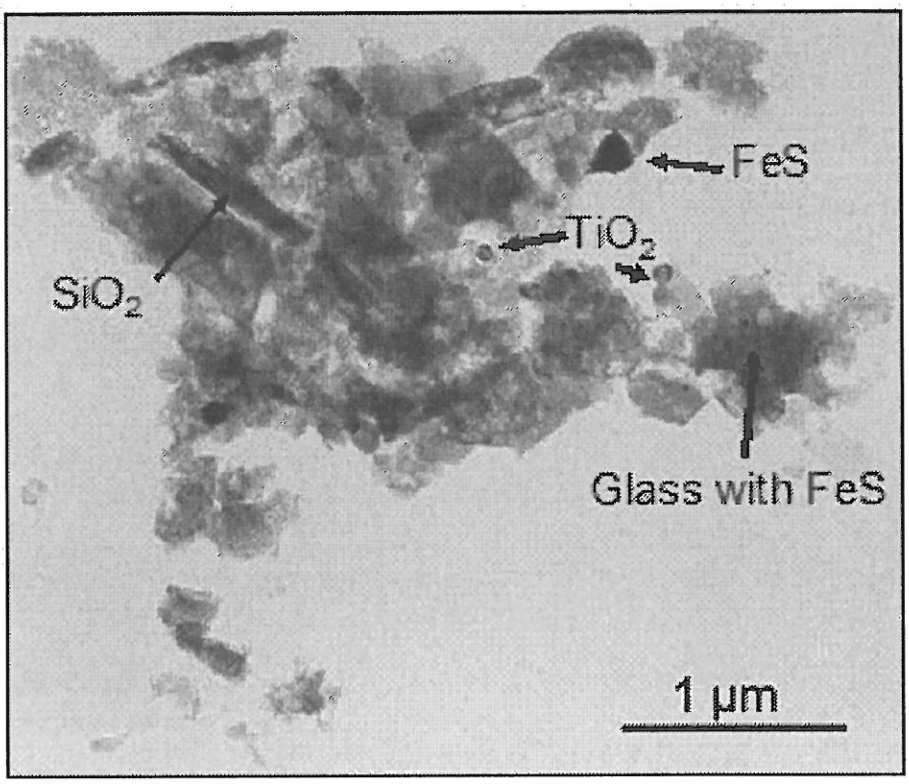

Fig. 2. Bright-field TEM image showing the various phases detected in Stardust sample C2054,0,35,32,1.

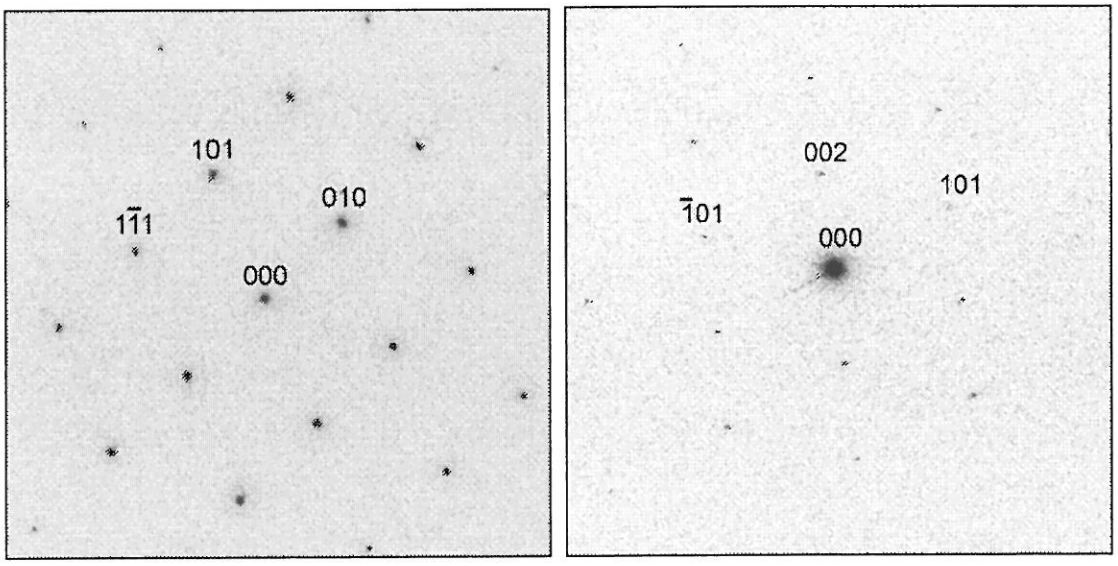

Fig. 3. SAED patterns of single crystal quartz (left) and anatase (right) from Stardust particle C2054,0,35,32,1. The quartz pattern is consistent with reflections expected for the $[10\bar{1}]$ zone axis, whereas the anatase pattern shows the reflections in the $[0\bar{1}0]$ zone axis.

