

# ANCIENT STARDUST IN THE LABORATORY

**A**mazingly, individual grains of dust from stars that existed before the Sun was born have made their way to Earth in meteorites. When subjected to a battery of cutting-edge laboratory techniques, these tiny grains provide thrilling new insights into such topics as the dynamics of supernova explosions, the age and chemical evolution of the Galaxy, fundamental nuclear physics and processes in the outer envelopes of stars. The path from dust grain to astrophysical insight is the subject of this article.

For the last half century, several key ideas have dominated how we think nuclides were formed and then distributed in nature. In the 1950s, Hans Suess and Harold Urey showed that it is possible to determine the average abundances of elements and isotopes in the Solar System from laboratory measurements of primitive meteorites. These meteoritic abundances turned out to be similar to those measured spectroscopically in the Sun. It was also realized that very few nuclides could have been produced in an initial "big bang" and that most element synthesis is in fact the result of nuclear reactions in stars. And ever since the publication of the two classic papers in 1957—one by Alastair Cameron, the other by Geoffrey and Margaret Burbidge, Willy Fowler and Fred Hoyle—we have known that the abundances of elements and isotopes in the Solar System must reflect contributions from many different types of stars.<sup>1</sup>

Until recently, it was believed that the contributions of individual stars to the solar mix could not be determined directly. This belief followed from the fact that closely similar isotopic ratios were measured in a wide variety of terrestrial and extraterrestrial samples, suggesting that all presolar solids had been vaporized when the Solar System formed. Complete isotopic homogenization was the result. We now know this is not quite true. Surprisingly, some individual grains of stardust survived the formation of the Solar System and can be isolated from primitive meteorites.

How can one identify a grain as ancient stardust? Because the solar isotopic compositions are the grand averages of contributions from many individual stars, any

**As it collapsed to form the Solar System, material in the solar nebula was churned up and homogenized. But not everything was lost in the mix . . .**

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grain of surviving stardust should generally have isotopic compositions in one or more elements that are markedly different from the solar values. Indeed, this is probably the *only* way ancient stardust can be distinguished from solids that formed within the Solar System.

Identifying individual stardust grains is just one

aspect of the larger study of isotopic anomalies in meteorites (an anomaly being defined as a significant deviation from a well-determined solar isotopic ratio). In themselves, isotopic anomalies do not necessarily imply that their carriers are stardust, for most anomalies are actually quite small and arise from well-understood processes. For example, there are anomalies due to nuclear spallation reactions in materials exposed to high-energy cosmic rays in space. And other anomalies—characterized by large enrichments of the heavy isotopes of hydrogen and nitrogen—could be carried by remnants of chemically processed material from interstellar molecular clouds. However, here we are concerned only with isotopic anomalies that are so huge that they constitute clear evidence that the grains carrying them are samples of ancient stardust—that is, dust that formed around stars other than our Sun before the Solar System was born 4.6 billion years (Ga) ago.

The variations in isotopic composition among individual presolar grains demonstrate that many stars of several different types have contributed to the Solar System mix, beautifully confirming the basic tenets of nucleosynthesis, as the theory of element formation is known.

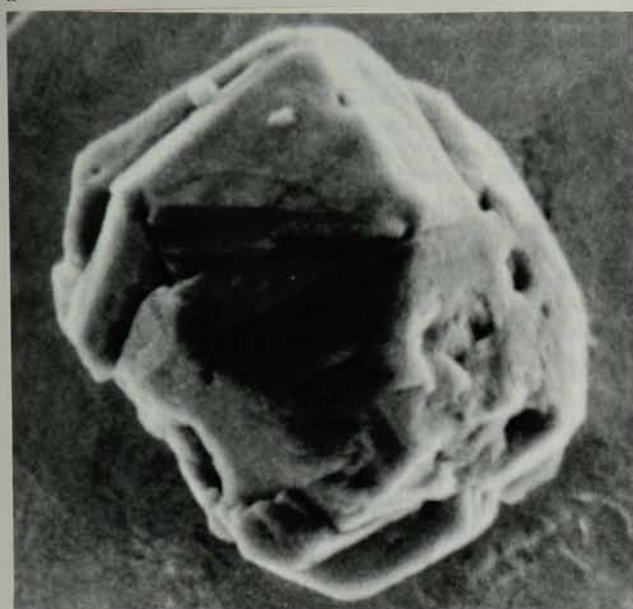
Presolar grains identified so far include aluminum oxide ( $\text{Al}_2\text{O}_3$ ), graphite spherules, silicon carbide (SiC), silicon nitride and a variety of metal carbides found as small inclusions in larger presolar grains. Each species of grain provides new information that must be integrated into the existing body of astrophysical knowledge. This eclectic process requires the work and insights of astronomers, theoretical astrophysicists and laboratory scientists.<sup>2</sup>

