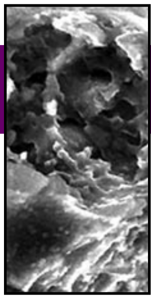

Clues to Stellar Evolution from Microscopy of Star Dust

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INTRODUCTION

Micron-sized dust grains that formed in stars older than the Sun can be found in the matrices of some meteorites.[1] Analysis of the structure and composition of this star dust in the transmission electron microscope (TEM) provides important constraints to stellar condensation models and spectroscopic stellar observations. Additional benefits of these presolar grain studies include the development of techniques for the micromanipulation of critical dust samples and a better understanding of the materials stability in extreme environments.

BACKGROUND

New solar systems form from the dusty residue of older stars. Most of the dust that was the raw material for the formation of our solar system was heavily processed in the early solar nebula. A few grains escaped heavy processing, and retain the isotopic, chemical, and structural record of their presolar origin (Fig. 1). By analyzing the structure of the presolar grains, we are able to infer the environmental conditions of the circumstellar space in which the dust condensed. Thus, microscopy studies of presolar grains provide data for direct constraint of stellar evolution models and for interpretation of spectroscopic measurements of stellar envelopes. Furthermore, presolar grains are test cases for studying the formation and survival of materials in extreme environments, including highly oxidizing atmospheres and heavy radiation fluxes.

GRAIN IDENTIFICATION

Presolar grains are trace constituents of primitive meteorites and can be identified as presolar on the basis of isotopic signatures that deviate from solar values by 100% to 1000% (Fig. 2). Such large variations from solar values can only be explained in terms of nuclear reactions occurring within the parent stars. In comparison, physical and chemical fractionation processes, which explain most of the isotope variations within terrestrial materials, produce variations two to three orders of magnitude lower, i.e., approximately 1% or lower variation from solar values.

To isolate presolar grains from the host meteorite, researchers use physical crushing, acid dissolution treatments or both to obtain a grain residue, then disperse the residue on a sample mount and measure the grain isotopes by secondary ion mass spectrometry (SIMS). The isotope measurements for the NRL presolar grain studies are performed by collaborators at the Carnegie Institution of Washington, Washington, DC; Laboratory for Space Sciences, Washington University in St. Louis; and the Max Planck Institute for Chemistry, Mainz, Germany.

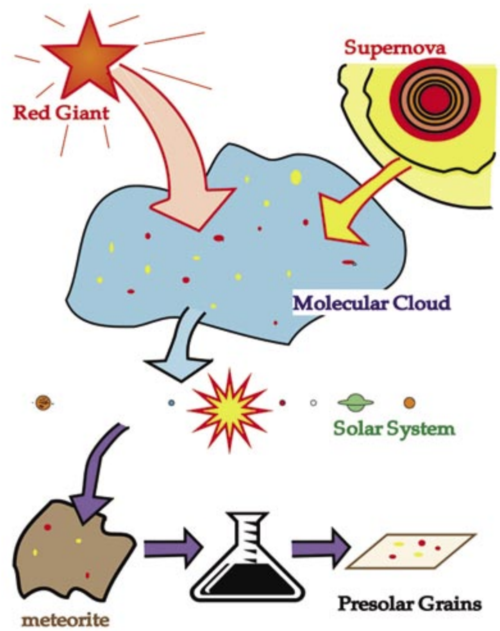


FIGURE 1
Schematic of presolar grain history. Presolar grains formed in the outflows of red giant stars and supernovae. The dust from many stars condensed into a molecular cloud that formed the solar nebula. In the early solar nebula, most material was isotopically homogenized, but the presolar grains escaped this processing and are preserved in asteroids and meteorites. We isolate the presolar grains by acid dissolution or other laboratory processing of meteorites. (Figure courtesy of Dr. Larry Nittler.)

