EXTRACTION AND ANALYSIS OF A PRESOLAR OXIDE GRAIN FROM THE ADELAIDE UNGROUPED C2 CHONDRITE. T. J. Zega[1] and C. Floss[2] [1]Lunar and Planetary Laboratory, University of Arizona, 1629 E. University Blvd., Tucson, AZ 85721, tzega@lpl.arizona.edu. [2]Washington University in St. Louis, Campus Box 1105, One Brookings Drive, St. Louis, MO 63130, floss@wustl.edu.

Introduction: Grains of dust that condensed in the outflows of ancient stars and survived transport through the interstellar medium were among the starting materials that formed our solar system. Some of these materials were preserved in primitive meteorites and information on their chemistry and structure can provide insights into the thermodynamic and nucleosynthetic histories of their parent stars [1,2]. However, identifying such grains in meteorites and isolating them for analysis is complicated by their small sizes and the fact that they are embedded in a 'sea' of matrix material that is largely solar in origin. A dissolving-the-haystack-in-order-to-find-the-needles approach to studying presolar grains has proven highly successful in identifying several types of circumstellar grains including graphite, nitrides, and oxide stardust [3-5].

While residue samples facilitate the analysis of large numbers of presolar grains, the acid-dissolution process required to produce them destroys their original petrographic context. Thus, we have a relatively poor understanding of the spatial relationships of presolar oxides to the matrix component of the host meteorites in which they occur. Understanding such relationships is important for deciphering the effects of parent-body processing on the grains and distinguishing these from the effects of condensation in their circumstellar envelope(s) and processing in the interstellar medium. Here we report on the in situ extraction and analysis of a presolar spinel grain.

Experimental: Isotopic mapping via secondary ion mass spectrometry (SIMS) revealed 12 presolar oxide grains in the matrix of the Adelaide C2 ungrouped carbonaceous chondrite [6]. We chose one of these grains (7a-1-o1) for microstructural analysis. The grain and surrounding matrix material were extracted and thinned to electron transparency with an FEI Nova 200 focused-ion-beam scanning-electron microscope (FIB-SEM) at Arizona State University using previously described methods [7].

We analyzed the FIB section using the newly developed 200 keV FEI Osiris transmission electron microscope (TEM) at FEI headquarters (Portland, OR). The 'ChemiSTEM' combines a new, high-brightness thermally assisted field-emission gun with a revolutionary new energy-dispersive X-ray spectrometer (EDS) system. The X-field-emission gun (X-FEG) generates up to five times more beam current at a given resolution compared to standard Schottky emitters, and the Super-X EDS system combines four (30 mm^2) silicon-drift detectors integrated into the objective-lens pole-piece gap. This radical new design brings the detectors very close to and around the sample, thus tripling the collection angle from 0.3 steradian (sr) for conventional Si(Li) systems to 0.9 sr [8]. We tested the feasibility of this system for analysis of presolar grains and planetary materials in general.

Results: Automated O-isotopic SIMS mapping showed that a thin section of the Adelaide chondrite contains an area with anomalous O-isotopic composition ($^{17}O/^{16}O=15.7\pm0.2\times10^{-4}$ and $^{18}O/^{16}O=1.88\pm0.02\times10^{-3}$) measuring approximately 570 nm wide [6]. Using the FIB-SEM, we created an electron transparent (≤100 nm) cross section transecting the long axis of the area for detailed microstructural and crystal-chemical analysis with TEM.

Figure 1a shows a bright-field TEM image of the FIB section extracted from the matrix of Adelaide in which presolar oxide grain 7a-1-o1 was previously identified. Using the Super-X EDS system on the ChemiSTEM, we chemically mapped the entire FIB section in 14 minutes and generated over 3×10^7 counts. The EDS map of the entire FIB slice (Fig. 1b) shows that a grain, which spatially correlates with the O-isotopic hotspot is rich in Mg (red) and Al (blue), occurs near to the top of the section (purple false color). High-angle annular-dark-field (HAADF) Z-contrast imaging in scanning TEM (STEM) mode shows that the grain has a saw-tooth shaped morphology and measures approximately 270 nm wide by 304 nm long (Fig. 1c). High-resolution spectrum imaging shows that the Mg and Al spatially correlate with O and minor Fe (cf., Fig. 1d-g), suggesting a spinel composition. Measurements on selected-area electron-diffraction (SAED) patterns from two different orientations (e.g., Fig. 1h) are consistent with the Mg-Al spinel structure, and nanobeam electron-diffraction (NBD) patterns (not shown) acquired across the width and length of the grain at 10 nm intervals, show that it is a single crystal. The intensity of the Fe map appears to be brightest near the bottom edge of the grain (Fig. 1f), suggesting local enrichment of Fe within a ≤50-nm wide band. Surrounding the spinel are Fe-Mg silicates that NBD patterns show are nanocrystalline. The Mg and Si outside of the spinel approximately parallel its grain boundaries.