## Searching for stardust

Presolar grains are so small (typically about  $1 \mu\text{m}$ ) and constitute such a minor fraction of meteorites by weight (typically less than five parts per million) that their presence was overlooked when the first measurements were made on gram-sized samples. Hydrogen isotopic anomalies were measured in meteorites as early as 1954, but it was not until the early 1970s that isotopic evidence of presolar grains was found. As with other important developments in meteoritics (for example, the discovery of the decay products of extinct radionuclides), isotopic stud-

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**FIGURE 1. STARDUST IN THE LABORATORY**, as evident in electron micrographs. **a:** A presolar silicon carbide crystal from the Murchison meteorite. The  $^{12}\text{C} / ^{13}\text{C}$  ratio of this crystal is 39—radically different from the solar value of 89, which tags it as a grain that originated outside the Solar System. This crystal most likely condensed in the expanding atmosphere of a star on the asymptotic giant branch. It measures about  $4\ \mu\text{m}$  across. (Photo courtesy of Sachiko Amari.) **b:** A 70 nm thick section of a presolar graphite spherule. The crystal in the center of the spherule is titanium carbide, a refractory mineral that formed before the graphite and served as a nucleation center for its growth. The dark, radial spokes in the image are an electron diffraction effect; in reality, the graphite layers are concentric.

ies of noble gases were the key to understanding.

In a landmark paper describing his thesis work done with Robert Pepin at the University of Minnesota, David Black identified several isotopically distinct components in meteorite samples heated to progressively higher temperatures.<sup>3</sup> One sample was very enriched in neon-22, a discovery that led Black to suggest that surviving presolar material was its host.

Subsequent work using various physical and chemical treatments of primitive meteorites produced samples that were even richer in  $^{22}\text{Ne}$ , buttressing the idea that an unknown mineral phase carried essentially pure  $^{22}\text{Ne}$ . One possible mechanism for the production of such an isotopically distinct component is the incorporation of radioactive  $^{22}\text{Na}$  as a minor constituent of condensing dust grains. This nuclide, which has a half-life of 2.6 years, decays to give pure  $^{22}\text{Ne}$ . Stepwise heating experiments by Peter Eberhardt at the University of Bern further showed that there were at least two separate carriers of the  $^{22}\text{Ne}$ -rich gases, one of which released Ne at low temperatures and the other did so at high temperatures.

Studying xenon and krypton in addition to neon, Edward Anders and Roy Lewis and their coworkers at the University of Chicago discovered a remarkable fact: Isotopically exotic noble gases were not removed by the acid dissolutions that destroyed most of the meteorite. The interpretation that these gases resided in acid-resistant carrier grains of presolar material was enormously strengthened when it was shown that the relative abundances of the xenon isotopes measured in one distinctive xenon component closely matched theoretical predictions made by Donald Clayton and Richard Ward for s-process nucleosynthesis.<sup>4,5</sup> (See box 1 on page 29 for a description of the various kinds of nucleosynthesis.)

Using noble gases as tracers of presolar grains—in a manner reminiscent of how the Curies isolated radium from radioactive minerals—the Chicago group developed procedures for producing mineral separates rich in various

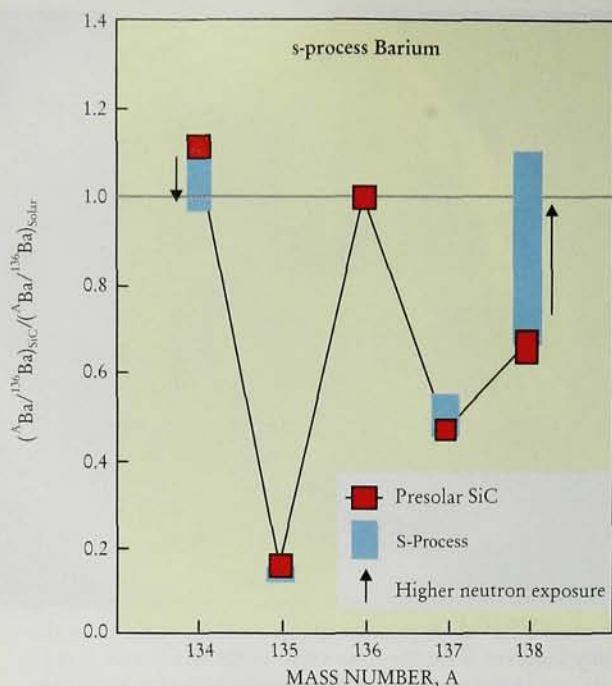
exotic components. Their technique included dissolving most of the meteorite in a variety of solvents—a process that Anders has described as “burning down the haystack to find the needle.” One such separate, which was characterized by enrichments in both the heavy and light isotopes of xenon, was found to consist primarily of nanodiamonds with a median grain size of 1–3 nm. This discovery was the first identification of a specific carrier phase for a component of exotic noble gas.<sup>6</sup>

At the time of this discovery in 1987, the Chicago scientists began collaborating with our group in St. Louis. We had a variety of instruments specifically tailored for microanalytic studies of extraterrestrial materials and had been actively searching for presolar grains—principally in interplanetary dust particles, but also in acid residues of primitive meteorites. As luck would have it, we concentrated at first on the meteorite Allende, which we now know is essentially devoid of circumstellar grains large enough to measure individually. (See box 2 on page 29.) Although a large SiC grain had been found in one of the residues, it proved to have a normal isotopic composition and was undoubtedly a terrestrial contaminant.

