1.02
Presolar Grains

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1.02.1 INTRODUCTION

Traditionally, astronomers have studied the stars by using, with rare exception, electromagnetic radiation received by telescopes on and above the Earth. Since the mid-1980s, an additional observational window has been opened in the form of microscopic presolar grains found in primitive meteorites. These grains had apparently formed in stellar outflows of late-type stars and in the ejecta of stellar explosions and had survived the formation of the solar system. They can be located in and extracted from their parent meteorites and studied in detail in the laboratory. Their stellar origin is recognized by their isotopic compositions, which are completely different from those of the solar system and, for some elements, cover extremely wide ranges, leaving little doubt that the grains are ancient stardust.

By the 1950s it had been conclusively established that the elements from carbon on up are produced by nuclear reactions in stars and the classic papers by Burbidge *et al.* (1957) and Cameron (1957) provided a theoretical framework for stellar nucleosynthesis. According to these authors, nuclear processes produce elements with very different isotopic compositions, depending on the specific stellar source. The newly produced elements are injected into the interstellar medium (ISM) by stellar winds or as supernova (SN) ejecta, enriching the galaxy in "metals" (all elements heavier than helium) and after a long galactic history the solar system is believed to have formed from a mix of this material. In fact, the original work by Burbidge *et al.* and Cameron was stimulated by the observation of regularities in the abundance of the nuclides in the solar system as obtained by the study of meteorites (Suess and Urey, 1956). Although providing only a grand average of many stellar sources, the solar system abundances of the elements and isotopes (Anders and Grevesse, 1989; Grevesse *et al.*, 1996; see Chapter 1.03; Lodders, 2003) remained an important test for nucleosynthesis theory (e.g., Timmes *et al.*, 1995).

In contrast, the study of stellar grains permits information to be obtained about individual stars, complementing astronomical observations of elemental and isotopic abundances in stars (e.g., Lambert, 1991), by extending measurements to elements that cannot be measured astronomically. In addition to nucleosynthesis and stellar evolution, presolar grains provide information about galactic chemical evolution, physical properties in stellar atmospheres, mixing of SN ejecta and conditions in the parent bodies of the meteorites in which the grains are found.

This new field of astronomy has grown to an extent that not all aspects of presolar grains can be treated in detail in this chapter. The interested reader is therefore referred to some recent reviews (Anders and Zinner, 1993; Ott, 1993; Zinner, 1998a,b; Hoppe and Zinner, 2000; Nittler, 2003) and to the compilation of papers found in Bernatowicz and Zinner (1997). The latter not only contains several detailed review papers on presolar dust grains but also a series of chapters on stellar nucleosynthesis. Further information on nucleosynthesis can be obtained from the textbooks by Clayton (1983b) and Arnett (1996), and from reviews by Käppeler *et al.* (1989), Meyer (1994), and Wallerstein *et al.* (1997).

1.02.2 HISTORICAL BACKGROUND

Although the work by Burbidge *et al.* (1957) and Cameron (1957), and subsequent work by nuclear astrophysicists made it clear that many different stellar sources must have contributed to the material that formed the solar system and although astronomical observations indicate that some of this material was in the form of interstellar (IS) grains (e. g., Mathis, 1990), it was generally believed that it had been thoroughly homogenized in a hot solar nebula (Cameron, 1962). The uniform isotopic composition of all available solar system material seemed to confirm this opinion.

The first evidence for isotopic heterogeneity of the solar nebula and a hint of the survival of presolar grains came from hydrogen (Boato, 1954) and the noble gases xenon (Reynolds and Turner, 1964) and neon (Black and Pepin, 1969; Black, 1972) but it was only after the discovery of anomalies in oxygen, a rock-forming element (Clayton *et al.*, 1973), that the concept of survival of presolar material in primitive meteorites was widely accepted. The finding of ^{16}O excesses was followed by the detection of isotopic anomalies in other elements such as magnesium, calcium, titanium, chromium, and barium in refractory inclusions (CAIs for calcium- and aluminum-rich inclusions) (Wasserburg, 1987; Clayton *et al.*, 1988; Lee, 1988). Also large anomalies in carbon (Halbout *et al.*, 1986) and nitrogen (Lewis *et al.*, 1983) indicated the presence of presolar grains. However, it was the pursuit of the carriers of the "exotic" (i.e., isotopically anomalous) noble gas components of neon and xenon (Figure 1) by Ed Anders and his colleagues at the University of Chicago that led to their ultimate isolation (see Anders and Zinner, 1993). The approach taken by these scientists, "burning down the haystack to find the needle," consisted of tracking the noble gas carriers through a series of increasingly harsher chemical dissolution and physical separation steps (Tang and Anders, 1988b; Amari *et al.*, 1994). Their effort culminated in the isolation and identification of diamond, the carrier of Xe-HL (Lewis *et al.*, 1987), silicon carbide, the carrier of Ne-E(H) and Xe-S

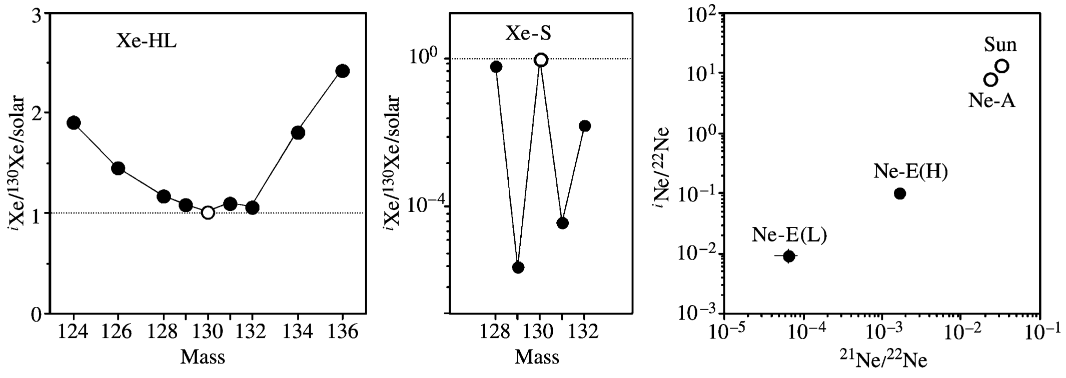

Figure 1 Exotic noble gas components present in presolar carbonaceous grains. Diamond is the carrier of Xe-HL, SiC the carrier of Xe-S and Ne-E(H), and graphite the carrier of Ne-E(L) (source Anders and Zinner, 1993).

(Bernatowicz *et al.*, 1987; Tang and Anders, 1988b), and graphite, the carrier of Ne-E(L) (Amari *et al.*, 1990).

Once isolated, SiC and graphite (for diamond, see below) were found to be anomalous in *all* their isotopic ratios, and it is this feature that identifies them as presolar grains. This distinguishes them from other materials in meteorites such as CAIs that also carry isotopic anomalies in some elements but, in contrast to bona fide stardust, formed in the solar system. They apparently inherited their anomalies from incompletely homogenized presolar material. Another distinguishing feature is that anomalies in presolar grains are up to several orders of magnitude larger than those in CAIs and match those expected for stellar atmospheres (Zinner, 1997).

1.02.3 TYPES OF PRESOLAR GRAINS

Table 1 shows the types of presolar grains identified as of early 2000s. It also lists the sizes, approximate abundances, and stellar sources. In addition to the three carbonaceous phases that were discovered because they carry exotic noble gas components (Figure 1) and which can be isolated from meteorites in almost pure form by chemical and physical processing, presolar oxide, silicon nitride (Si_3N_4), and silicates were identified by isotopic measurements in the ion microprobe and the number of such grains available for study is much smaller than for the carbonaceous phases. Most oxide grains are spinel ($MgAl_2O_4$) and corundum (Al_2O_3), but hibonite ($CaAl_{12}O_{19}$) and possibly titanium oxide have also been found (Hutcheon *et al.*, 1994; Nittler *et al.*, 1994; Nittler and Alexander, 1999; Choi *et al.*, 1998; Zinner *et al.*, 2003). While all these grains, as well as presolar Si_3N_4 (Nittler *et al.*, 1995), were located by single grain analysis of acid residues, presolar silicates were discovered by isotopic imaging of chemically untreated interplanetary dust particles (IDPs) (Messenger *et al.*, 2003).

Finally, titanium-, zirconium-, and molybdenum-rich carbides, cohenite ($(Fe,Ni)_3C$), kamacite (Fe–Ni), and elemental iron were found as tiny subgrains inside of graphite spheres (Bernatowicz *et al.*, 1991, 1996, 1999; Croat *et al.*, 2003). While TiC inside of an SiC grain (Bernatowicz *et al.*, 1992) could have formed by exsolution, there can be little doubt that interior grains in graphite must have formed prior to the condensation of the spherules.

