**A PRESOLAR SILICATE TRILOGY: CONDENSATION, COAGULATION AND TRANSFORMATION – NEW INSIGHTS FROM NANOSIMS/TEM INVESTIGATIONS** C. Vollmer[1], P. Hoppe[1], F. E. Brenker[2] and C. Holzapfel[3], [1]Max-Planck-Institute for Chemistry, Particle Chemistry Dept., 55020 Mainz, Germany (cvollmer@mpch-mainz.mpg.de), [2]JWG – University, Institute of Mineralogy, 60054 Frankfurt/Main, Germany, [3]Saarland University, Institute of Functional Materials, Saarbruecken, Germany.

**Introduction:** Circumstellar silicates condense in the disks and outflows of young and late type stars and supernovae [e.g. 1]. Small quantities of such grains are recognized in primitive meteorites and interplanetary dust particles (IDPs) by their highly anomalous isotopic compositions. The characterization of these presolar silicates by transmission electron microscopy (TEM) has begun just recently and helps to understand silicate condensation and transformation processes in circumstellar/interstellar environments. Here we report on new results of a combined NanoSIMS/ SEM/ TEM study of presolar silicates detected in the ungrouped carbonaceous chondrite Acfer 094.

**Experimental:** The methods for the identification of presolar silicates by NanoSIMS oxygen isotope mapping have been described in [2]. To prepare electron transparent sections of the grains, we applied the Focused Ion Beam (FIB) technique with an attached lift out tool (Kleindiek MM3A). The grain of interest is first relocated and marked with a small Pt-spot. This simplifies later re-identification because this Pt-spot is clearly visible in subsequent TEM analyses. Second, a row of ~300 nm wide holes are shot into the meteorite along the line of the final section (Fig. 1).

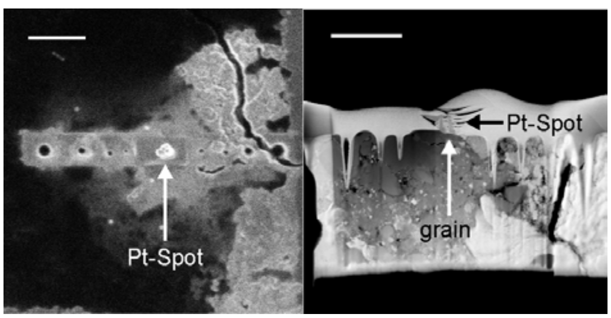


***Figure 1.*** **Left**: FIB/SEM image of the meteorite matrix. The grain is located underneath the Pt-spot. **Right**: STEM image of the grain in the final FIB section with the marking holes on both sides. Scale bars are 2 µm.

The section is milled using a 30 kV $Ga^+$ ion beam, extracted with the nanomanipulator and welded to a half-cut TEM grid in situ. The plane of the section is parallel to the plane of the TEM grid. During final thinning to ~100 nm the holes become visible and provide orientation marks to ensure that the section follows the originally intended line. This new preparation technique therefore minimizes the problem to accurately center submicron grains in a FIB section. TEM investigations were carried out with a 200 kV Philips CM 200. After TEM measurements the grains were successfully relocated in the NanoSIMS.

**Results and discussion:** *Abundance update.* We have identified 65 presolar silicate grains in the fine-grained matrix of an Acfer 094 thin section. Only five of these grains are larger than 500 nm. The matrix-normalized abundance was calculated by dividing the area of the relocated grains as measured in the SEM by the total area of all rastered ion images. Due to sample shift during ion image acquisition the relevant area of the 10x10 $\mu m^2$ sized ion images is only some 9.7x9.7 $\mu m^2$ on average. This gives a presolar silicate abundance in the matrix of Acfer 094 of ~175 ppm (total area analyzed: $3.9 \times 10^4$ $\mu m^2$). This value is remarkably close to the one reported for a grain separate of Acfer 094 [3]. This meteorite is therefore unique in its preservation of presolar silicates as only IDPs yield higher abundances [4]. It is questionable though if abundance calculations from different labs are comparable. 88% of the silicates found so far in this study lie in the range of O isotope groups I-II [5] and come from RGB/AGB stars of maximum two times the solar mass and close-to-solar metallicity (Fig. 2).

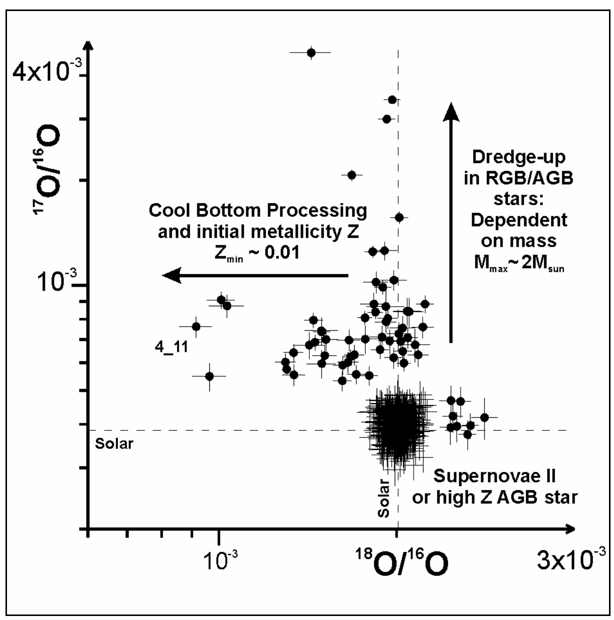


***Figure 2.*** O isotopes of presolar silicates (this study only). Black crosses show matrix scatter. Error bars are 1σ.