Fortunately, the same was decidedly not true of the abundant, micrometer-sized SiC grains found in the Chicago separate of the Murray meteorite, which are rich in  $^{22}\text{Ne}$ . Ernst Zinner, who is the director of the Washington University ion probe laboratory, pioneered the development of techniques to make isotopic measurements on these small grains (see box 3 on page 30). He quickly found that each SiC grain had silicon and carbon isotopic ratios that differed by astonishingly large amounts from the Solar System average.<sup>7</sup> With this discovery, the laboratory study of individual grains of stardust was born. (We recall wryly a reviewer's description of our original proposal to make ion probe isotopic measurements of grains in meteorite separates as “a poorly focused fishing expedition unlikely to lead to interesting scientific results.”)

In 1990, Sachiko Amari and Roy Lewis's physical and





**FIGURE 2. ISOTOPIC PATTERN OF BARIUM** measured by Frank Podosek and coworkers in collections of presolar silicon carbide grains (pink), compared with predictions made by Roberto Gallino and coworkers for the composition of barium created in s-process nucleosynthesis (gray). Gallino found that the Ba composition could be reconciled with s-process theory only by revising the neutron capture cross sections for  $^{135}\text{Ba}$ ,  $^{136}\text{Ba}$  and  $^{137}\text{Ba}$ —a proposition that was vindicated in lab experiments. What the figure shows is that when the revised cross sections are used, the neutron exposure—as indicated by the width of the gray bars and the arrows—must have been lower for the presolar grains than for average Solar System material.

chemical processing of the Murchison meteorite at Chicago indicated that the presolar carriers of the  $^{22}\text{Ne}$  released at low temperatures were micrometer-sized spherules of graphite (see figure 1). Amazingly, grain-to-grain carbon isotopic ratios of these spherules varied by more than three orders of magnitude!<sup>18</sup> That SiC and graphite were indeed the carriers of the  $^{22}\text{Ne}$ -rich components was proven by our colleagues Robert Nichols and Charles Hohenberg, who successfully measured the Ne isotopes in individual grains.

### Stellar parents: how many and what kind?

Thanks to astronomical observations, we know that most dust in the Galaxy (constituting about 1% of the mass of a typical giant molecular cloud, or  $10^3$ – $10^4 M_{\odot}$  per cloud, where  $M_{\odot}$  is the Sun's mass) is produced by red giant stars—specifically, red giants in the thermally pulsing asymptotic giant branch (AGB) phase of their evolution.<sup>9</sup>

In the early AGB phase, the outer envelopes of these stars are rich in oxygen. Some of this oxygen ends up in silicate and oxide dust, which condenses into material that flows from the outer envelopes into the interstellar medium. However, as the stars age, the envelopes of the low-mass ( $M \leq 4 M_{\odot}$ ) AGB stars become rich in carbon due to the convective dredge-up of material produced by nuclear reactions in the hot interior. Once the envelopes' carbon-to-oxygen ratio exceeds unity, carbonaceous dust like SiC can condense.

Several lines of evidence confirm that most presolar SiC comes from low-mass AGB stars. First, such stars are believed to be the sites of s-process nucleosynthesis. This is consistent with measurements of the minor elements barium, strontium, neodymium and samarium in ensembles of presolar SiC grains that show characteristic enrichments in s-process isotopes. (See box 1 and figure 2.) The evidence for s-process nucleosynthesis has been nicely extended by recent analyses of molybdenum and zirconium isotopes in individual presolar SiC grains at Argonne by Gunther Nicolussi, Andrew Davis and Roy Lewis, who used resonant ionization mass spectrometry to study these elements separately.

But perhaps the most compelling evidence for the AGB origin of most grains is the close match between two distributions of  $^{12}\text{C}/^{13}\text{C}$  ratios—namely, that of presolar SiC grains and that measured remotely in carbon stars today. The similarity of these distributions, which are shown in figure 3, further emphasizes that the SiC grains are not simply from a single presolar star. Rather, they constitute a sample from at least several tens of AGB stars that injected this matter into the molecular cloud from which our Solar System formed. Input from many AGB stars is also implied by the compositional trends of Si isotopes revealed by ion microprobe analyses of hundreds of presolar SiC grains (figure 4).

Detailed analysis of the oxygen isotopic compositions of presolar corundum ( $\text{Al}_2\text{O}_3$ ) grains indicates that most of these grains also originated in red giant stars. Oxides pose a formidable experimental problem because, unlike either graphite or SiC grains, most oxide grains were formed within the Solar System and therefore have a normal isotopic composition. The first task of the experimenter is therefore to find the rare presolar oxide grains in a sea of isotopically normal material.