1.02.4 ANALYSIS TECHNIQUES

Although the abundance of carbonaceous presolar grains in meteorites is low, once they are identified, almost pure samples can be prepared and studied in detail. Enough material of these phases can be obtained for "bulk" analysis, i.e., analysis of collections of large numbers of grains either by gas mass spectrometry (GMS) of carbon, nitrogen, and the noble gases (Lewis *et al.*, 1994; Russell *et al.*, 1996, 1997), by thermal ionization mass spectrometry (TIMS) of strontium, barium, neodymium, samarium, dysprosium (Ott and Begemann, 1990; Prombo *et al.*, 1993; Richter *et al.*, 1993, 1994; Podosek *et al.*, 2003) or secondary ion mass spectrometry (SIMS) (Zinner *et al.*, 1991; Amari *et al.*, 2000). While only averages over many grains are obtained in this way, it allows the measurement of trace elements such as the noble gases and heavy elements that cannot be analyzed otherwise.

However, because presolar grains come from different stellar sources, information on individual stars is obtained by the study of single grains. This challenge has been successfully taken up by the application of a series of microanalytical techniques. For isotopic analysis, the ion microprobe has become the instrument of choice. While most SIMS measurements have been on grains 1 μm in size or larger, a new type of ion probe, the NanoSIMS, allows measurements of grains an order of magnitude smaller (e.g., Zinner *et al.*, 2003). Ion probe analysis has led to the discovery of new types of presolar grains such as corundum (Hutcheon *et al.*, 1994; Nittler *et al.*, 1994) and silicon nitride (Nittler *et al.*, 1995). It also has led to the identification of rare subpopulations of presolar dust such as SiC grains

Table 1 Types of presolar grains in primitive meteorites and IDPs.

Grain type	*Noble-gas components*	*Size*	*Abundance*	*Stellar sources*
Diamond	Xe-HL	2 nm	1,000 ppm	Supernovae?
Silicon carbide	Ne-E(H), Xe-S	0.1–20 μm	10 ppm	AGB, SNe, J-stars, novae
Graphite	Ne-E(L)	1–20 μm	1–2 ppm	SNe, AGB
Oxides		0.15–3 μm	1 ppm	RG, AGB, SNe
Silicon nitride		0.3–1 μm	~3 ppb	SNe, AGB
Ti-, Fe-, Zr-, Mo-carbides		10–200 nm		SNe
Kamacite, iron		~10–20 nm		SNe
Olivine		0.1–0.3 μm		

of type X (Amari *et al*., 1992) and type Y (Hoppe *et al*., 1994). Searches for presolar oxide grains and rare subpopulations of SiC profited from the application of isotopic imaging in the ion probe, which allows the rapid analysis of a large number of grains (Nittler *et al*., 1997). Laser ablation and resonant ionization mass spectrometry (RIMS) (Savina *et al*., 2003b) has been successfully applied to isotopic analysis of the heavy elements strontium, zirconium, molybdenum, and barium in individual SiC and graphite grains (Nicolussi *et al*., 1997, 1998a,b,c; Savina *et al*., 2003a). Single grain measurements of helium and neon have been made by laser heating and gas mass spectrometry (Nichols *et al*., 2003).

The surface morphology of grains has been studied by secondary electron microscopy (SEM) (Hoppe *et al*., 1995). Such studies have been especially useful for pristine SiC grains that have not been subjected to any chemical treatment (Bernatowicz *et al*., 2003). Finally, the transmission electron microscope (TEM) played an important role in the discovery of presolar SiC (Bernatowicz *et al*., 1987) and internal TiC and other subgrains in graphite (Bernatowicz *et al*., 1991). It has also been successfully applied to the study of diamonds (Daulton *et al*., 1996) and of polytypes of SiC (Daulton *et al*., 2002, 2003).

1.02.5 ASTROPHYSICAL IMPLICATIONS OF THE STUDY OF PRESOLAR GRAINS

There are many stages in the long history of presolar grains from their stellar birth to their incorporation into primitive meteorites and, in principle, the study of the grains can provide information on all of them.

The isotopic composition of a given circumstellar grain reflects that of the stellar atmosphere from which the grain condensed. The atmosphere's composition in turn is determined by several factors: (i) by the galactic history of the material from which the star itself formed, (ii) by nucleosynthetic processes in the star's interior, and (iii) by mixing episodes in which newly synthesized material is dredged from the interior into the star's envelope. In supernovae, mixing of different layers with different nucleosynthetic history accompanies the explosion and the ejection of material. The isotopic compositions of grains provide information on these processes.

Grain formation occurs when temperatures in the expanding envelope of red giants (RGs) or in SN ejecta are low enough for the condensation of minerals. Many late-type stars are observed to be surrounded by dust shells of grains whose mineral compositions reflect the major chemistry of the gas (e.g., Little-Marenin, 1986). The study of morphological features of pristine grains, of internal grains, and of trace-element abundances can give information on the physical and chemical properties of stellar atmospheres (Bernatowicz *et al*., 1996; Amari *et al*., 1995a; Lodders and Fegley, 1998; Kashiv *et al*., 2001, 2002; Croat *et al*., 2003).

After their formation as circumstellar grains or as SN condensates, grains enter a long journey through the ISM. They should be distinguished from true IS grains that form in the ISM, e.g., in dense molecular clouds. Grains of stellar origin are most likely to be covered by mantles of IS cloud material. During their IS history, grains are subjected to a variety of destructive processes, such as evaporation in SN shocks and sputtering by shocks and stellar winds. They are also exposed to galactic cosmic rays that leave a record in the form of cosmogenic nuclides (Tang and Anders, 1988a; Ott and Begemann, 2000).

Grains might go in and out of IS clouds before some were finally incorporated into the dense molecular cloud from which our solar system formed. The final step in the complex history of stellar grains is the formation of planetesimals and of the parent bodies of the meteorites in which we find these presolar fossils. By far the largest fraction of the solids, even in primitive meteorites, formed in the solar system and the fraction of surviving presolar grains is small (see Table 1). Primitive meteorites experienced varying degrees of metamorphism on their parent bodies and these metamorphic processes affected different types of presolar grains in different ways. The abundance of different grain types can thus give information about conditions in the solar nebula and about parent-body processes (Huss and Lewis, 1995; Mendybaev *et al*., 2002).

1.02.6 SILICON CARBIDE

Silicon carbide is the best-studied presolar grain type. It has been found in carbonaceous, unequilibrated ordinary, and enstatite chondrites with concentrations ranging up to ~10 ppm (Huss and Lewis, 1995). Most SiC grains are less than 0.5 μm in diameter. Murchison is an exception in that grain sizes are, on average, much larger than those in other meteorites (Amari *et al*., 1994; Huss *et al*., 1997; Russell *et al*., 1997). This difference is still not understood but it, and the fact that plenty of Murchison is available, is the reason that by far most measurements have been made on Murchison SiC. Many SiC grains show euhedral crystal features (Figure 2) but there are large variations. Morphological studies by high-resolution SEM (Bernatowicz *et al*., 2003) reveal detailed crystallographic features that give information about growth conditions. Such information is also obtained from TEM studies that show that only the cubic (3C) (~80%) and hexagonal (2H) polytypes are present, indicating low pressures and condensation temperatures in stellar outflows (Bernatowicz *et al.*, 1987; Daulton *et al.*, 2002, 2003). A preponderance of cubic SiC has been observed astronomically in carbon stars (Speck *et al.*, 1999).

The availability of >1 μm SiC grains and relatively high concentrations of trace elements (Amari *et al.*, 1995a) allow the isotopic analysis of the major and of many trace elements in individual grains. In addition to the major elements, carbon and silicon, isotopic data are available for the diagnostic (in terms of nucleosynthesis and stellar origin) elements nitrogen, magnesium, calcium, titanium, the noble gases, and the heavy refractory elements strontium, zirconium, molybdenum, barium, neodymium, samarium, and dysprosium. Carbon, nitrogen, and silicon isotopic as well as inferred ^{26}Al/^{27}Al ratios in a large number of individual grains (Figures 3–5) have led to the classification into different populations (Hoppe and Ott, 1997): mainstream grains (~93% of the total), and the minor subtypes A, B, X, Y, Z, and nova grains.