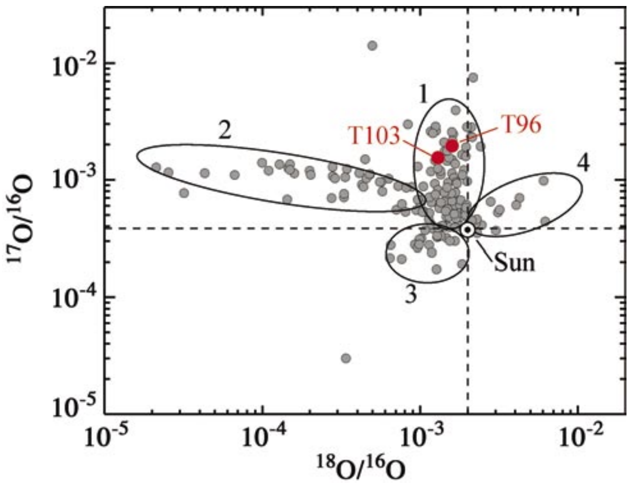


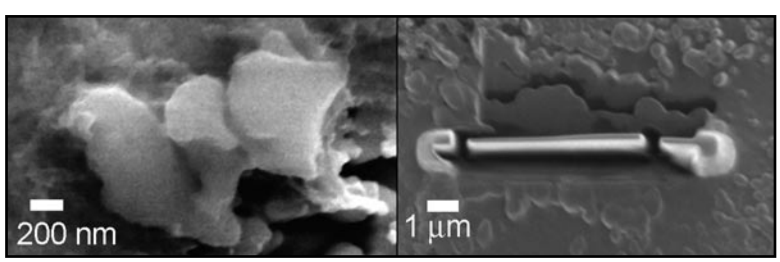
FIGURE 2
Oxygen isotope plot of presolar Al_2O_3 grains. The grains fall into four groups representing four distinct types of progenitor stars. The labels T103 and T96 indicate the isotope values for two grains that we examined in the TEM. (Figure courtesy of Dr. Larry Nittler.)

MICROMANIPULATION OF MICRON-SIZED DUST

After isotopic characterization, the grains must be extracted from the mounts and thinned to electron transparency. This simple sounding task is extremely challenging due to the size of the grains (0.3 to 2 μm), and because the individual isotopically significant grains are surrounded by thousands of isotopically normal (solar) grains. In order to solve this microscopic needle-in-a-haystack problem at NRL, we have adapted focused ion beam (FIB) lift-out techniques traditionally applied to site-specific defect analysis in semiconductor devices (Fig. 3). Using an FIB workstation, we deposit a protective Pt mask (1 μm wide, 10 μm long, and 1 μm high) on the grain of interest. This creates a large enough "handle" that we can manipulate the grain. Next, we use the approximately 10-nm Ga^+ beam to sputter excess material from the grain, creating an electron transparent slice less than 100 nm thick. After cutting the sides and bottom of the slice free using the Ga^+ beam, we take the grain to an optical microscope. There we use a glass needle mounted on a micromanipulator to move the FIB-prepared slice from the SIMS mount to a TEM support grid. This technique for extraction of individual micron-to-submicron grains is not limited to presolar grain analysis, but broadly applicable to the forensic analysis of dust and materials failure analysis, and will be an important use of the newly arrived dual-beam FIB system in the Institute for Nanoscience. A major advantage of this particle-handling technique is that it permits coordinated analyses of the same particles using multiple techniques, e.g., isotope and structure determination.

TRANSMISSION ELECTRON MICROSCOPY

To determine the structure and composition of the grains, we take advantage of the state-of-the-art TEM facilities at NRL, including the new energy-filtered high-resolution TEM housed in the Institute for Nanoscience, which will replace the temporary JEOL 2010F facility in early 2005. For greater flexibility in tilt angle for diffraction analysis, the Philips CM30, housed in the Materials Science and Technology Division, is also used.



(a) (b)

FIGURE 3
(a) Secondary electron image of a presolar oxide grain. (b) Secondary electron image of a focused-ion-beam-prepared thin section of the grain ready for lift-out.