Pursuing an alternative method—a laborious grain-by-grain isotopic analysis—Gary Huss of Caltech discovered, in 1992, the first presolar oxide grain in the meteorite Orgueil.<sup>10</sup> Later, as part of his PhD thesis with our group, Larry Nittler located another 90 of these grains (out of around 50 000 grains tested!) using the more efficient technique of ion imaging. (See box 3.)

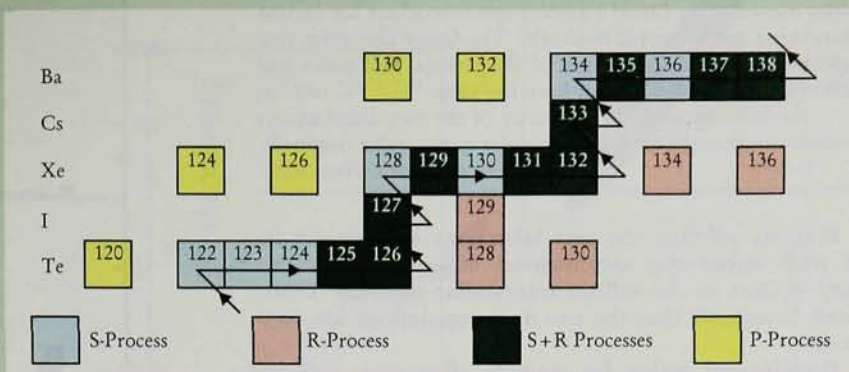
The oxygen isotopes in corundum grains are consistent with those predicted to result from the first dredge-up. (See figure 5.) At this stage of its evolution, the star becomes a red giant, and deep convection mixes the ashes of main sequence nucleosynthesis into its outer envelope. As shown in figure 5, the variation in the isotopic composition of oxygen among these grains indicates that they formed in at least several tens of red giant parents, each having a different initial mass and metallicity. (Metallicity is the mass fraction of atoms of elements heavier than and including carbon, which astronomers call metals.)

An interesting outgrowth of these studies is the realization that the oxygen compositions can be used to determine the age of the Galaxy.<sup>11</sup> The basic idea, as devised by Nittler and Ramanath Cowsik (head of the Indian Institute of Astrophysics in Bangalore and a frequent visitor to our group), is that one can estimate the time required for the average metallicity of the Galaxy to evolve to the stage of that inferred for the parent stars, as well as use stellar evolution models to determine the life span of parent stars having the inferred masses. Since grains are expelled from their parent stars at the end of the stars' lives, the Galaxy's age is given by summing the known age of the Solar System (4.6 Ga), the stellar life span and the metallicity evolution time. The Galactic age derived in this way (14–15 Ga) is nicely consistent with



## Box 1: Nucleosynthesis in the Xenon-Barium Region

This section of the chart of nuclides shows the stable nuclei (but including iodine-129, which has a half-life of 16 million years) in the xenon-barium region, indicating various nucleosynthesis mechanisms responsible for their production. Rapid neutron addition in supernovae explosions (known as the r-process) produces neutron-rich nuclei that ultimately beta-decay to stable isobars. Supernovae are also responsible for the p-process nuclei that are produced by photodissociation of more neutron-rich nuclei. Slow neutron addition (known as the s-process) occurs primarily in low-mass stars at low neutron densities. In this process, neutrons from ( $\alpha, n$ ) reactions, in which oxygen-16 is made from carbon-13, build successively heavier isotopes, until an unstable nuclide is reached that can beta-decay before the next neutron is added, as shown in figure 2. The s-process path is indicated by the dark line in the figure. Note that some nuclides are made in both the s- and r-processes.



the ages that have been obtained from various astronomical studies (for example, of globular clusters). Inadequacies in the theoretical models make the age uncertain by several billion years, but the reliability of the estimate will improve if better models of stellar and Galactic chemical evolution can be constructed.

Presolar grains provide direct evidence that supernovae also contributed to the solar mix. About 1% of the presolar SiC grains were originally given the noncommittal name X grains because of their extremely exotic silicon isotopes—specifically, an excess of  $^{28}\text{Si}$ , which is produced when oxygen burns deep in the interior of massive stars. One way to remove this silicon to where it can form grains is by blasting the star apart in a supernova. Two decades ago, Donald Clayton noted that short-lived titanium-44, which has a half-life of about 60 years, is produced exclusively in explosive nucleosynthesis. Therefore, if  $^{44}\text{Ti}$  could be incorporated in a forming grain, its complete decay might result in measurable excesses of its daughter calcium-44, thereby earmarking the grain as a supernova condensate. And indeed, large excesses of  $^{44}\text{Ca}$  have been detected in X grains.<sup>12</sup> Some graphite grains also appear to have been produced in supernova explosions.

The X grains provide unique insights into the hydrodynamical behavior of exploding stars. Isotopic measurements of different elements in single grains show that nuclear products from the deep stellar interior (for example,  $^{28}\text{Si}$  and  $^{44}\text{Ca}$ ) must have been joined with carbon-rich material from outer stellar layers, reflecting extensive mixing at all length scales. This finding augments inferences from astronomical observations of supernovae like 1987A, which indicate large-scale mixing in the ejecta.