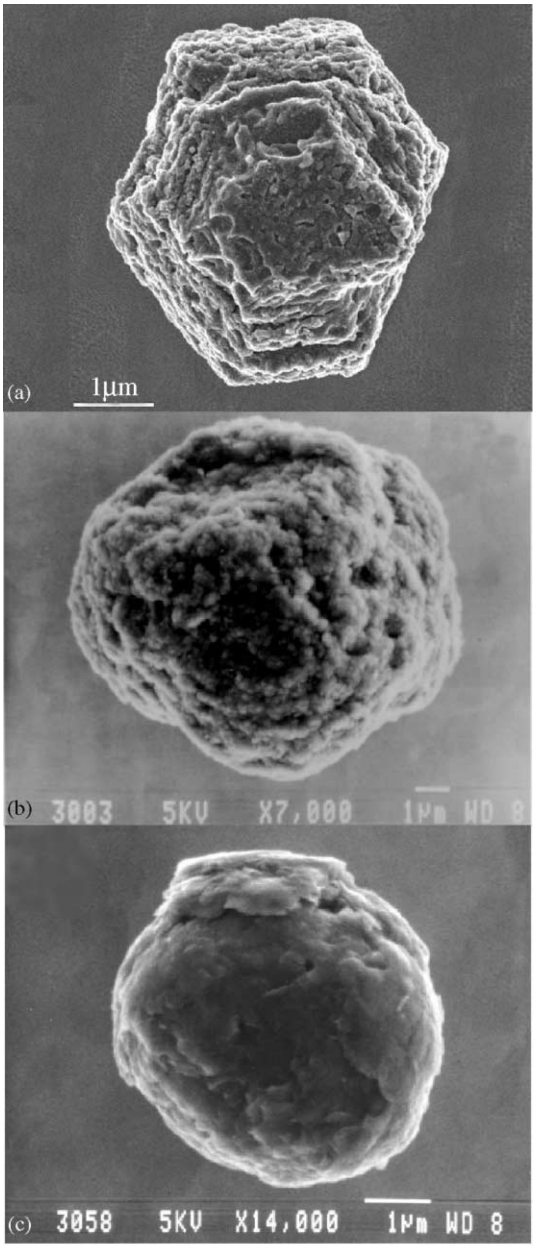

Figure 2 Secondary electron micrographs of (a) presolar SiC, (b) presolar graphite (cauliflower type), and (c) presolar graphite (onion type). Photographs courtesy of Sachiko Amari and Scott Messenger.

Most of presolar SiC is believed to have originated from carbon stars, late-type stars of low mass (1–3$M_\odot$) in the thermally pulsing (TP) asymptotic giant branch (AGB) phase of evolution (Iben and Renzini, 1983). Dust from such stars has been proposed already one decade prior to identification of SiC to be a minor constituent of primitive meteorites (Clayton and Ward, 1978; Srinivasan and Anders, 1978; Clayton, 1983a). Several pieces of evidence point to such an origin. Mainstream grains have ^{12}C/^{13}C ratios similar to those found in carbon stars (Figure 6), which are considered to be the most prolific injectors of carbonaceous dust grains into the ISM (Tielens, 1990). Many carbon stars show the 11.3 μm

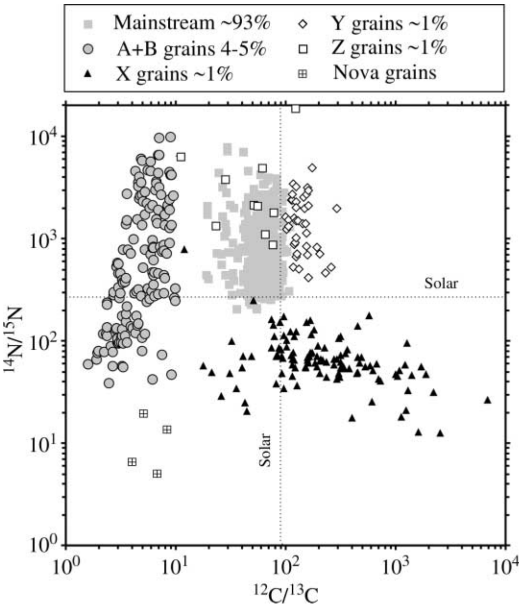

Figure 3 Nitrogen and carbon isotopic ratios of individual presolar SiC grains. Because rare grain types were located by automatic ion imaging, the number of grains of different types do not correspond to their abundances in the meteorites; these abundances are given in the legend (sources Alexander, 1993; Hoppe *et al.*, 1994, 1996a; Nittler *et al.*, 1995; Huss *et al.*, 1997; Amari *et al.*, 2001a,b,c).

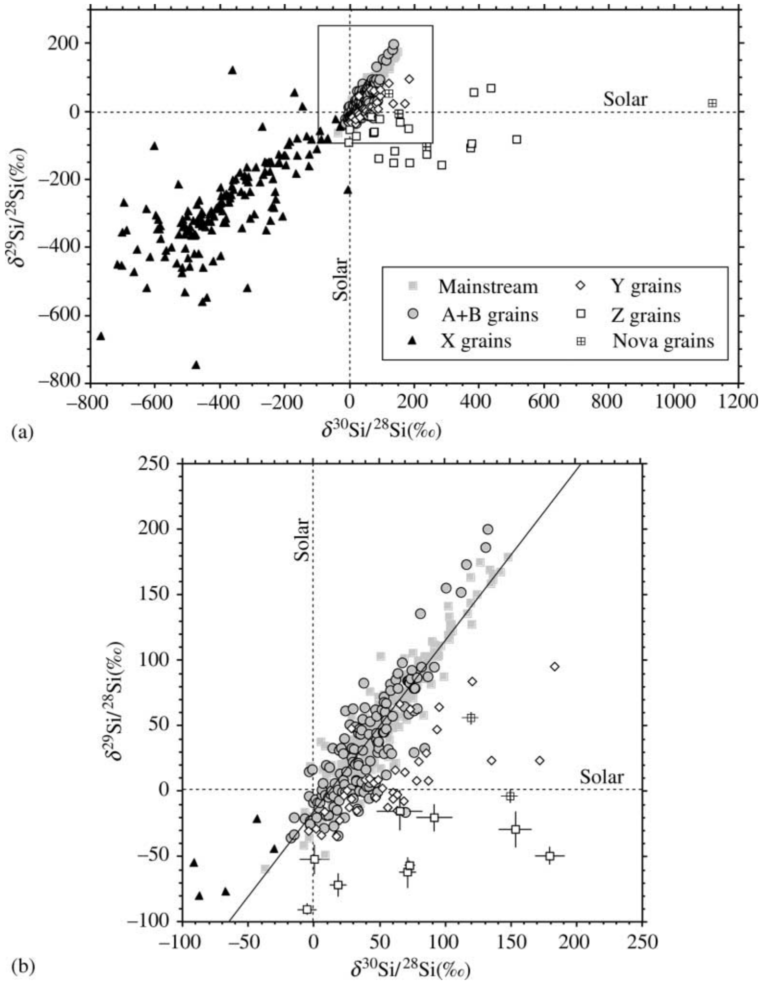

Figure 4 Si isotopic ratios of different types of presolar SiC grains plotted as δ-values, deviations in per mil (‰) from the solar ratios: $\delta^{i}\mathrm{Si}/^{28}\mathrm{Si} = [(^{i}\mathrm{Si}/^{28}\mathrm{Si})_{\mathrm{meas}}/(^{i}\mathrm{Si}/^{28}\mathrm{Si})_{\odot -1}] \times 1{,}000$. Mainstream grains plot along a line of slope 1.4 (solid line). Symbols are the same as those in Figure 3. Sources same as in Figure 3.

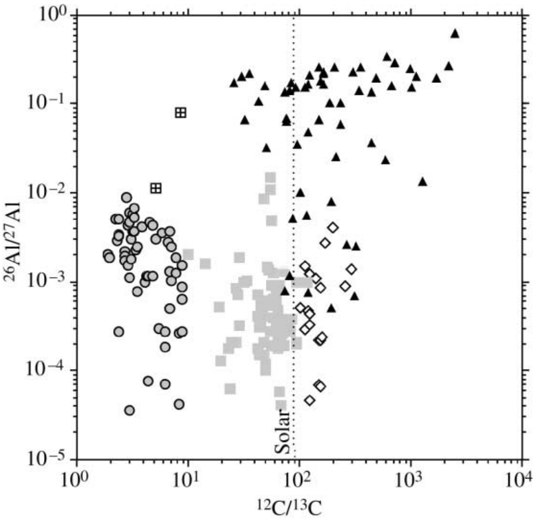

Figure 5 Aluminum and carbon isotopic ratios of individual presolar SiC grains. Symbols and sources for the data are the same as those for Figure 3.

emission feature typical of SiC (Treffers and Cohen, 1974; Speck *et al.*, 1997). Finally, AGB stars are believed to be the main source of the *s*-process (slow neutron capture nucleosynthesis) elements (e.g., Busso *et al.*, 2001), and the *s*-process isotopic patterns of the heavy elements exhibited by mainstream SiC provide the most convincing argument for their origin in carbon stars (see below).