RESULTS

To date, we have investigated four different kinds of presolar grains: Al_2O_3 grains from O-rich asymptotic giant branch (AGB) stars, SiC from C-rich AGB stars, SiC from type-II supernovae, and silicates from O-rich AGB stars. The information to be gained from the grains depends on the grain composition and structure, as well as the stellar origin. The most refractory, radiation hard materials, i.e., Al_2O_3 and SiC, primarily provide information about the circumstellar environments in which the dust condensed. The less refractory silicates are likely to reflect both the condensation environment and the alteration processes by which dust is converted from molecular clouds to the solar nebula to rocky materials, i.e., asteroids, planets, and planetesimals.

PRESOLAR AL_2O_3

Aluminum oxide is a cosmically important phase because many equilibrium thermodynamic calculations predict it to be the first solid phase to condense in O-rich AGB stars. However, the formation of solid Al_2O_3 is kinetically inhibited because Al_2O_3 molecules are rare in the gas phase, and there is a 30-yr debate in the astrophysical community over whether other oxide phases, such as TiO_2, form first. Furthermore, it has typically been assumed that when Al_2O_3 condenses, it takes the stable corundum (α-Al_2O_3) structure, despite the established laboratory synthesis of many metastable structures close in free energy.

A parallel debate in the observational astronomy community concerns the identification of broad and narrow features at 12.5 μm in the infrared spectra of some AGB stars. Some astronomers have attributed these features to amorphous and corundum forms of Al_2O_3, while others have argued for assignment to spinel or polymerized silicate phases.

Our research helps resolve both of these controversies by providing data from direct laboratory study of the star dust in question (Fig. 4). Our TEM studies of two presolar aluminum oxides show that at a minimum, both corundum (Fig. 5) and amorphous forms of Al_2O_3 form in O-rich AGB stars, without prior formation of any seed phases such as TiO_2. Our data also indicate that Ti could be important in determining the structure, as we find detectable levels of Ti in the corundum grain, and not in the amorphous grain. The results of this study appeared in the September 3, 2004 issue of *Science*.[2]

SiC FROM C-RICH AGB STARS

One of the mysteries of presolar SiC is how it survives the highly oxidizing, O-rich plasma of the early solar system, without sublimation of the C to form CO gas. On Earth, SiC is extremely rare except as a synthetic material, but it is a large fraction of the dust produced by C-rich AGB stars. The structure of AGB SiC extracted from meteorites by acid treatments has been well studied by NRL staff scientist Dr. Tyrone Daulton, Code 7431.[3] However, the acid treatments drastically alter the grain surfaces, and remove all the surrounding meteoritic material, including any protective surface coatings (Fig. 6). Our FIB-based sample micromanipulation techniques allow us to extract thin sections of SiC grains located in situ in the host meteorite, or as physical separates, without the use of acids, thus preserving the grain context and surface.

The TEM results from the "pristine" (i.e., not acid exposed) AGB SiC shows that the SiC can indeed survive the early solar nebula without the formation of a protective oxide layer. The grain shapes range from heavily faceted to well-rounded (Fig. 7). The surrounding meteoritic materials show no orientation or compositional relationship to the enclosed SiC grains. This indicates that the accretion of the SiC

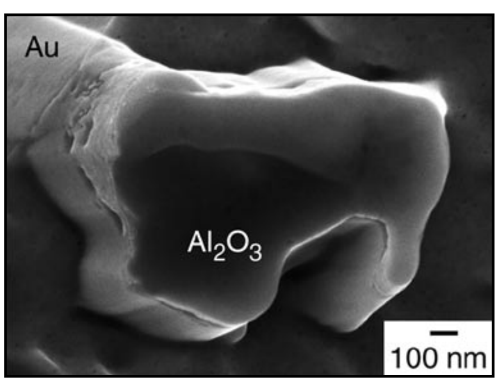


FIGURE 4
Scanning electron micrograph of a presolar Al_2O_3 grain.

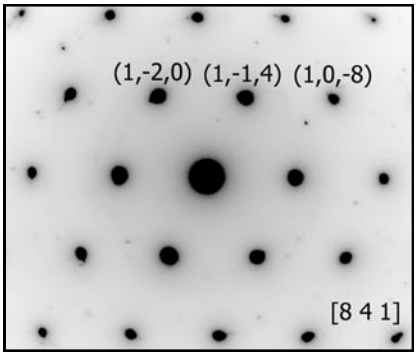


FIGURE 5
Electron diffraction pattern of a presolar Al_2O_3 grain. The indexing of the pattern confirms that the grain has the corundum crystal structure.