The anatase crystals are commonly 100 nm in diameter, equant shaped and absolutely free of lattice defects (Fig. 4). They seem to stick on the surfaces of aggregates and have not been observed as inclusions.

The iron sulphide occurs either as isolated large grain (Fig. 2) or as rounded inclusions within Mg-bearing glass beads (Fig. 4). The SAED pattern of the large FeS crystal can be indexed as hexagonal troilite with perfect NiAs structure and lattice constants a = 3.45 Å and c = 5.80 Å. Energy-dispersive X-ray (EDX) microanalyses of troilite reveal besides Fe and S subordinate concentrations of Ni, Cr, and Mn. The glass beads are dominated by silicon but also contain some Mg, whereby the Mg:Si is low than for pyroxene.

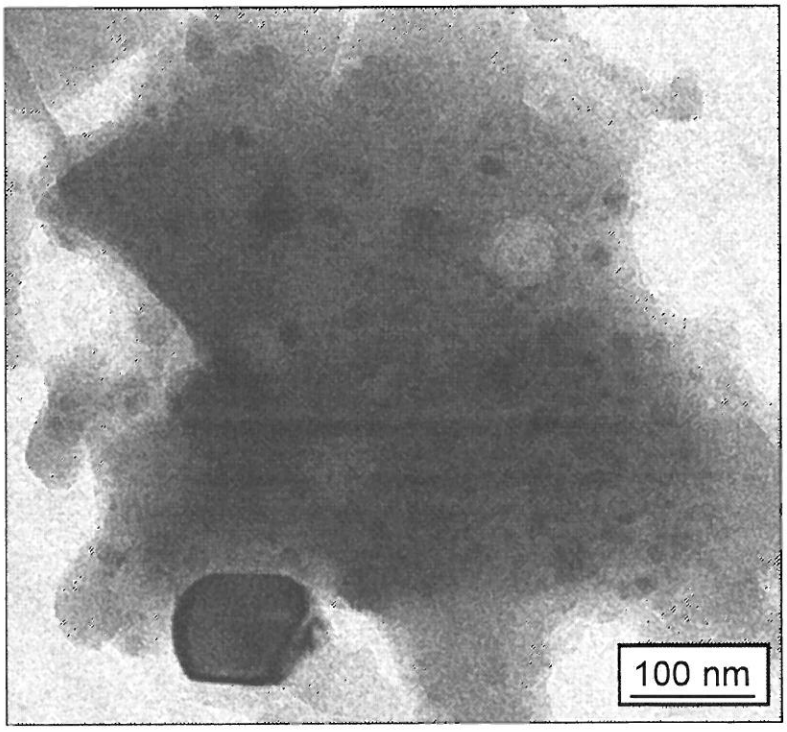

Fig. 4. Bright-field TEM image of a glass bead with rounded to spherically shaped FeS and an attached 100 nm size anatase crystal (lower left).

3.2 Sample C2115,24,22,1,1

This sample has also been mapped using EDX-STEM (Fig. 5). The element maps show that the sample is dominated by carbon but organic material as originally suggested could not be confirmed. The C *K* electron energy loss spectrum resembles that of amorphous carbon and is consistent with solely C-C bonding. SAED on the particle confirmed its non-crystalline nature.

In addition, the sample shows one attached anatase grain that is likely contamination (Fig. 5). The anatase is about 200 nm in diameter and thus of the same diameter as for the previous sample.

Further phases are revealed by EDX microanalyses. Inclusions inside the carbon-rich material are for example $BaSO_4$ (Figs. 6 and 7). The existence of sulfates in carbon-rich amorphous material suggests the whole sample represents some plastic material such as nylon fibers and has thus to be considered as complete contaminant .