1.02.6.1 Mainstream Grains

Mainstream grains have ^{12}C/^{13}C ratios between 10 and 100 (Figure 2). They have carbon and nitrogen isotopic compositions (Zinner *et al.*, 1989; Stone *et al.*, 1991; Virag *et al.*, 1992; Alexander, 1993; Hoppe *et al.*, 1994, 1996a; Nittler *et al.*, 1995; Huss *et al.*, 1997; Amari *et al.*, 2002; Nittler and Alexander, 2003) that are roughly in agreement with an AGB origin.

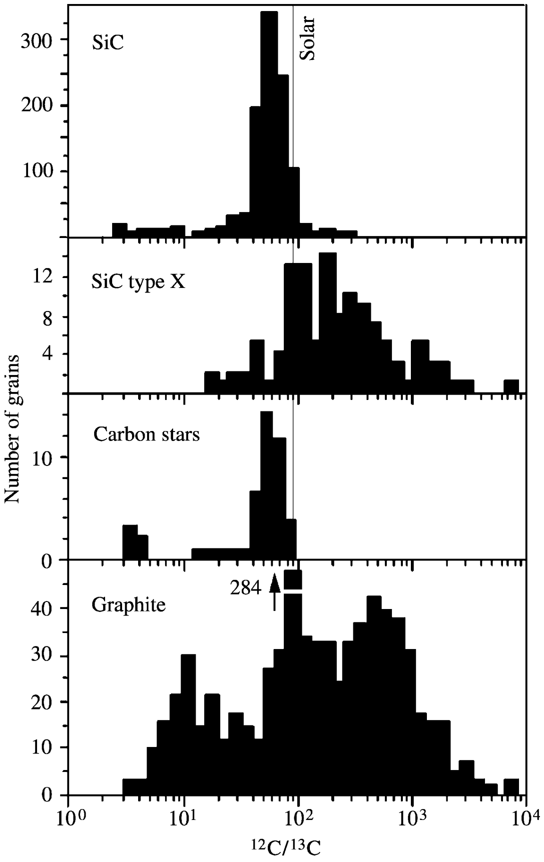

Figure 6 The distributions of carbon isotopic ratios measured in presolar SiC (Hoppe *et al.*, 1994; Nittler *et al.*, 1995) and graphite grains (Hoppe *et al.*, 1995) from the Murchison meteorite are compared to astronomical measurements of the atmospheres of carbon stars (Lambert *et al.*, 1986).

Carbon-13 and ^{15}N excesses relative to solar are the signature of hydrogen burning via the CNO cycle that occurred during the main sequence phase of the stars. This material is brought to the star's surface by the first (and second) dredge-up. The carbon isotopic ratios are also affected by shell helium burning and the third dredge-up during the TP-AGB phase (Busso *et al.*, 1999). This process adds ^{12}C to the envelope, increases the ^{12}C/^{13}C ratio from the low values resulting from the first dredge-up, and, by making $C > O$, causes the star to become a carbon star.

Envelope ^{12}C/^{13}C ratios predicted by canonical stellar evolution models range from ~20 after first dredge-up in the RG phase to ~300 in the late TP-AGB phases (El Eid, 1994; Gallino *et al.*, 1994; Amari *et al.*, 2001b). Predicted ^{14}N/^{15}N ratios are 600–1,600 (Becker and Iben, 1979; El Eid, 1994), falling short of the range observed in the grains. However, the assumption of deep mixing ("cool bottom processing") of envelope material to deep hot regions in $M < 2.5 M_{\odot}$ stars during their RG and AGB phases (Charbonnel, 1995; Wasserburg *et al.*, 1995; Langer *et al.*, 1999; Nollett *et al.*, 2003) results in partial hydrogen burning, with higher ^{14}N/^{15}N and lower ^{12}C/^{13}C ratios in the envelope than in canonical models (see also Huss *et al.*, 1997).

Two other isotopes that are a signature for AGB stars are ^{26}Al and ^{22}Ne. Figure 5 shows inferred ^{26}Al/^{27}Al ratios in different types of SiC grains. The existence of the short-lived radioisotope ^{26}Al ($T_{1/2} = 7.3 \times 10^5$ yr) is inferred from large ^{26}Mg excesses. Aluminium-26 is produced in the hydrogen shell by proton capture on ^{25}Mg and mixed to the surface by the third dredge-up (Forestini *et al.*, 1991). It can also be produced during "hot bottom burning" (Lattanzio *et al.*, 1997), but this process is believed to prevent carbon-star formation (Frost and Lattanzio, 1996). Neon-22, the main component in Ne-E, is produced in the helium shell by $^{14}\mathrm{N} + 2\alpha$. The neon isotopic ratios measured in SiC bulk samples (Lewis *et al.*, 1990, 1994) are very close to those expected for helium shell material (Gallino *et al.*, 1990). In contrast to krypton and xenon and heavy refractory elements, neon as well as helium and argon show very little dilution of helium shell material with envelope material, indicating a special implantation mechanism by an ionized wind. Another piece of evidence that the Ne-E(H) component originated from the helium shell of AGB stars and not from the decay of ^{22}Na (Clayton, 1975) is the fact that in individual grains, of which only ~5% carry ^{22}Ne, it is always accompanied by ^{4}He (Nichols *et al.*, 2003). Excesses in ^{21}Ne in SiC relative to the predicted helium-shell composition have been interpreted as being due to spallation by galactic cosmic rays (Tang and Anders, 1988a; Lewis *et al.*, 1990, 1994), which allows the determination of grain lifetimes in the IS medium. Inferred exposure ages depend on grain size and range from 10 Myr to 130 Myr (Lewis *et al.*, 1994). However, this interpretation has recently been challenged (Ott and Begemann, 2000) and the question of IS ages of SiC is not settled.

The silicon isotopic compositions of most mainstream grains are characterized by enrichments in the heavy silicon isotopes of up to 200‰ relative to their solar abundances (Figure 4). In a silicon three-isotope plot the data fall along a line with slope 1.4, which is shifted slightly to the right of the solar system composition. In contrast to the light elements carbon, nitrogen, neon, and aluminum and the heavy elements (see below), the silicon isotopic ratios of mainstream grains cannot be explained by nuclear processes taking place within their parent stars. In AGB stars the silicon isotopes are affected by neutron capture in the helium shell leading to excesses in ^{29}Si and ^{30}Si along a slope 0.2–0.5

line in a δ-value silicon three-isotope plot (Gallino *et al.*, 1990, 1994; Brown and Clayton, 1992; Lugaro *et al.*, 1999; Amari *et al.*, 2001b). Predicted excesses are only on the order of 20‰ in low-mass AGB stars of close-to-solar metallicity (metallicity is the abundance of all elements heavier than helium). This led to the proposal that many stars with varying initial silicon isotopic compositions contributed SiC grains to the solar system (Clayton *et al.*, 1991; Alexander, 1993) and that neutron-capture nucleosynthesis in these stars only plays a secondary role in modifying these compositions. Several explanations have been given for the initial silicon ratios in the parent stars, which in their late stages of evolution became the carbon stars that produced the SiC. One is the evolution of the silicon isotopic ratios through galactic history as different generations of supernovae produced silicon with increasing ratios of the secondary isotopes ^{29}Si and ^{30}Si to the primary ^{28}Si (Gallino *et al.*, 1994; Timmes and Clayton, 1996; Clayton and Timmes, 1997a,b). Clayton (1997) addressed the problem that most SiC grains have higher than solar ^{29}Si/^{28}Si and ^{30}Si/^{28}Si ratios by considering the possibility that the mainstream grains originated from stars that were born in central, more metal-rich regions of the galaxy and moved to the molecular cloud from which our sun formed. Alexander and Nittler (1999), alternatively, suggested that the Sun has an atypical silicon isotopic composition. Lugaro *et al.* (1999) explained the spread in the isotopic compositions of the parent stars by local heterogeneities in the galaxy caused by the stochastic nature of the admixture of the ejecta from supernovae of varying type and mass.

