COMBINED NANO COMPUTED TOMOGRAPHY AND X-RAY FLUORESCENCE MEASUREMENTS OF A PRESOLAR GRAIN. M. Jadhav[1†], M. Holt[2], R. Winarski[2], and D. J. Miller[2]. [1]Department of the Geophysical Sciences, The University of Chicago, Chicago, IL 60637, [2]Center for Nanoscale Materials, Argonne National Laboratory, Lemont, IL 60439. [†]Currently at Department of Physics, University of Louisiana, Lafayette, LA 70504. E-mail: manavi@louisiana.edu

Introduction: Correlated multi-technique analyses of individual presolar grains are the most effective way to constrain the stellar sources of these grains and gain information about their nucleosynthetic and chemical environments. Traditionally, destructive techniques like NanoSIMS and TEM of ultramicrotome sections of grains have been used to decipher the chemical, isotopic, and structural properties of stardust that provide an understanding of the circumstellar environments in which the grains condensed.

Numerous TEM studies have provided major and minor elemental information on the nm-sized subgrains in presolar grains [e.g., 1,2]. TEM is, in principle, a non-destructive technique but it requires the grains to be ultra-microtomed before analysis. This sample preparation procedure disrupts 3-d chemical information that could otherwise be recovered if the grains were studied intact. Presolar grains inherit their chemistry and subgrain population from the circumstellar gas, as they are blown through cooling outflows of dying stars. Observed radial trends in the chemical composition and subgrain size in individual grains reflect changes in gas chemistry and number density in the circumstellar region [1]. Circumstellar pressure and temperature conditions can be determined based on the condensation sequence of elements observed in the stardust grains. These measurements provide important constraints to theoretical models of circumstellar regions that cannot otherwise be obtained by astrophysical observations of such stars. Thus, it is vital to preserve the 3-d spatial structure of the grains during measurements. In the past, such studies of chemical trends vs. radial distance in grains have been painstakingly reconstructed by analyzing numerous ultramicrotome sections with a TEM [1]. This abstract presents a new technique to study presolar grains and their sub-grain population. Combined computed tomography (CT) and X-ray fluorescence (XRF) measurements at the nanoscale level will make constructing a 3-d chemical picture of presolar grains incredibly straightforward.

Prior synchrotron XRF studies of presolar SiC grains contained no 3-d information and had a lower spatial resolution of ~ 1 μm [e.g., 3, 4]. 3-d reconstructions by atom probe analysis, while non-destructive, require the sample to be sharpened into a tip, leading to a loss of material [5]. Nano-XRF data of three presolar graphite grains obtained at DESY and ESRF by [6, 7] was promising but was limited in the capability to construct a tomographic image of the grains. We present preliminary data from a new study that can combine nano CT (nCT) and nano XRF (nXRF) measurements to produce 3-d maps of the chemistry and microstructure of presolar grains.

Experimental details: A 6 micron presolar graphite grain from the low density fraction of Orgueil was selected for this study because of the large number of refractory sub-grains expected in the grains from these fractions [8]. The grain was identified and attached onto an STM tip with Pt-deposition using the Zeiss 1540XB FIB-SEM at Argonne National Lab. This attachment is necessary to prevent losing the grain in transport or when the analysis chamber is evacuated (10^{-6} torr) for the scanning nXRF measurements. nCT and nXRF measurements were then obtained using the Hard X-ray nanoprobe (26-ID-C) operated by the Center for Nanoscale Materials at the Advanced Photon Source, Argonne National Lab. This beamline provides the unique capability of carrying out non-destructive, combined nCT and nXRF measurements, not available at other beam lines or synchrotron sources at this length scale [9].

Spatially localized x-ray fluorescence spectra were generated by raster scanning a focused ~50 nm x-ray beam of 9 keV energy over an 8 μm^2 spatial area at 5 sample angles (0˚, 45˚, 90˚, 135˚, and 180˚). These spectra were detected using a Vortex Me4 energy dispersive detector at a dwell time of 0.25-1.0 s per point. From these spectra 2D maps of elemental concentrations of Al, Si, Zr, Sr, Y, Au, S, Pb, Cl, Cd, K, In, Ca, Te, Sc, Ti, Nd, V, Cr, Mn, Gd, Mn, Fe, Ni, Cu, Zn, Ga, and Co were created using the MAPS analysis software package [10], quantified with a thin film XRF reference standard (RF8-200-S2455). Full-field imaging and nanotomography of the grain were also carried out by obtaining projection images at regular angular intervals as the precision stage was rotated through 180˚. These images were aligned and reconstructed using TXM-Wizard [11] to obtain a 3-d view of the grain's internal structure at a spatial resolution of ~25 nm. In the near future, the nXRF and nCT data will be fused to produce a 3-d map of the microstructure and chemistry of the grain. This method is called nano tomography-assisted chemical correlation (nTACCo) and is described in detail in [12].