Presolar grains thus provide new information about nucleosynthesis and the nature of the stellar sources in which it occurs, sometimes in astonishing detail. As a further example, Roberto Gallino and his coworkers in Turin, Italy, showed that the composition of s-process barium in presolar SiC (figure 2) mandated revisions in the standard neutron capture cross sections by as much as 50% for several Ba isotopes—a conclusion vindicated by subsequent laboratory measurements.

Various isotopically distinct subgroups of presolar grains have been found in addition to those considered above. The result is what some refer to as the alphabet soup of grain studies—for example, SiC types X, Y, Z, A, B; group 4 oxide grains and so on. Indeed, there may be

almost as many subtypes of grains as there are subtypes of stars in the Galaxy. Relating the various grain subtypes to specific kinds of stellar objects such as novae and the very hot, very luminous Wolf-Rayet stars is a continuing enterprise that should deepen the understanding of a wide variety of astronomical objects.

### Forming stellar grains

The laboratory studies directly measure the chemical and physical properties of ancient stardust. Some of these properties—for example, grain size distributions and external morphologies—must also reflect the rigors of transit from the circumstellar birthplace to the laboratory environment.

## Box 2: Presolar Grains and Primitive Meteorites

Meteorites fall into three major groups: stones, stony-irons and irons. The last two groups, as well as some stones, are made up of material that has suffered substantial chemical differentiation caused by heating in large asteroidal bodies. This material is therefore not likely to contain undigested presolar grains.

The chondrites, a subgroup of the stones, have more primitive textures and compositions than the stony-irons and irons. They get their name from the presence of spherical silicate chondrules (*chondros* is Greek for grain) that were once probably molten droplets. More specifically, carbonaceous meteorites are chondrites that have not been subjected to significant thermal metamorphism in their asteroidal parent bodies, as evidenced by their having compositions similar to that of the solar photosphere in all but the most volatile elements. Their comparative primitiveness is confirmed by the observation that they contain the highest concentrations of presolar grains.

Allende and Murchison are carbonaceous meteorites that fortuitously fell in the same year (1969, in Mexico and Australia, respectively). These carbonaceous meteorites, as well as Murray (which fell in 1950 in Kentucky) and Orgueil (which fell in 1864 in France), have been particularly important in the search for presolar grains because many kilograms of each were recovered—enough for the dissolution experiments on gram-sized samples needed to isolate presolar grains.



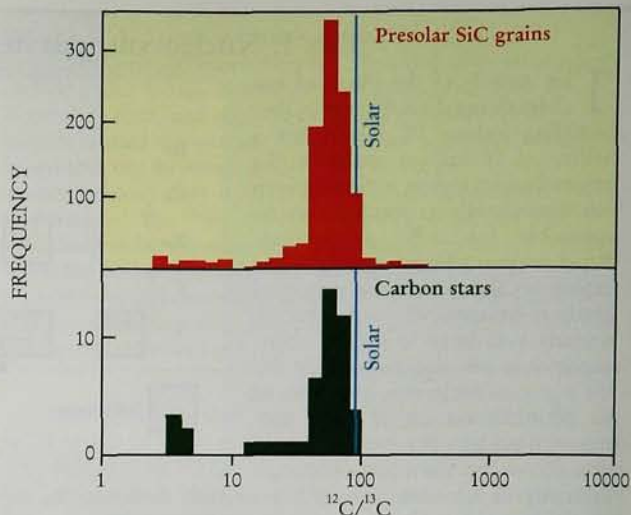
**FIGURE 3. COMPARING THE DISTRIBUTION OF  $^{12}\text{C}/^{13}\text{C}$  ratios among presolar silicon carbide grains with astronomical values measured by David Lambert and coworkers for carbon stars using spectroscopic methods. The figure illustrates two important findings. First, most of the presolar SiC grains and carbon stars measured do not have the same  $^{12}\text{C}/^{13}\text{C}$  ratio as the Sun. Second, the similarity of the two distributions demonstrates that the carbon in presolar grains came originally from many presolar carbon stars.**

It turns out that the new laboratory data do not fit well with either the astronomical observations or the theory of dust in the diffuse interstellar medium (ISM). Indeed, it appears that the two dust populations are very different.

Presolar SiC grains, for example, often show well-defined crystal faces, indicating a lack of severe erosion and pulverization. Contradicting these findings, existing theories of ISM dust predict that interstellar shocks quickly erode and destroy grains. Moreover, the characteristic size inferred for ISM dust from the scattering of starlight is ten times smaller than the roughly 1 micrometer size typical of presolar grains. Finally, neither very small, needle-like graphite nor silicate grains have yet been identified in the presolar grain population—even though polarization measurements indicate their presence in interstellar dust. A variety of possible explanations exists, but finding the right one remains a major goal.