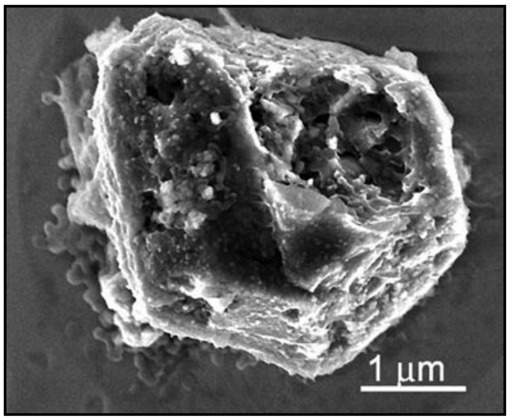


FIGURE 6
Scanning electron micrograph of an acid-treated presolar SiC grain.

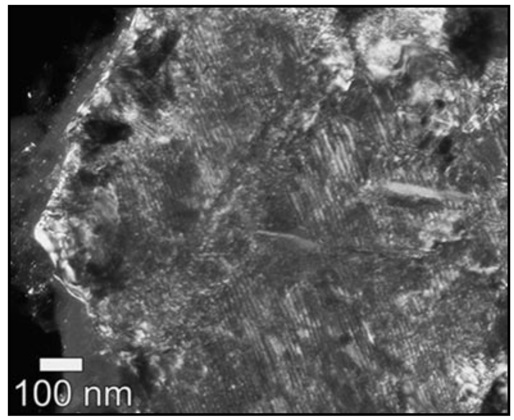


FIGURE 7
Transmission electron micrograph of an in situ presolar SiC grain.

dust onto the asteroid parent body occurred at a great enough distance from the center of the solar nebula (> 2 a.u.) that the ambient temperature was < 900 K. A surprise find in the SiC grains was an interior Ru-bearing subgrain in one SiC and many 100-nm graphite subgrains in another SiC. The occurrence of these subgrains was not observed in the acid-treated grains, possibly because the acid treatment removes subgrains, but should aid in the study of C-rich AGB star atmospheres.

SUPERNOVA SiC

Type-II supernovae are among the most extreme environments known. They are literally the rapid explosion of astronomical-sized nuclear reactors, initiated by the collapse and reexpansion of the core of massive stars (> 8 × solar mass), after they become too Fe-rich to support continued nuclear fusion. During the supernova event, most of the mass of the star is ejected in the form of jets of gas and dust. These events are the largest production source of the elements heavier than He in the galaxy. The isotope ratios of grains from supernovae are even more different from the solar materials than even the pre-solar AGB stars. One class of supernova SiC is known as the "X grains" due to the "eXtreme" isotope values. Prior to our study, it was not clear whether the supernova origin of the X grains was reflected in the structure of the grains as well as in the isotopes. In other words, do the highly nonequilibirum condensation conditions and high radiation fluxes leave specific signatures on the crystal structure? Our TEM study of two X grains provides an affirmative answer. Unlike the AGB SiC grains that are all essentially single crystals of approximately 1 µm in diameter with varying levels of defects, the X grains are agglomerates of many smaller crystallites. We have observed both a dense aggregate of 10-nm crystallites and a porous aggregate of 100-nm crystallites. In addition, the dense aggregate shows evidence of directional recrystallization of the grains due to both radiation and physical shock, two events characteristic of supernovae (Fig. 8).