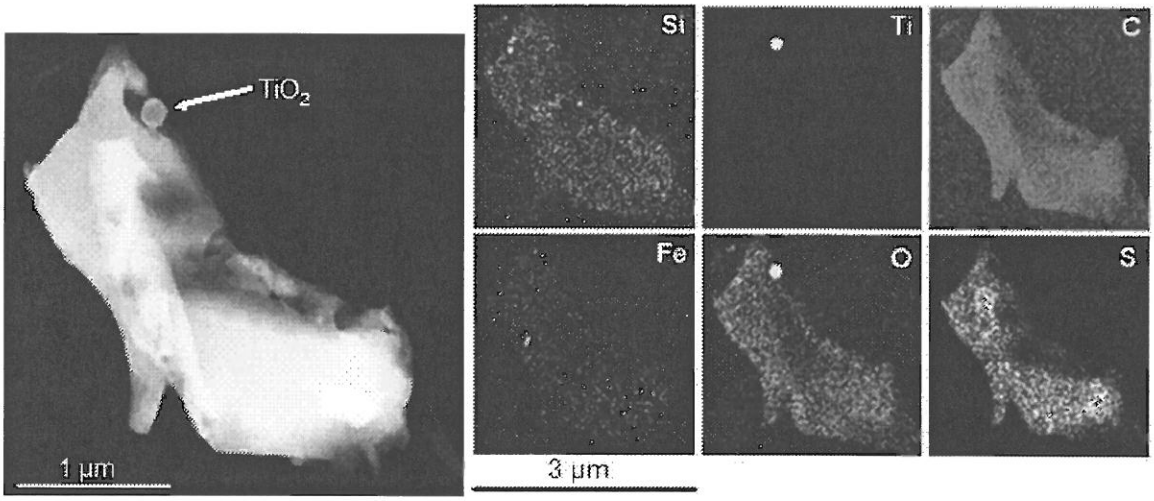

Fig. 5. Dark-field STEM image (left) and corresponding element maps of one particle in sample C2115,24,22,1,1.

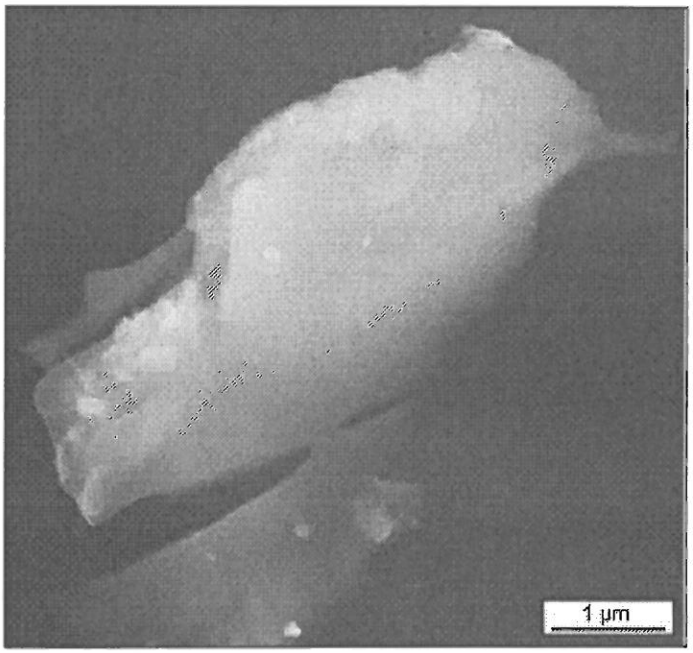

Fig. 6. Bright-field of carbon-rich particle in sample C2115,24,22,1,1.

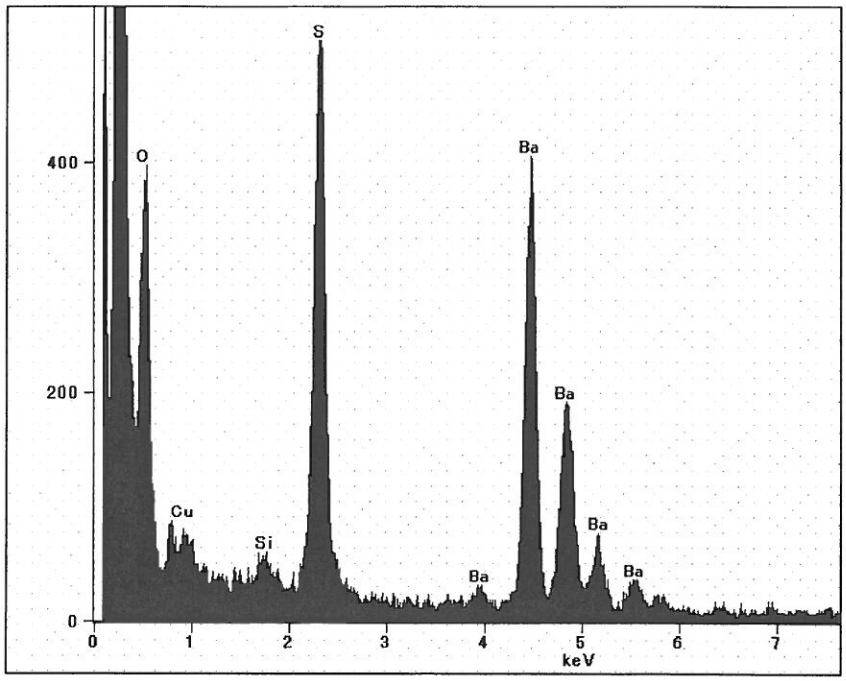

Fig. 7. EDX spectrum of a $BaSO_4$ inclusion within the particle shown in Fig. 6.