Whatever the explanations, presolar grains do exist, and—as first demonstrated by Jerry Wasserburg (Caltech), Chris Sharp (Service d'Astrophysique, Centre d'Etudes de Saclay) and our colleagues Katharina Lodders and Bruce Fegley Jr—they can be used to infer the temperatures, pressures and times required to produce them in stellar outflows.<sup>13</sup>

For example, transmission electron microscope studies of ultrathin sections of presolar graphite spherules show that the spherules are often nucleated on small (5–100 nm) crystals of very refractory carbides like tita-

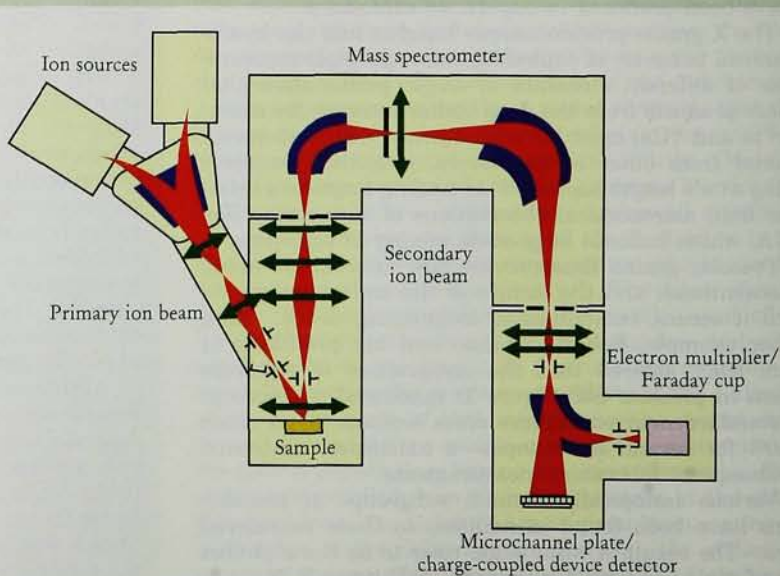


nium carbide and zirconium carbide (figure 1b). But they also show that less refractory carbides like SiC do not generally occur within the graphite. According to equilibrium thermodynamics, the inferred condensation sequence—the very refractory carbides first, then graphite, then SiC—can occur only at total pressures greater than about 0.1 Pa, which is roughly a hundred times greater than pressures generally assumed to exist in the regions of AGB atmospheres where grains condense.

Considering the kinetics of ideal grain growth yields the same conclusion. However, higher pressures in a stellar atmosphere inevitably lead to proportionally higher rates of mass loss. To avoid contradicting astronomically measured AGB mass loss rates, the pressures inferred from presolar grain studies must be assumed to pertain not to smooth, spherically symmetric stellar outflows, but to irregularly distributed blobs of material that are sporadically ejected from stellar surfaces. Interestingly, this

### Box 3: Ion Microprobe Isotopic Analysis

The imaging ion microprobe, invented by Georges Slodzian of the University of Paris—South, works by firing a fine beam of primary ions (typically either  $\text{Cs}^+$  or  $\text{O}^+$  accelerated by 10 kV) at a sample to produce a plume of secondary ions that acts as the source of ions for a double-focusing mass spectrometer. In conventional high-mass resolution analyses, the secondary ion beam is intercepted by a Faraday cup or electron multiplier detector. The ion optics of the instrument are such that a defocused beam of primary ions illuminating a sample (for example, a collection of small grains) produces a magnified real image of the object area in mass-selected secondary ions. In the ion imaging technique, an isotopic image of the sample is formed on a microchannel plate—fluorescent screen detector, then digitized with a charge-coupled device camera. An image formed in one isotope can be compared with an image in another isotope, so that isotopic ratios for the whole field of view can be calculated using image processing. One can then zero in on isotopically anomalous grains and perform detailed analyses, often on more than one chemical element.





conclusion is in agreement with recent studies of maser emission in AGB atmospheres that similarly require large-scale blobbiness in the outflowing matter.<sup>14</sup>

## Presolar organic molecules

The isolation of circumstellar grains permits experiments that were previously impossible. One example is the identification of indigenous organic molecules in individual circumstellar graphite grains.

Polycyclic aromatic hydrocarbons (PAHs) are detected with great sensitivity in the two-step laser desorption-laser ionization mass spectrometer developed by Richard Zare and his collaborators at Stanford University. Although the mass spectra are complex, in favorable situations  $^{12}\text{C}/^{13}\text{C}$  ratios can be measured in individual molecules desorbed from single circumstellar graphite grains. For some grains, our student Scott Messenger has found that the isotopic ratios of the molecules correlate well with those of the atoms in the parent graphite grain. Those organic molecules are clearly indigenous. However, most of the molecules found in the graphite grains have normal isotopic ratios and must have been added after the grains themselves formed.