Titanium isotopic ratios in single grains (Ireland *et al.*, 1991; Hoppe *et al.*, 1994; Alexander and Nittler, 1999) and in bulk samples (Amari *et al.*, 2000) show excesses in all isotopes relative to ^{48}Ti, a result expected of neutron capture in AGB stars. However, as for silicon, theoretical models (Lugaro *et al.*, 1999) cannot explain the range of ratios observed in single grains. Furthermore, titanium ratios are correlated with those of silicon, also indicating that the titanium isotopic compositions are dominated by galactic evolution effects (Alexander and Nittler, 1999). Excesses of ^{42}Ca and ^{43}Ca relative to ^{40}Ca observed in bulk samples (Amari *et al.*, 2000) agree with predictions for neutron capture. Large ^{44}Ca excesses are apparently due to the presence of type X grains (see below). Iron isotopic ratios have been measured by RIMS in single grains (Davis *et al.*, 2002; Tripa *et al.*, 2002). Depletions in ^{54}Fe are much larger than predicted by neutron capture in AGB stars and ^{57}Fe does not show the predicted excesses. While it is quite possible that, as for silicon and titanium, galactic evolution effects dominate and while admixture of SN ejecta results in the observed ^{54}Fe depletions, the SN mixing model also predicts substantial ^{57}Fe excesses, which are not observed (see also Clayton *et al.*, 2002).

All heavy elements measured so far show the signature of the s-process (Figure 7, see also Figure 10). They include the noble gases krypton and xenon (Lewis *et al.*, 1990, 1994) but also the heavy elements strontium (Podosek *et al.*, 2003), barium (Ott and Begemann, 1990; Zinner *et al.*, 1991; Prombo *et al.*, 1993), neodymium and samarium (Zinner *et al.*, 1991; Richter *et al.*, 1993), and dysprosium (Richter *et al.*, 1994). Although most measurements were made on bulk samples, it is clear that mainstream grains dominate. Single-grain measurements of strontium (Nicolussi *et al.*, 1998b), zirconium (Nicolussi *et al.*, 1997), molybdenum (Nicolussi *et al.*, 1998a), and barium (Savina *et al.*, 2003a) have been made with RIMS. Large enrichments of certain heavy elements such as yttrium, zirconium, barium, and cerium in single mainstream grains also indicate large overabundances of s-process elements in the parent stars (Amari *et al.*, 1995a). For all the isotopic compositions of the elements listed above except for dysprosium there is good agreement with theoretical models of the s-process in low-mass AGB stars (Gallino *et al.*, 1993, 1997; Lugaro *et al.*, 2003). Discrepancies with earlier model calculations were caused by incorrect nuclear cross-sections and could be resolved by improved experimental determinations (e.g., Guber *et al.*, 1997; Wisshak *et al.*, 1997; Koehler *et al.*, 1998).

The s-process isotopic patterns observed in grains allow the determination of different parameters affecting the s-process such as neutron exposure, temperature, and neutron density (Hoppe and Ott, 1997). Since these parameters depend in turn on stellar mass and metallicity as well as on the neutron source operating in AGB stars, they allow information to be obtained about the parent stars of the grains. For example, the barium isotopic ratios indicate a neutron exposure that is only half of that inferred for the solar system (Ott and Begemann, 1990; Gallino *et al.*, 1993). Another example is provided by the abundance of ^{96}Zr in single grains, which is sensitive to neutron density because of the relatively short half-life of ^{95}Zr (~64 d). While the ^{13}C(α,n) source with its low neutron density destroys ^{96}Zr, activation of the ^{22}Ne(α,n) source during later thermal pulses in AGB stars restores some of this isotope, whose abundance thus varies with pulse number. Some grains have essentially no ^{96}Zr, indicating that the ^{22}Ne(α,n) source was weak in their parent stars, pointing to low-mass AGB stars as the source of mainstream grains (Lugaro *et al.*, 2003).

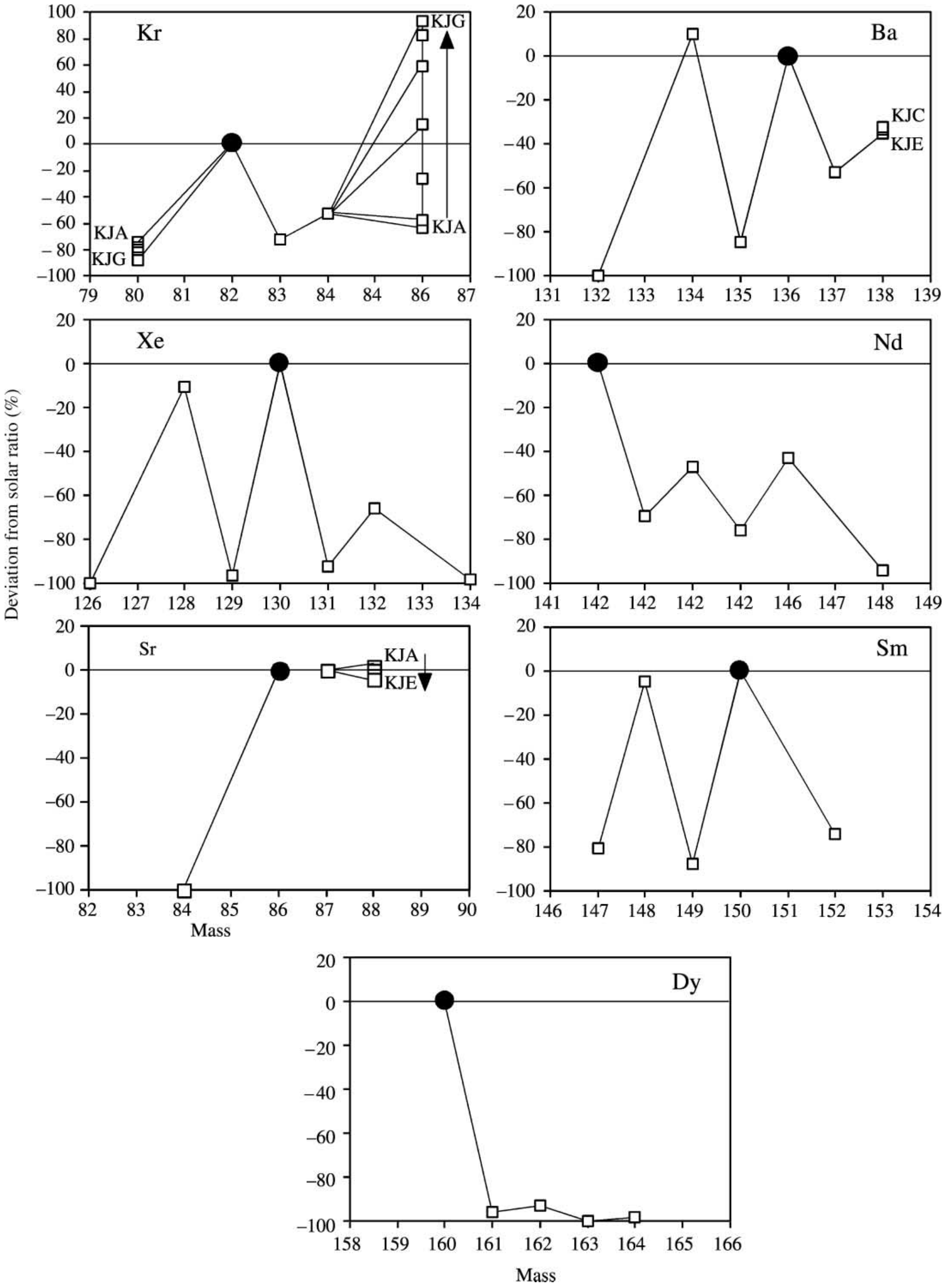

Figure 7 Isotopic patterns measured in bulk samples of SiC extracted from the Murchison meteorite. Isotopic ratios are relative to the reference isotope plotted as a solid circle and are normalized to the solar isotopic ratios. Data are from Lewis *et al*. (1994) (Kr and Xe), Podosek *et al*. (2003) (Sr), Prombo *et al*. (1993) (Ba), Richter *et al*. (1993) (Nd and Sm), and Richter *et al*. (1994) (Dy).

1.02.6.2 Type Y and Z Grains

Type Y grains have ^{12}C/^{13}C $>$ 100 and silicon isotopic compositions that lie to the right of the mainstream correlation line (Figures 3 and 4(b)) (Hoppe *et al*., 1994; Amari *et al*., 2001b). Type Z grains have even larger ^{30}Si excesses relative to ^{29}Si and, on average, lower δ^{29}Si values than Y grains. However, they are distinguished from Y grains by having ^{12}C/^{13}C $<$ 100

(Alexander, 1993; Hoppe *et al.*, 1997). Comparison of the carbon, silicon, and titanium isotopic ratios of Y grains with models of nucleosynthesis indicates an origin in low-to-intermediate-mass AGB stars with approximately half the solar metallicity (Amari *et al.*, 2001b). Such stars dredge up more ^{12}C, and silicon and titanium that experienced neutron capture, from the helium shell (see also Lugaro *et al.*, 1999). According to their silicon isotopic ratios, Z grains came from low-mass stars of even lower (~one-third solar) metallicity (Hoppe *et al.*, 1997). In order to achieve the relatively low ^{12}C/^{13}C ratios of these grains, the parent stars must have experienced cool bottom processing (Wasserburg *et al.*, 1995; Nollett *et al.*, 2003) during their RG and AGB phase. From the theoretically inferred metallicities and average silicon isotopic ratios of mainstream Y and Z grains, Zinner *et al.* (2001) derived the galactic evolution of the silicon isotopic ratios as a function of metallicity. This evolution differs from the results of galactic evolution models based on the yields of supernovae (Timmes and Clayton, 1996) and has important implications concerning the relative contributions from type II and type Ia supernovae during the history of our galaxy.

