LOCATION OF SMALL SiC CRYSTALS IN METEORITES USING A LOW-VOLTAGE X-RAY MAPPING TECHNIQUE. Swan P. S., Walker R. M., and Yuan, J., McDonnell for the Space Sciences and Physics Department, Washington University, St. Louis, MO 63130 USA.

SiC crystals found in acid residues of Murray and Murchison prepared by the University of Chicago group have unusual isotopic compositions suggesting that the SiC is interstellar material [1,2]. We describe here an X-ray mapping technique that can be used to locate sub-micron SiC grains present in low concentrations in meteorites. In principle, the method can be used to find SiC grains in the bulk or to measure the concentration and size distribution of SiC grains in partially digested samples if the bulk concentration is ≥ 0.1 ppm. The technique is also useful for finding (rare) SiC grains large enough to permit measurement of Si and C isotopes using the Washington University ion probe.

The method relies on the fact that the number of Si X-ray photons generated per incident electron is much higher from SiC than from the other common Si bearing minerals. Using the image processing software that is standard with our equipment (JEOL 840 SEM, Tracor-Northern ultra thin window Si-Li Micro ZII detector and a 5400 Series II Image processing system), we can display separately only those pixels in the X-ray image whose value exceeds a chosen threshold. With a typical set of operating conditions which give averages of 123 Si counts/pixel from SiC and 25 Si counts/pixel from the general Si background in a bulk meteorite sample, setting the threshold at 60 Si counts/pixel means that essentially no false signals are generated in a 128 x 128 pixel image. The number of Si counts per incident electron decreases as the size of the SiC grain becomes smaller. This reduction arises from several sources: a) penetration of the exciting beam through the crystal; this also gives a background from any Si-bearing compounds underlying the crystal and dilutes the signal from the object of interest; b) multiple scattering of the beam out of the sides of the grain; and c) instrumental effects which cause the beam to spread beyond the boundaries of the grain. The average Si count rate also drops sharply when the magnification is such that the object covers an area less than two pixels in size. To test the combined effects of a-c above, we have measured three types of calibration samples. The first was a sample of a SiC-rich residue from Murchison (Chicago sample HN) dispersed on an electron microscope grid. The EDS spectrum of this sample showed a large O signal (presumably due to the strong oxidizing treatments that had been used to produce the residue) and was not satisfactory. We then studied the small size fraction of a sample of industrial SiC which we pressed into an Al foil. The final, and most realistic, standard was an acid residue sample that we had previously prepared from the meteorite Murray using four stages of HF-HCl treatment. To complement these measurements we also determined the excitation yield of Si photons as a function of bombarding energy and, also, as a function of the distance of the beam from the edge of a large SiC terrestrial standard.

To locate small grains it is imperative to operate at low excitation energies where the electrons exciting the Si photons are stopped within the grains themselves. In practice, we have found that accelerating voltages between 4 and 5 KeV represent a reasonable compromise between localization of the Si photons in small grains and a rapidly decreasing excitation function. Repeated scans of the residue sample showed that grains with a surface area of 1.6×10^{-9} cm^2 were reliably found (> 80% probability) at a magnification of 1600X.

In the Murray residue sample, each picture taken at a magnification of 1600X typically showed ~ 3 grains of SiC with diameters $\geq .4$ μm. Our estimated weight fraction of SiC of ~ 9 ppm - is close to that measured independently (~ 4 ppm) for the meteorite Murchison by the Chicago group. Assuming that Murray and Murchison are similar, this result shows that the bulk of the SiC does <u>not</u> reside in minerals such as spinel and chromite which were not affected by the relatively mild acid treatment. Rather, much of the SiC must be either in the matrix material or encapsulated in the larger silicate grains that were destroyed by the etching.

SiC searches were also made in previously prepared acid residue samples of two CV meteorites, Allende and Leoville that had received more extensive HF-HCl treatments than the Murray sample. <u>No</u> SiC grains were found in the Allende residue and we estimate a maximum concentration of ≤ 0.02 ppm for SiC grains ≥ 0.4 μm in size. SiC grains with sizes similar to those in Murray were found in Leoville, but at a lower concentration of ~ 1 ppm. Several grains large enough to measure in the ion probe were readily located in both the Murray and Leoville residues.

Once the conditions under which small SiC grains could be located had been established, we made 43 X-ray maps of a "representative" area of a polished (Al-oxide abrasive) section of the bulk Murchison meteorite, at a magnification of 1600 X and an exciting voltage of 5 keV. Regions rich in Mg, Si (~ 10% of the surface) were not studied in this survey because of higher Si X-ray background rates in these areas.

Although the probability of finding an isolated grain in the matrix was estimated at > 95%, none were found. Several explanations of this null result are possible: a) it may be that the SiC grains found in the acid residue are encapsulated in Mg-rich silicate grains in the bulk meteorite; b) prior to acid treatment the SiC grains may be coated with thick (> 1000 Å) layers of carbonaceous (or other) material that absorb the exciting electrons; c) the SiC crystals may have been removed by plucking during preparation of the polished sections; and d) the SiC grains may not be randomly distributed in the bulk meteorite. The latter is by far the most interesting possibility since it would imply the presence of "pockets" of interstellar material in the meteorite.

The X-ray mapping procedure introduces a carbon contamination on the grain surfaces. However, the level is sufficiently low that it does not interfere with the location and identification of the SiC grains. Although the surface coating would interfere somewhat with ion probe measurements of carbon isotopes, the coatings can be easily removed by cleaning the samples in an oxygen plasma.

In summary, we have demonstrated an X-ray mapping technique that makes it possible to quickly locate and measure SiC grains ≥ 0.4 μm in size in samples that have been subjected to only modest etching treatments. Application of this method to many meteorites should be relatively simple requiring only mg-sized starting samples for SiC concentrations ≥ 1 ppm. The technique should also make it possible to locate SiC grains in bulk meteorite samples so that the mineral can be put in its proper petrographic context. Additional studies of acid residues and bulk meteorites are in progress.

References: [1] Bernatowicz T. *et al.* (1987) *Nature* **330**, 728-730. [2] Zinner *et al.* (1987) *Nature* **330**, 730-7321.