Discussion: The O-isotopic composition of the spinel places it within the Group-1 field for presolar oxides [see 9, and references therein] and indicates that it formed in the circumstellar envelope (CSE) around a low-mass star that evolved through the red-giant/asymptotic giant-branch of stellar evolution. In comparison, the microstructural data indicate that the

grain conforms to spinel chemistry and structure, which can be used to place constraints on the conditions under which it formed in the CSE. Equilibrium thermodynamic calculations predict that spinel will form from a gas of solar composition, but its purity will vary with condensation temperature. At higher temperature, nearly pure $MgAl_2O_4$ will condense at 1500 K (total pressure, $P_T = 10^{-3}$ atm), but a spinel solid solution is predicted to condense at 1221 K at the expense of Cr-bearing metal, plagioclase, and Mg-silicates [10,11]. The EDS measurements on grain 7a-1-o1 show that it contains Fe, suggesting that it could have condensed at temperatures below 1500 K assuming $P_T \leq 10^{-3}$ atm. Condensation temperature drops with decreasing P_T, and pressures of 10^{-6} atm are probably more realistic for CSEs [12]. However, the Fe in the grain is not uniformly distributed as might be expected if the grain formed under equilibrium condensation [e.g., 13,14]. A possible explanation for the compositional heterogeneity we observe here is that secondary processing of Adelaide matrix redistributed chemical components, such as Fe, which partially affected the chemistry of grain 7a-1-01. We note that many presolar silicate grains in Adelaide were observed to contain elevated Fe contents which led [6] to suggest that thermal metamorphism after dust aggregation may have led to Fe diffusion from the matrix into some of the presolar silicates. The EDS map of the entire FIB section shows that Fe occurs throughout it, but there is a localized band of Fe measuring several microns across in its center (Fig. 1b). Why such localized Fe-rich material would accrete in this manner is unclear; rather, it may be consistent with Fe-redistribution via thermal processing after dust aggregation. If such a process is responsible for the Fe distribution we observe for 7a-1-o1, then the spinel is likely to have originally condensed as pure $MgAl_2O_4$ and thus have recorded a higher condensation temperature.

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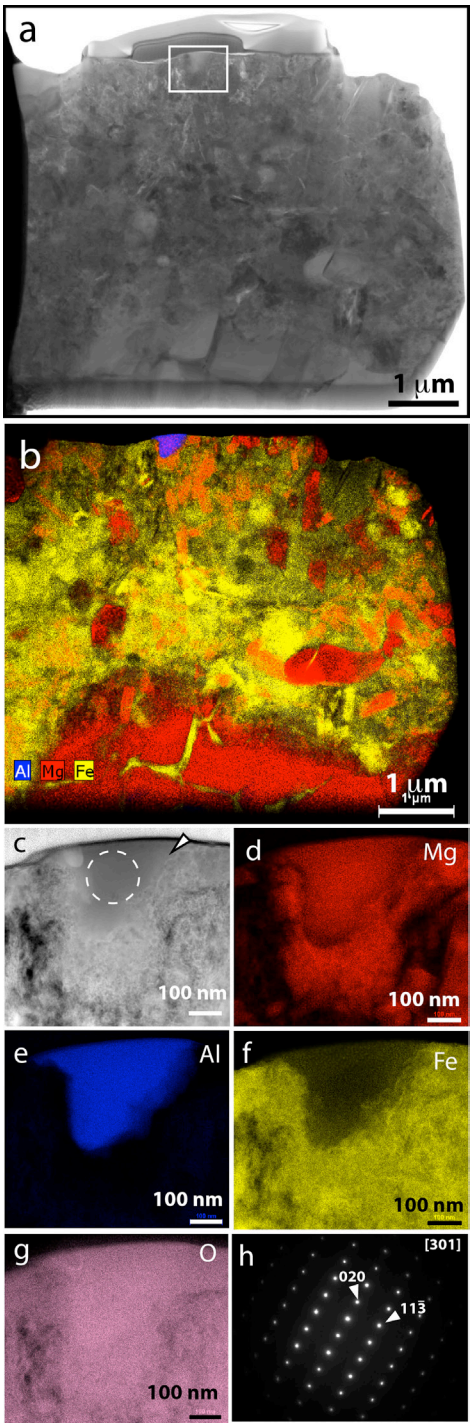

Fig. 1. ChemiSTEM data on Adelaide grain 7a-1-01. **(a)** BF-STEM image of the FIB section. **(b)** EDS map of the FIB section (color legend inset). **(c)** HAADF image of presolar spinel grain (white arrowhead). **(d-g)** Element maps, as indicated. **(h)** SAED pattern acquired from the area outlined by the dashed circle in **(c)**.