1.02.6.3 Type A + B Grains

Grains of type A + B have ^{12}C/^{13}C < 10, but their silicon isotopic ratios plot along the mainstream line (Figures 3 and 4). In contrast to mainstream grains, many A + B grains have lower than solar ^{14}N/^{15}N ratios (Hoppe *et al.*, 1995, 1996a; Huss *et al.*, 1997; Amari *et al.*, 2001c). While the isotopic ratios of mainstream, Y and Z grains find an explanation in nucleosynthetic models of AGB stars, a satisfactory explanation of the data in terms of stellar nucleosynthesis is more elusive for the A + B grains. The low ^{12}C/^{13}C ratios of these grains combined with the requirement for a carbon-rich environment during their formation indicate helium burning followed by limited hydrogen burning in their stellar sources. However, the astrophysical sites for this process are not well known. There might be two different kinds of A + B grains with corresponding different stellar sources (Amari *et al.*, 2001c). Grains with no s-process enhancements (Amari *et al.*, 1995a; Pellin *et al.*, 2000b; Savina *et al.*, 2003c) probably come from J-type carbon stars that also have low ^{12}C/^{13}C ratios (Lambert *et al.*, 1986). Unfortunately, J stars are not well understood and there are no astronomical observations of nitrogen isotopic ratios in such stars. Furthermore, the low ^{14}N/^{15}N ratios observed in some of the grains as well as the carbon-rich nature of their parent stars appear to be incompatible with the consequences of hydrogen burning in the CNO cycle, which seems to be responsible for the low ^{12}C/^{13}C ratios of J stars and the grains. A + B grains with s-process enhancements might come from post-AGB stars that undergo a very late thermal pulse. An example of such a star is Sakurai's object (e.g., Asplund *et al.*, 1999; Herwig, 2001). However, grains with low ^{14}N/^{15}N ratios pose a problem. Huss *et al.* (1997) proposed that the currently used ^{18}O(p,α)^{15}N reaction rate is too low by a factor of 1,000. This would result in low ^{12}C/^{13}C and ^{14}N/^{15}N ratios if an appropriate level of cool bottom processing is considered.

1.02.6.4 Type X Grains

Although SiC grains of type X account for only 1% of presolar SiC, a fairly large number can be located by ion imaging (Nittler *et al.*, 1997; Hoppe *et al.*, 1996b, 2000; Lin *et al.*, 2002; Besmehn and Hoppe, 2003). X grains are characterized by mostly ^{12}C and ^{15}N excesses relative to solar (Figures 3 and 6), excesses in ^{28}Si (Figure 4) and very large ^{26}Al/^{27}Al ratios, ranging up to 0.6 (Figure 5). About 10–20% of the grains show large ^{44}Ca excesses, which must come from the decay of short-lived ^{44}Ti ($T_{1/2} = 60$ yr) (Amari *et al.*, 1992; Hoppe *et al.*, 1996b, 2000; Nittler *et al.*, 1996; Besmehn and Hoppe, 2003). Inferred ^{44}Ti/^{48}Ti ratios range up to 0.6 (Figure 8). In contrast to presolar graphite, which contains subgrains of TiC, titanium in SiC seems to occur in solid solution and radiogenic ^{44}Ca is uniformly distributed in most of the grains. Only in one X grains a pronounced heterogeneity points to a titanium-rich subgrain (Besmehn and Hoppe, 2003). Because ^{44}Ti can only be produced in SN explosions (Timmes *et al.*, 1996), grains with evidence for ^{44}Ti, and by implications all X grains, must have an SN origin. In type II supernovae ^{44}Ti is produced in the nickel- and silicon-rich inner zones (see Figure 9) (Woosley and Weaver, 1995; Timmes *et al.*, 1996). Silicon in the Si/S zone consists of almost pure ^{28}Si. Also the other isotopic signatures of X grains are compatible with an origin in type II supernovae: high ^{12}C/^{13}C and low ^{14}N/^{15}N ratios are the signature of helium burning (Figure 9) and high ^{26}Al/^{27}Al ratios can be reached in the He/N zone by hydrogen burning.

However, these isotopic signatures occur in massive stars in very different stellar zones, which experienced different stages of nuclear burning before the SN explosion (Figure 9) (e.g., Woosley and Weaver, 1995; Rauscher *et al.*, 2002). The isotopic signatures of the X grains suggest deep and inhomogeneous mixing of matter from these different zones in the SN ejecta. While the titanium and silicon isotopic signature of the X

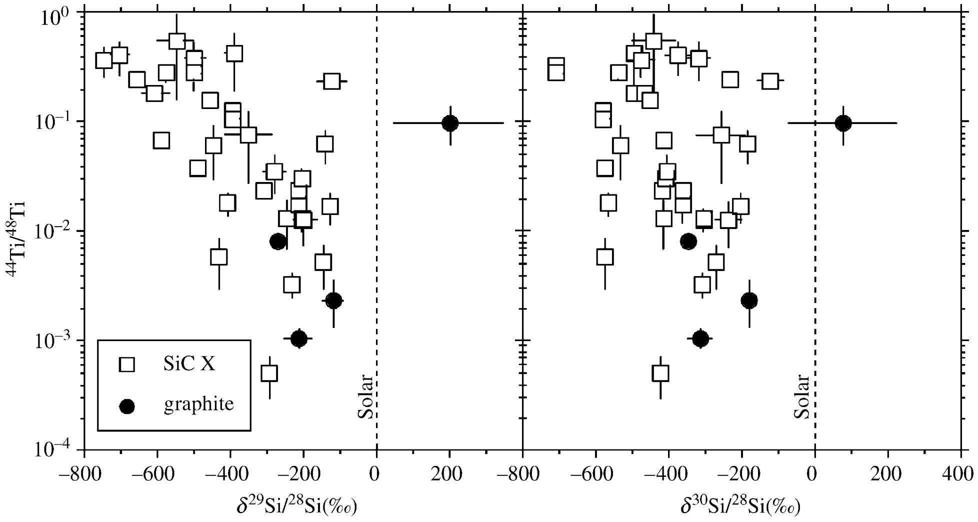

Figure 8 $^{44}Ti/^{48}Ti$ ratios inferred from ^{44}Ca excesses in SiC grains of type X and graphite grains are plotted against Si isotopic ratios. Except for one graphite, all grains with evidence for ^{44}Ti have ^{28}Si excesses (sources Amari *et al.*, 1992, unpublished; Hoppe *et al.*, 1994, 1996b, 2000; Nittler *et al.*, 1996; Besmehn and Hoppe, 2003).

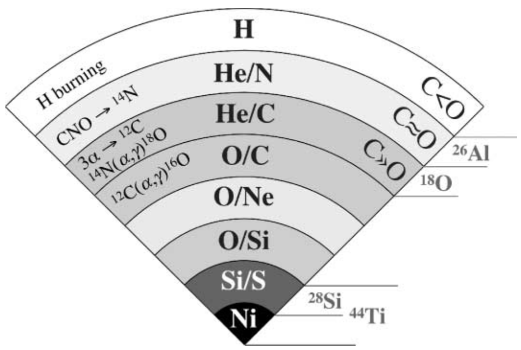

Figure 9 Schematic structure of a massive star before its explosion as a type II supernova (source Woosley and Weaver, 1995). Such a star consists of different layers, which are labeled according to their most abundant elements that experienced different stages of nucleosynthesis. Indicated are dominant nuclear reactions in some layers and the layers in which isotopes abundant in grains of an inferred SN origin are produced.

grains requires contributions from the Ni, O/Si and Si/S zones, which experienced silicon-, neon-, and oxygen-burning, significant contributions must also come from the He/N and He/C zones that experienced hydrogen and incomplete helium burning in order to achieve C > O, the condition for SiC condensation (Larimer and Bartholomay, 1979; Lodders and Fegley, 1997). Furthermore, addition of material from the intermediate oxygen-rich layers must be severely limited. Astronomical observations indicate extensive mixing of SN ejecta (e.g., Ebisuzaki and Shibazaki, 1988; Hughes *et al.*, 2000) and hydrodynamic models of SN explosions predict mixing in the ejecta initiated by the formation of Rayleigh–Taylor instabilities (e.g., Herant *et al.*, 1994). However, it still has to be seen whether mixing can occur on a microscopic scale and whether these instabilities allow mixing of matter from nonneighboring zones while excluding large contributions from the intermediate oxygen-rich zones. Clayton *et al.* (1999) and Deneault *et al.* (2003) suggested condensation of carbonaceous phases in type II SN ejecta even while C < O because of the destruction of CO in the high-radiation environment of the ejecta. While this might work for graphite, it is doubtful whether SiC can condense from a gas with C < O (Ebel and Grossman, 2001). Even for graphite, the presence of subgrains of elemental iron inside of graphite grains whose isotopic signatures indicate an SN origin argues against formation in an oxygen-rich environment (Croat *et al.*, 2003).

