CORRELATED NANOSCALE STUDY OF A UNIQUE COMPLEX STARDUST GRAIN.

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Introduction: Primitive chondritic meteorites, interplanetary dust particles (IDPs), and cometary dust host small amounts of isotopically anomalous stardust grains that have formed in the outflows of evolved stars [1]. Apart from nanodiamonds, whose origin is still under debate, silicates are the most abundant type of presolar dust, with abundances of ~375 ppm on average in isotopically primitive interplanetary dust particles (IDPs) and of ~200 ppm in the fine-grained matrix of the most primitive meteorites [2]. Oxide stardust is less abundant, with concentrations of up to tens of ppm in meteorites [3,4] and of ~20 ppm for IDPs [5]. A third rare type of O-rich stardust are so-called "complex" grains, which consist of two or more different mineral phases. The first complex presolar grain was reported by [6], consisting of an Al oxide core surrounded by a silicate mantle. To date, less than a dozen of these grains have been identified. Here we report on results from a combined study of an unusually large (0.8×3.8 µm) complex grain from the Krymka meteorite by NanoSIMS, Auger Electron Spectroscopy (AES), SEM-based energy-dispersive X-ray spectroscopy (EDS), and time-of-flight secondary ion mass spectrometry (TOF-SIMS).

Experimental: Grain KRY#I_37 was found during a search for O-anomalous grains with the NanoSIMS 50 at the Max Planck Institute for Chemistry (MPIC). It was then remeasured with high (60 nm) lateral resolution. Secondary ion images (256×256 px, ~130 min integration time) of $^{16,17,18}O^-$, $^{28}Si^-$, and $^{27}Al^{16}O^-$ were collected in multi-collection mode with a 0.1-0.2 pA Cs^+ primary ion beam. For the silicon isotopes, 8 image planes (4 min each) were acquired over a 6×6 μm^2 sized area with a 100 nm Cs^+ beam (1 pA). Mg and Ti isotopes were measured with a NanoSIMS 50L equipped with a new RF plasma O-ion source at Cameca, Paris, with a primary ion current of ~3 pA and a lateral resolution of ~100 nm. Six electron multipliers were used with two different mass settings. First, secondary ion images of $^{24,26}Mg^+$, $^{27}Al^+$, and $^{47,48,49}Ti^+$ were recorded in multi-collection mode. Then, the setting was changed to detect $^{24,25,26}Mg^+$ and $^{27}Al^+$. The elemental composition of the grain was analyzed with the PHI 700 Auger Nanoprobe at Washington University in St. Louis with 10-20 nm resolution. Additional element investigation was carried out by SEM-EDS with a LEO 1530 FE-SEM equipped with a Oxford X-Max 80 SDD EDS-detector at the MPIC. Finally, the grain's composition was studied with the IDLE3 TOF-SIMS instrument at University of Manchester, equipped with a Au cluster liquid metal ion source (lateral resolution ~500 nm).

Results & Discussion: From the $^{28}Si^-$ and $^{27}Al^{16}O^-$ ion images, the complex structure of the grain was clearly visible, with an Al-oxide core and a Si-bearing mantle. KRY#I_37 has an elevated $^{17}O/^{16}O$ ratio ($8.40 \pm 0.16 \times 10^{-4}$) compared to solar system values, and slightly sub-solar $^{18}O/^{16}O$. It belongs to the isotopic Group 1 as defined by [7,8], originating from 1.2–2.2 $M_\odot$ red giant and asymptotic giant branch (AGB) stars of approximately solar metallicity. There is evidence for the presence of initial ^{26}Al with an inferred $^{26}Al/^{27}Al$ ratio of ~7 x 10^{-4}, suggesting formation after the first dredge-up, during one of the early third dredge-up (TDU) episodes. We observed a small excess in ^{30}Si ($\delta^{29}Si$=11±4 ‰, $\delta^{30}Si$=41±5 ‰), while Ti isotopes are indistinguishable from terrestrial values. The Ti-isotopic composition is consistent with a formation during early TDU episodes, while the Si isotopic composition could be attributed to the parent star's initial composition [9]. The O-, Al-, and Ti-isotopic compositions are largely compatible with an origin from a ~1.5 $M_\odot$ AGB star with a metallicity Z~0.02. For this mass and metallicity, the stellar envelope has C/O < 1 even after several TDU episodes, facilitating the formation of O-rich dust [10].

The core of the grain is a heterogeneous assembly of Al-, Ca- and Ti-oxides, while the mantle consists of Mg-Ca-silicate, largely resembling a condensation sequence. A P-hotspot was found within the grain boundaries, and the TOF-SIMS-analysis showed that the P is present as a P-oxide phase. PO molecules have been observed in the circumstellar envelope of the O-rich AGB star IK Tauri [11], thus, the formation of P-oxides should be feasible. To our knowledge, this is the first observation of a P-bearing phase in a presolar grain.

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