Results: Figure 1 presents one frame from the movie showing the tomographic reconstruction of the entire graphite grain. A preliminary examination of the nCT and nXRF datasets indicates the presence of several (Ti,V)C subgrains in the host graphite. Figure 2 shows 2-d nXRF maps for Ti-Kα and V-Kα measured at 0˚. The low resolution nXRF maps were able to easily resolve several 50 nm subgrains. Figure 3 shows one frame from the preliminary 3-d nXRF coincidence analysis for the grain. Ti and V signals appear to be correlated, as expected from (Ti,V)C grains often seen in presolar graphite grains [e.g., 8] . The 3-d movie of the chemistry of the grain shows that the Ti, V, and some of the Fe signal originate from the interior of the grain, while the Ca signal is from the surface. The nTACCo analysis that will overlay the 3-d chemical map shown in Figure 3 on the tomographic reconstruction will give a better 3-d picture of the grain. We will present the result of this analysis at the meeting.

The XRF data will also be quantified to obtain maximum elemental concentrations for the entire grain.

Conclusion: Our preliminary data on a presolar graphite grain is promising and demonstrates that nTACCo analysis can be a powerful technique to obtain nanoscale 3-d chemical and microstructural information on presolar grains (and other cosmochemically relevant micro-particles).

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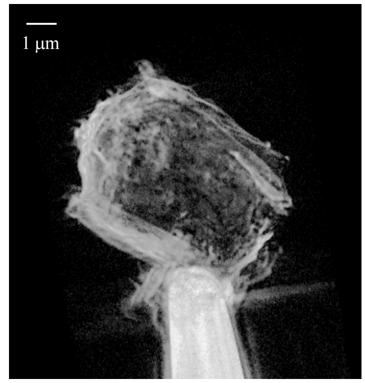

Figure 1: A single frame from the nano tomographic reconstruction of the presolar graphite grain. The movie will be presented at the meeting.

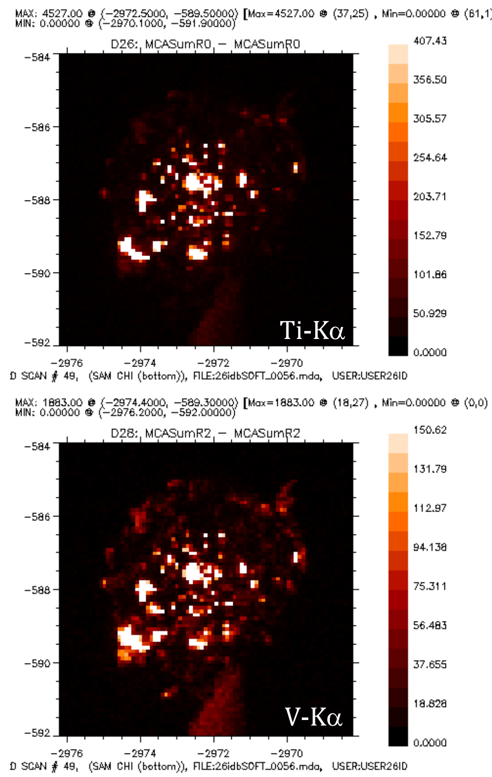

Figure 2: 2-d element maps from nXRF measurements of Ti-Kα and V-Kα at 0˚ orientation.

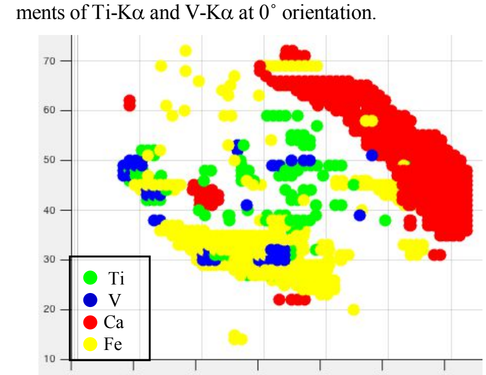

Figure 3: A single frame from the 3-d nXRF map of the graphite grain. This map will be overlaid on the tomography movie and presented at the conference.