AGB SILICATES

Silicate minerals constitute the greatest fraction of the dust that condenses around O-rich AGB stars, but

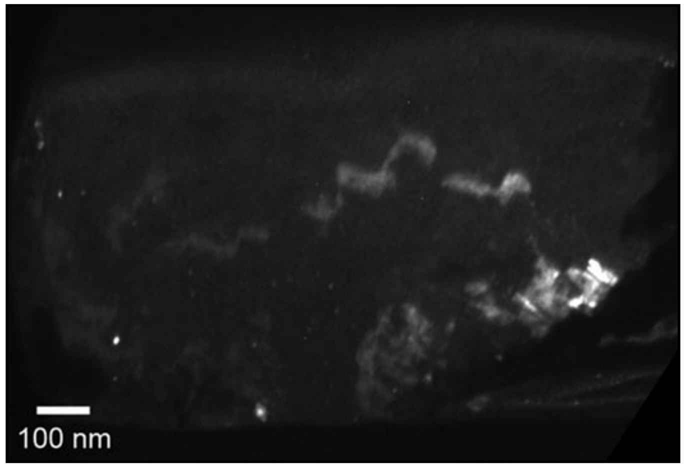


FIGURE 8
Dark-field transmission electron micrograph of a supernova SiC grain. The white "smoke trail" that crosses the grain highlights oriented recrystallization due to a radiation event.

presolar silicates have been the hardest of the presolar grains to identify. The biggest difficulties have been (1) that the host meteorites are primarily made up of silicate minerals with a solar, rather than presolar origin, so that thousands of individual isotope measurements must be made to locate a single presolar grain, and (2) that the spatial resolution of the SIMS instruments has only recently reached the submicron range required to measure these grains individually.

We have examined the structure of one presolar silicate (Fig. 9), and it was revealed to be glassy, with a composition not corresponding to a known mineral stoichiometry. This immediately raises the issue of whether this grain condensed as a nonstoichiometric amorphous grain, or was structurally and chemically altered in the trajectory from stellar condensate to meteorite inclusion. Solar silicate minerals are known to have experienced varying degrees of thermal and aqueous alteration due to the processing that occurred during and after accretion of the individual grain onto the meteorite parent body (asteroid). At some point, heavy alteration would completely erase the presolar isotopic signature, but low levels of structural and compositional alteration might preserve a distinctly nonsolar isotope composition. By comparing the isotope signatures with the structural and compositional data from more presolar silicate grains, we aim to learn about (a) the dust that was the biggest fraction of the materials that went into the initial formation of our solar system, and (b) the processes that converted those raw materials from dust to larger bodies.

SUMMARY

The microscopy of star dust is a mixture of extraterrestrial materials science and astronomy in the laboratory. We literally have samples of star dust that we can experimentally trace to stars of specific class and composition in order to better understand the stars themselves and the formation of our own solar

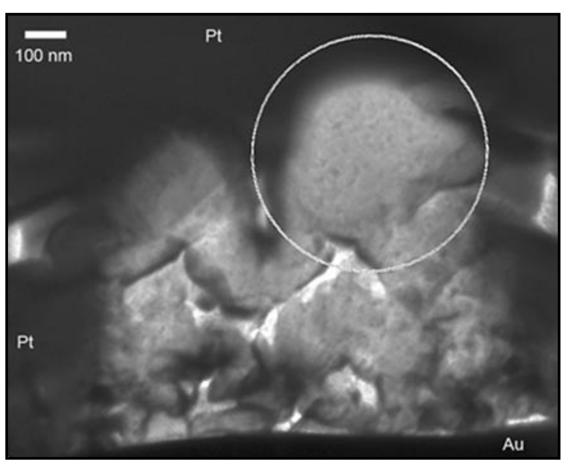


FIGURE 9
Transmission electron micrograph of a presolar silicate grain.

system. The tools, e.g., the high-resolution analytical transmission electron microscope and focused ion beam workstation, that we use for these analyses are more conventionally applied to the development of materials for new electronic devices. The same fundamental laws of nature apply to materials that condense inside a laboratory vacuum chamber as in circumstellar space. By putting presolar grains under the electron microscope, we are helping to answer some of the big picture questions that have long challenged astronomers, i.e., how does stellar material get recycled from old stars to new stars. In addition, we are building a knowledge base about the long-term survival of electronic insulators (Al_2O_3) and conductors (SiC) in space, and determining protocols to handle submicron grains for parallel analytical studies. This is a prime example of the synergistic relationship between cutting-edge research in seemingly unrelated fields, and the unexpected collateral benefits of interdisciplinary basic research.

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