Eight grains (12%) belong to group IV and originate most likely from supernovae of type II or high Z AGB stars. No grain was found with an O isotope composition of group III. As these abundances are based on a large data set, we have strong argument that they are representative of the major silicate dust component from which the solar system has formed.

*TEM investigations.* Two grains have been prepared by FIB for further TEM analysis. Grain 8_10 shows moderate $^{17}O$ enrichment and $^{18}O$ depletion ($^{17}O/^{16}O = 8.07 \pm 0.37 \times 10^{-4}$; $^{18}O/^{16}O = 1.77 \pm 0.05 \times 10^{-3}$). It consists of a coagulated cluster of at least 10 small (~50 nm) amorphous silicate particles which form a grain of ~800 nm in diameter (Fig. 3). Amorphous, probably organic mantles separate the grains from each other. It is known that such organic mantles support the coagulation of circumstellar grains [6]. Fe rich particles are also present along grain boundaries and might be of secondary solar system origin [7]. The grains themselves show a wide compositional range (Si content 27-56 mol.%) and confirm that condensation in the fast cooling winds of RGB/AGB stars leads to chemical heterogeneity. The particles have most likely coagulated in the ejecta of the red giant as coagulation in the ISM is very improbable, though not impossible [8]. This grain also proves that transfer times of presolar silicates are very short, as this fragile "microplanet" would not have survived extensive destruction processes in the ISM. HR-SEM imaging of presolar silicates in this study reveal that a lot of these grains are composed of even smaller (<50 nm) subparticles. Therefore, the existence of aggregates is not limited to grains of possible supernova origin [1], but appears to be a more general characteristic of presolar silicates independent from their origins.

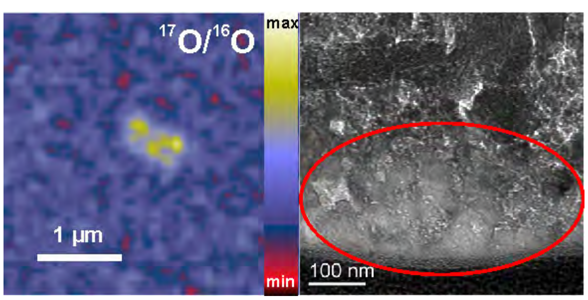


***Figure 3.*** NanoSIMS $^{17}O/^{16}O$ ratio image (left) of the coagulated presolar silicate 8_10 and the corresponding TEM brightfield image of the grain in the FIB section (right).

Grain 4_11 is strongly depleted in $^{18}O$ and moderately enriched in $^{17}O$ ($^{17}O/^{16}O = 7.61 \pm 0.54 \times 10^{-4}$; $^{18}O/^{16}O = 9.16 \pm 0.59 \times 10^{-4}$). It is amorphous as well and contains up to 80 mol.% $SiO_2$ (Fig. 4). Tiny specks of Fe metal grains, 5-10 nm in size, are dispersed inside the glass and are seen either in brightfield imaging mode as diffracting crystals and in energy-filtered TEM mode. Sulfur was not detected at levels >1 mol.% throughout the grain, which indicates that this is not a classical GEMS. It can not be distinguished whether the Fe particles are a secondary product due to parent body or nebular processing [7] or of interstellar or circumstellar origin. There are no obvious particle tracks visible in grain 4_11 which would point to a circumstellar origin. Astronomical observations demonstrate that Fe in amorphous circumstellar silicates can account for the opacity in the near-infrared [9]. Fe is not dispersed homogeneously in grain 4_11, but rather concentrates in discrete metal subgrains. It has been proposed that $Fe_n$ clusters can also act as seed nuclei for silicate condensation in O-rich environments at high pressures [10], which should be considered a possible alternative to subgrains such as $TiO_2$ or $Al_2O_3$.

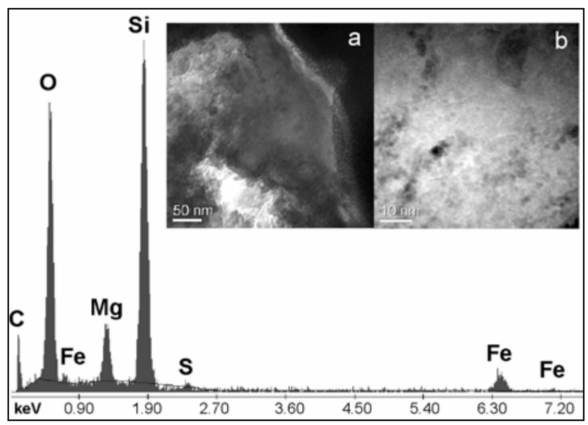


***Figure 4.*** EDX point measurement of presolar silicate 4_11 (acquisition time 100s), FeO content is < 5 mol.% **a**. TEM brightfield micrograph of the grain in the FIB section (Pt strap in the upper right). **b**. Internal Fe subgrains.

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