3.3 Sample C2054,0,35,45,1

This tiny sample is purely composed of carbon. SAED reveals the amorphous nature of the particle, which easily contaminates under the electron beam (see dark circle on Fig. 8).

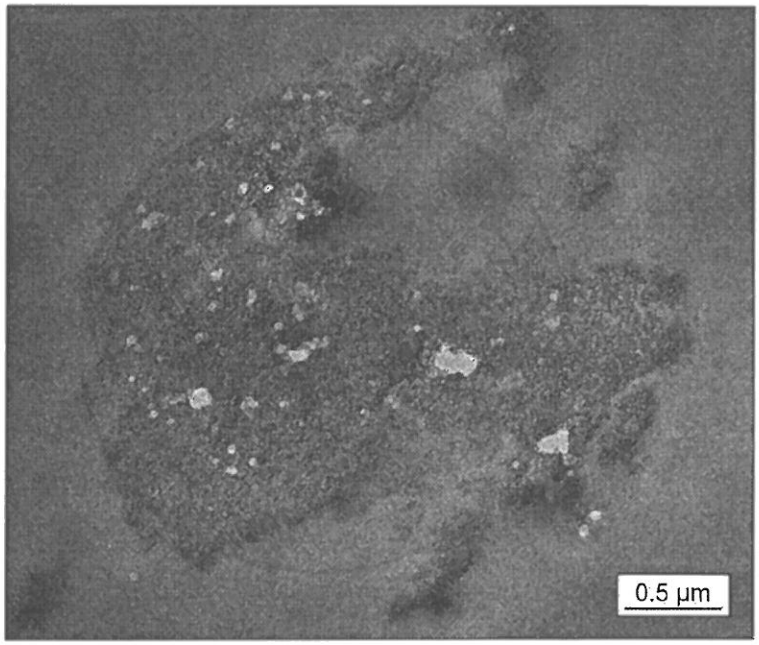

Fig. 8. Bright-field TEM image of amorphous carbon particle in Stardust sample C2054,0,35,45,1.

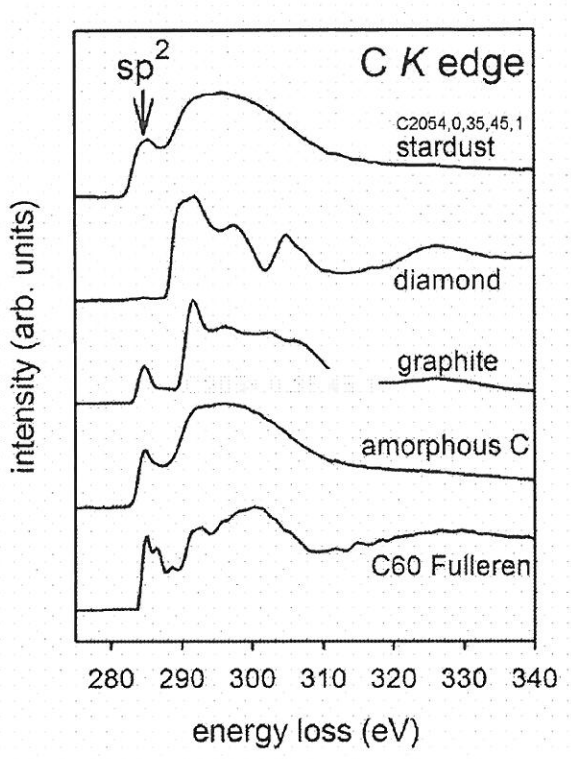

Fig. 9. C *K* ELNES of carbon particle in Stardust sample C2054,0,35,45,1. The spectrum is compared with those of various standard carbon polymorphs.

The EELS measurements of the C *K* edge show that the ELNES region is fully consistent with amorphous carbon. This is obvious from the comparison with the C *K* spectra of various standard carbon polymorphs (Fig. 9).

4. Discussion and conclusions

Besides the glass beads and troilite, other phases present in the three Stardust samples (such as anatase) have to be regarded as contaminants. Stardust sample C2115,24,22,1,1 seems to be a complete contaminant and does not contain any extraterrestrial material.

The presence of crystalline quartz indicates that high temperature prevailed along tracks leading to crystallization of aerogel. The glass beads seem to contain an excess of silicon which could also stem from strong heating sufficient for melting.

Stardust sample C2054,0,35,45,1 contains indeed amorphous carbon, which could thus be refractory presolar material. Thus, a NanoSIMS study of the carbon isotopes is highly demanded to verify or discard an extraterrestrial origin.