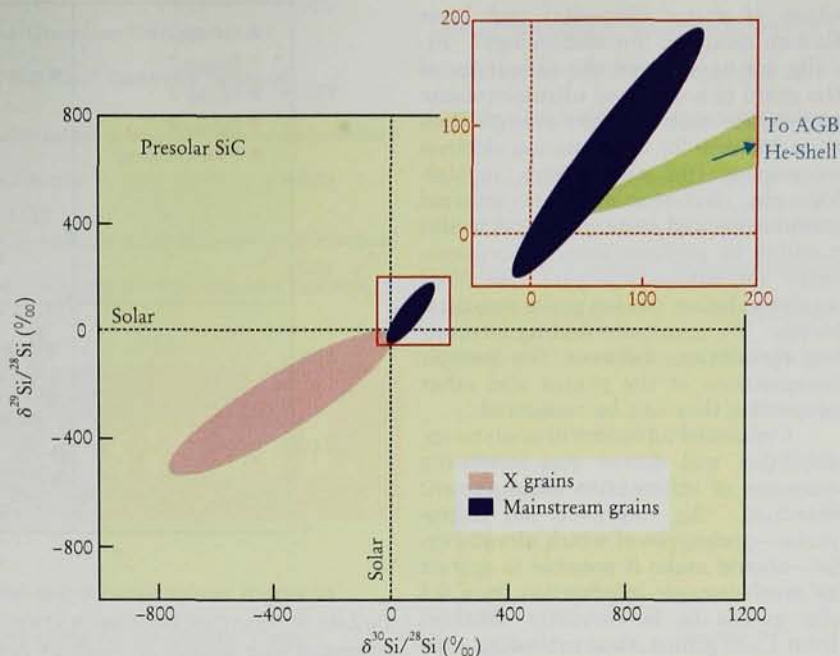
Progress in understanding where and how the indigenous molecules formed—whether simultaneously or later than the grains (for example, by radiation processing in interstellar space)—will depend on the extent to which the indigenous molecules can be isolated from the isotopically normal components.

## Probing the early Solar System

Presolar grains provide a new way to study various processes in the early Solar System. Whether measured directly or indirectly, both the absolute and relative concentrations of different presolar components are found to vary in different types of meteorites. The source of these differences probably lies in how and where the grains were processed—in the solar nebula and in asteroidal meteorite parent bodies.

Using the abundances of anomalous noble gases as tracers of presolar grain content, Gary Huss and Roy Lewis have found that the abundance of presolar grains progressively decreases as the metamorphic grade of meteorite increases.<sup>15</sup> No presolar phases are found in meteorites of the highest metamorphic grades—undoubtedly because presolar material is destroyed in the asteroidal parent bodies of the meteorites. But even in meteorites of the same type and metamorphic grade, the quantity and characteristics of their presolar grains can differ. These differences could reflect grain processing in the solar nebula. For example, different size distributions of presolar SiC observed in different meteorites suggest pre-accretionary sorting by size.

Electron-beam mapping can be used to locate presolar grains *in situ*. Use of this technique avoids the undesirable destruction of diagnostic surface features by the harsh



**FIGURE 4.** SILICON ISOTOPIC RATIOS measured in presolar silicon carbide grains, in terms of departures from solar ratios in parts per thousand. The grains are separable into two major compositional fields (and several additional minor fields, not shown)—namely, mainstream and X grains. The mainstream grains are thought to originate around stars on the asymptotic giant branch and have excesses of both  $^{29}\text{Si}$  and  $^{30}\text{Si}$ . By contrast, the X grains, which probably originate in supernova ejecta, have  $^{28}\text{Si}$  excesses relative to either of the other two silicon isotopes. Of the total number of SiC grains analyzed, X grains represent only about 1%, whereas mainstream grains represent about 98%. The mainstream field is defined by isotopic compositions measured in about 700 individual grains. The X grain field is defined from measurements on about 100 grains located by ion imaging. (See box 3.) The inset displays the compositional trend expected when silicon of solar composition in the AGB stellar envelope is mixed with silicon produced in the helium shell. This trend cannot account for the observed distribution along a straight line of slope of 1.3 found in mainstream grains. It is thought that this spread, instead, reflects Galactic chemical evolution as sampled by multiple AGB stars of different age that contributed SiC grains to the solar mix.

chemical treatments normally used to isolate presolar grains. To our surprise, with electron-beam mapping we have found that presolar SiC occurs as individual grains that lack the silicon oxide rims expected from exposure of SiC to the oxidizing nebula at high temperatures.<sup>16</sup> We do not yet know whether this means that conditions in the solar nebula prevented the formation of silicon oxide rims or whether they were once present and were subsequently removed.

## Some future perspectives

It seems to us that, although now a decade old, the study of ancient stardust is only just beginning. And much remains to be understood about the data already obtained. Explaining the slope of the correlation line that characterizes 98% of the SiC grains in the silicon three-isotope plot (figure 4) has emerged as a major mystery. We also see a number of ways to amplify the study of stardust in the laboratory. If nothing else, it will be important simply to expand the coordinated studies on single grains that already use most of the techniques currently available.

For example, we have already performed sequentially—on single, micrometer-sized grains—scanning electron microscopy (for morphology and chemistry), ion microprobe analyses (for chemistry and for isotopic compo-



sition of major elements) and laser Raman analyses (for mineralogy). Finally, we have sliced the remainder of the grain in a diamond ultramicrotome to produce sections thin enough (less than 100 nm) for transmission electron microscopy (for composition, crystallography, electronic structure, internal morphology and mineralogy). It is also possible to perform laser desorption-laser ionization analysis (for PAH analysis) before the ion probe measurements. We anticipate finding interesting correlations between the isotopic compositions of the grains and other properties that can be measured.