Although multizone mixing models can qualitatively reproduce the isotopic signatures of X grains, several ratios, in particular the large ^{15}N excesses and excesses of ^{29}Si over ^{30}Si found in most grains, cannot be explained quantitatively and indicate deficiencies in the existing models. The latter is a long-standing problem: SN models cannot account for the solar $^{29}Si/^{30}Si$ ratio (Timmes and Clayton, 1996). Studies of SiC X grains isolated from the Qingzhen enstatite chondrite (Lin *et al.*, 2002) suggest that there are two population of X grains with different trends in the silicon isotopic ratios, the minor population having lower-than-solar $^{29}Si/^{30}Si$ ratios. Recently, Clayton *et al.* (2002) and Deneault *et al.* (2003) have tried to account for isotopic signatures from different SN zones by considering implantation into newly condensed grains as they pass through different regions of the ejecta, specifically through zones with reverse shocks.

Some SiC X grains also show large excesses in ^{49}Ti (Amari *et al.*, 1992; Nittler *et al.*, 1996; Hoppe and Besmehn, 2002). The correlation of these excesses with the V/Ti ratio (Hoppe and Besmehn, 2002) indicates that they come from the decay of short-lived ^{49}V ($T_{1/2} = 330$ d) and that the grains must have formed within a few months of the explosion. ^{49}V is produced in the Si/S zone, which contains almost pure ^{28}Si. RIMS isotopic measurements of iron, strontium, zirconium, molybdenum, and barium have been made on X grains (Pellin *et al.*, 1999, 2000a; Davis *et al.*, 2002). The most complete and interesting are the molybdenum measurements, which reveal large excesses in ^{95}Mo and ^{97}Mo. Figure 10 shows the molybdenum isotopic patterns of a mainstream and an X grain. The mainstream grain has a typical s-process pattern, in agreement with bulk measurements of other heavy elements such as xenon, barium, and neodymium (Figure 7). The molybdenum pattern of the X grain is completely different and indicates neutron capture at much higher neutron densities. While it does not agree with the pattern expected for the r-process, it is successfully explained by a neutron-burst model (Meyer *et al.*, 2000). In the type II SN models by Rauscher *et al.* (2002) an intense neutron burst is predicted to occur in the oxygen layer just below the He/C zone, accounting for the molybdenum isotopic patterns observed in X grains.

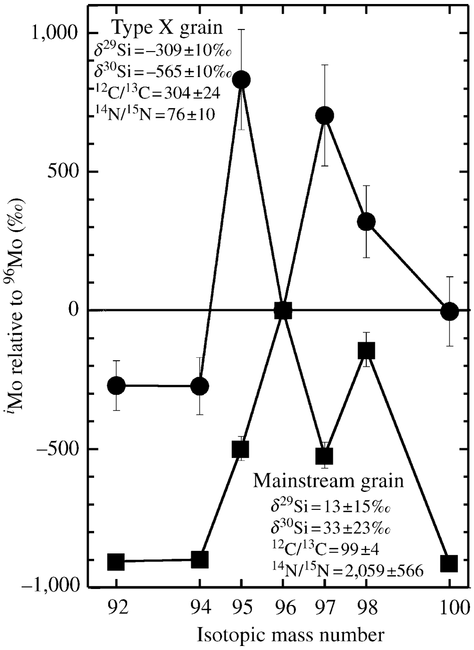

Figure 10 Molybdenum isotopic patterns measured by RIMS in a type X and a mainstream SiC grain (source Pellin *et al.*, 1999).

Type Ia supernovae offer an alternative explanation for the isotopic signature of X grains. In the model by Clayton *et al.* (1997) nucleosynthesis takes place by explosive helium burning of a helium cap on top of a white dwarf. This process produces most of the isotopic signatures of the SN grains. The isotopes ^{12}C, ^{15}N, ^{26}Al, ^{28}Si, and ^{44}Ti are all made by helium burning during the explosion, which makes the transport of ^{28}Si and ^{44}Ti through the massive oxygen-rich zone into the overlying carbon-rich zones of a type II SN unnecessary. Mixing is limited to material from helium burning and to matter that experienced CNO processing. The best match with the X grain data, however, is achieved for mixing scenarios that yield $O > C$ (Amari *et al.*, 1998). Other problems include the questions whether high enough gas densities can be achieved in the ejecta for the condensation of micrometer-sized grains and whether type Ia supernovae can generate a neutron burst necessary for the molybdenum isotopic pattern. More work is needed to decide whether a type Ia SN origin for X grains is a realistic alternative.

1.02.6.5 Nova Grains

A few grains have isotopic ratios that are best explained by a nova origin (Amari *et al.*, 2001a). These grains have low ^{12}C/^{13}C and ^{14}N/^{15}N ratios (Figure 3), large ^{30}Si excesses (Figure 4), and high ^{26}Al/^{27}Al ratios (Figure 5). All these features are predicted to be produced by explosive hydrogen burning taking place in classical novae (e.g., Kovetz and Prialnik, 1997; Starrfield *et al.*, 1998; José *et al.*, 1999) but the predicted anomalies are much larger than those found in the grains, and the nova ejecta have to be mixed with material of close-to-solar isotopic compositions. A comparison of the data with the models implicates ONe novae with a white dwarf mass of at least 1.25 $M_\odot$ as the most likely sources (Amari *et al.*, 2001a).

1.02.6.6 Grain Size Effect

Grain size distributions of SiC have been determined for several meteorites and while grain sizes vary from 0.1 μm to 20 μm, the distributions are different for different meteorites. Murchison appears to have, on average, the largest grains (Amari *et al.*, 1994), while SiC from Indarch (Russell *et al.*, 1997) and Orgueil (Huss *et al.*, 1997) is much finer grained. Various isotopic and other properties vary with grain size. Both the ^{22}Ne-E(H)/^{130}Xe-S and the ^{86}Kr/^{82}Kr ratios increase with grain size (Lewis *et al.*, 1994) and the first ratio has been used as a measure for the average grain size in meteorites for which no detailed size distributions have been determined (Russell *et al.*, 1997). The ^{86}Kr/^{82}Kr ratio is a function of neutron exposure and the data indicate that exposure decreases with increasing grains size. The ^{88}Sr/^{86}Sr and ^{138}Ba/^{136}Ba ratios also depend on grain size but the dependence of neutron exposure on grain size inferred from these isotopic ratios is just the opposite of that inferred from the ^{86}Kr/^{82}Kr ratio. This puzzle has not been resolved. A possible explanation is a different trapping mechanism for noble gases and refractory elements, respectively (Zinner *et al.*, 1991), or different populations of carrier grains if, as for neon (Nichols *et al.*, 2003), only a small fraction of the grains carry krypton. Excesses in ^{21}Ne relative to the predicted helium-shell composition, interpreted as being due to spallation by galactic cosmic rays, increase with grain size (Tang and Anders, 1988a; Lewis *et al.*, 1990, 1994). However, the correlation of the ^{21}Ne/^{22}Ne ratio with the s-process ^{86}Kr/^{82}Kr ratio (Hoppe and Ott, 1997) and the recent determination of spallation recoil ranges (Ott and Begemann, 2000) cast doubt on a chronological interpretation. Other grain size effects are, on average, larger ^{14}N/^{15}N ratios for smaller grains (Hoppe *et al.*, 1996a) and an increasing abundance of Z grains among smaller SiC grains (Hoppe *et al.*, 1996a, 1997). There are also differences in the distribution of different grain types in SiC from different meteorites: whereas the abundance of X grains in SiC from Murchison and other carbonaceous chondrites is ~1%, it is only ~0.1% in SiC from the enstatite chondrites Indarch and Qingzhen (Besmehn and Hoppe, 2001; Lin *et al.*, 2002).