Continued advances in analytic capabilities will derive ever-increasing amounts of information from ancient stardust. An improved ion microprobe—prototypes of which already exist—should make it possible to extract as much isotopic information from 0.1  $\mu\text{m}$  grains as is currently obtained from 1  $\mu\text{m}$  grains, thus extending stardust studies into a size range characteristic of dust seen in most astrophysical settings. Increased instrumental sensitivity will also permit isotopic measurements of a greater number of minor and trace elements in larger single grains.

Complementary laser resonant ionization techniques, as demonstrated by the Chicago group and their collaborators at Argonne with the instrument CHARISMA, will also extend the range of isotopic measurements, making it possible to test nucleosynthesis theories in ever more exquisite detail. A specific case in point is the prediction, which is now being tested experimentally, that SiC X grains should be enriched in the r-process nuclide  $^{96}\text{Zr}$ . Of course, the number of atoms present in small grains sets a fundamental limit on the feasibility of isotopic measurements. But we are still a long way from realizing this limit, even though the increasing pace of discovery indicates that the limit is perhaps not too far off.

New, nondestructive techniques to locate interesting grains should increase the types of presolar grains that can be studied. Such techniques will be particularly important for studying presolar silicate grains (none of which have yet been isotopically identified in meteorites) that are destroyed by the etching treatments used to isolate chemically resistant presolar phases. It could prove possible to use ion imaging techniques to locate silicates in meteorites, as well as other types of interesting grains. For example, dust from oxygen-rich stars is estimated to be as prevalent as dust from carbon stars, yet 100 times more carbon-rich than oxygen-rich material has been identified so far. This "missing" material could reside in grains currently too small to measure individually, or perhaps in silicates rather than oxides.

The integration of the stardust data with emerging information on the isotopic properties of the high-energy and low-energy cosmic rays that sample the local interstellar medium should lead to deeper insights into the production and evolution of nuclides. Streams of interstellar dust currently intersecting the Solar System have been reported. Comparison of the isotopic properties of these grains with those of the ancient dust discussed here would certainly be

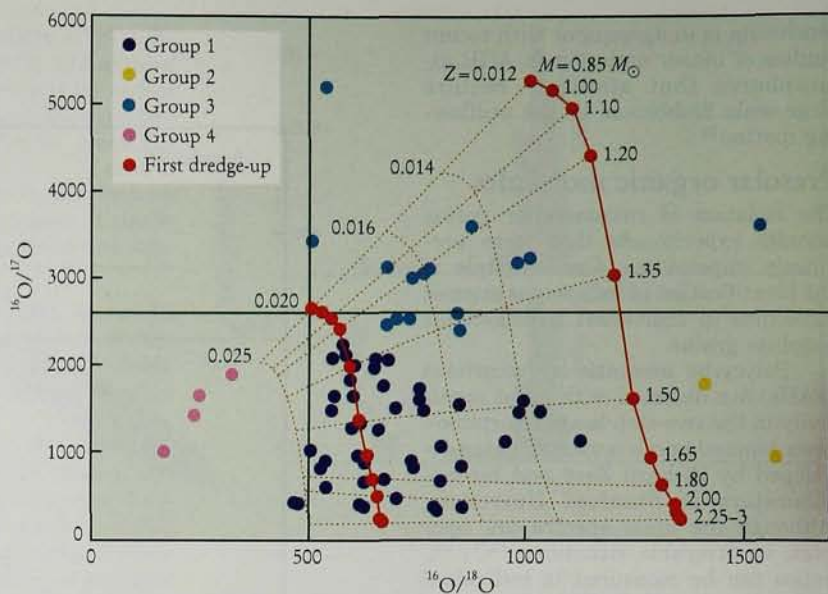


FIGURE 5. OXYGEN ISOTOPIC RATIOS in presolar corundum ( $\text{Al}_2\text{O}_3$ ) grains, compared with predictions made by Arnold Boothroyd and Irene-Juliana Sackmann for compositions resulting from dredge-up of main sequence nucleosynthesis products into the stellar envelope, as a function of stellar mass  $M$  and initial metallicity  $Z$  ( $Z$  is the mass fraction of atoms heavier than and including carbon). Grains of group 1 have oxygen compositions similar to those observed in oxygen-rich red giant stars. Group 3 grains have no observed stellar counterparts, but theory suggests that they (as well as group 2 grains) originated in low-mass red giants. The group 3 oxygen compositions indicate that when the parent stars of these grains were born, the average  $^{17}\text{O}$  and  $^{18}\text{O}$  abundances in the Galaxy were lower than when the Sun was born, reflecting Galactic chemical evolution. As described in the text, these compositions can be used to constrain the age of the Galaxy. Group 4 grains are of unknown origin. (Figure courtesy of Larry Nittler.)

interesting and seems within the realm of possibility.

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