1.02.7 SILICON NITRIDE

Presolar silicon nitride (Si_3N_4) grains are extremely rare (in Murchison SiC-rich separates ~5% of SiC of type X), but automatic ion imaging has been successfully used to detect those with large ^{28}Si excesses (Nittler *et al.*, 1995; Besmehn and Hoppe, 2001; Lin *et al.*, 2002; Nittler and Alexander, 2003). The carbon, nitrogen, aluminum, and silicon isotopic signatures of these grains are the same as those of SiC grains of type X, i.e., large ^{15}N and ^{28}Si excesses and high ^{26}Al/^{27}Al ratios (Figure 12). Although so far no resolvable ^{44}Ca excesses have been detected (Besmehn and Hoppe, 2001), the similarity with X grains implies an SN origin for these grains. While Si_3N_4 grains in SiC-rich residues from Murchison are extremely rare and, if present, are of type X, enstatite chondrites contain much higher abundances of Si_3N_4 (Alexander *et al.*, 1994; Besmehn and Hoppe, 2001; Amari *et al.*, 2002). Most of them have normal isotopic compositions. Recent measurements of small (0.25–0.65 μm) grains from Indarch revealed several Si_3N_4 grains with carbon and nitrogen isotopic ratios similar to those of mainstream SiC grains, but contamination from attached SiC grains cannot be excluded (Amari *et al.*, 2002).

1.02.8 GRAPHITE

Graphite, the third type of carbonaceous presolar grains, was isolated because it is the carrier of Ne-E(L) (Amari *et al.*, 1990; Amari *et al.*, 1995b). Subsequent isotopic measurements of individual grains revealed anomalies in many different elements.

1.02.8.1 Physical Properties

Only grains ≥1 μm in diameter carry Ne-E(L) and only round grains, which range up to 20 μm in size, appear to be of presolar origin (Amari *et al.*, 1990; Zinner *et al.*, 1995). Presolar graphite has a range in density (1.6–2.2 g cm^{-3}) and four different density fractions have been isolated (Amari *et al.*, 1994). Average grain sizes decrease with increasing density, and density fractions differ in the distribution of their carbon and noble gas isotopic compositions (Amari *et al.*, 1995b; Hoppe *et al.*, 1995). SEM studies revealed two basic morphologies (Hoppe *et al.*, 1995): dense aggregates of small scales ("cauliflowers," Figure 2(b)) and grains with smooth or shell-like platy surfaces ("onions," Figure 2(c)). TEM analysis of microtomed sections of graphite spherules (Bernatowicz *et al.*, 1991, 1996) found the surface morphology reflected in the internal structure of the grains. Cauliflowers consist of concentrically packed scales of poorly crystallized carbon, whereas onions either consist of well-crystallized graphite throughout or of a core of tightly packed graphene sheets of only several atomic layers surrounded by a mantle of well-crystallized graphite. Most graphite spherules contain small (20–500 nm) internal grains of mostly titanium carbide (TiC) (Bernatowicz *et al.*, 1991); however, also zirconium- and molybdenum-rich carbides have been found (Bernatowicz *et al.*, 1996). Recent studies of graphite spherules whose oxygen and silicon isotopic compositions indicated an SN origin (see below) did not detect Zr–Mo-rich carbides but revealed internal kamacite, cohenite, and iron grains in addition to TiC (Bernatowicz *et al.*, 1999; Croat *et al.*, 2003). Both cauliflowers and onions contain internal grains, which must have condensed before the graphite, and were apparently captured and included by the growing

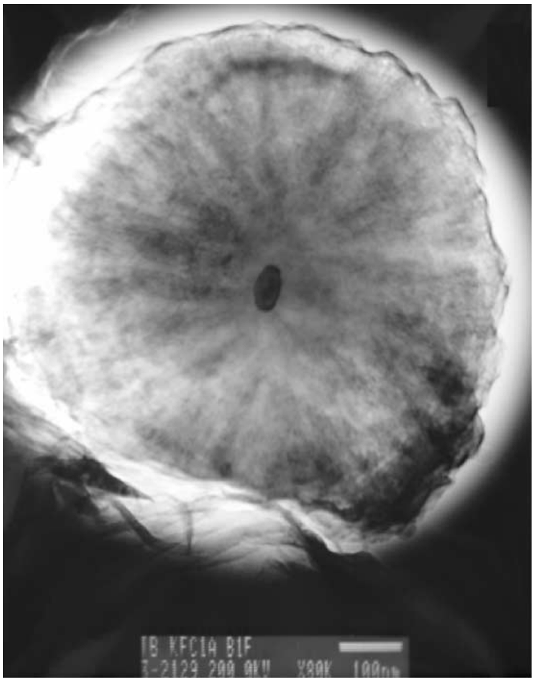

Figure 11 Transmission electron micrograph of a slice through a presolar graphite grain (onion). The grain in the center is TiC and apparently acted as condensation nucleus for the growth of the graphite spherule. Photo courtesy of Thomas Bernatowicz.

spherules. Some onions show TiC grains at their center that apparently acted as condensation nuclei for the graphite (Figure 11). Sizes of internal grains and graphite spherules and their relationship and chemical compositions provide information about physical properties such as pressure, temperature, and C/O ratio in the gas from which the grains condensed (Bernatowicz *et al.*, 1996; Croat *et al.*, 2003).

1.02.8.2 Isotopic Compositions

Noble gas measurements were made on bulk samples of four density fractions (Amari *et al.*, 1995b). In contrast to SiC, a substantial fraction of Ne-E in graphite seems to come from the decay of short-lived ($T_{1/2} = 2.6$ yr) ^{22}Na (Clayton, 1975). This is supported by the low ^{4}He/^{22}Ne ratios measured in individual grains (Nichols *et al.*, 2003). Krypton in graphite has two s-process components with apparent different neutron exposures, residing in different density fractions (Amari *et al.*, 1995b).

Ion microprobe analyses of single grains revealed the same range of ^{12}C/^{13}C ratios as in SiC grains but the distribution is quite different (Figure 6). Most anomalous grains have ^{12}C excesses, similar to SiC X grains. A substantial fraction has low ^{12}C/^{13}C ratios like SiC A + B grains. Most graphite grains have close-to-solar nitrogen isotopic ratios (Hoppe *et al.*, 1995; Zinner *et al.*, 1995). In view of the enormous range in carbon isotopic ratios these normal nitrogen ratios cannot be intrinsic and most likely are the result of isotopic equilibration, either on the meteorite parent body or in the laboratory. Apparently, elements such as nitrogen are much more mobile in graphite than in SiC. An exception are graphite grains of low density (≤ 2.05 g cm^{-3}), which have anomalous nitrogen (Figure 12). Low-density (LD) graphite grains have in general higher trace-element concentrations than those with higher densities and for this reason have been studied for their isotopic compositions in detail (Travaglio *et al.*, 1999). Those with nitrogen anomalies have ^{15}N excesses (Figure 12). Many LD grains have large ^{18}O excesses (Amari *et al.*, 1995c) and high ^{26}Al/^{27}Al ratios that almost reach those of SiC X grains (Figure 12) and are much higher than those of mainstream SiC grains (Figure 5). ^{18}O excesses are correlated with ^{12}C/^{13}C ratios. Many grains for which silicon isotopic ratios could be determined with sufficient precision show ^{28}Si excesses, although large ^{29}Si and ^{30}Si excesses are also seen. The similarities of the isotopic signatures with those of SiC X point to an SN origin of LD graphite grains. The ^{18}O excesses are compatible with such an origin. Helium burning produces ^{18}O from ^{14}N, which dominates the CNO isotopes in material that had undergone hydrogen burning via the CNO cycle. As a consequence, the H/C zone in pre-SNII massive stars (see Figure 9), which experienced partial helium burning, has a high ^{18}O abundance (Woosley and Weaver, 1995). Wolf-Rayet stars during the WN–WC transitions are predicted to also show ^{12}C, ^{15}N, and ^{18}O excesses and high ^{26}Al/^{27}Al ratios (Arnould *et al.*, 1997) but also large excesses in ^{29}Si and ^{30}Si and are therefore excluded for LD graphite grains with ^{28}Si excesses.

There are additional features that indicate an SN origin of LD graphite grains. A few grains show evidence for ^{44}Ti (Nittler *et al.*, 1996), others have large excesses of ^{41}K, which must be due to the decay of the radioisotope ^{41}Ca ($T_{1/2} = 1.05 \times 10^{5}$ yr) (Amari *et al.*, 1996). Inferred ^{41}Ca/^{40}Ca ratios are much higher (0.001–0.01) than those predicted for the envelopes of AGB stars (Wasserburg *et al.*, 1994) but are in the range expected for the carbon- and oxygen-rich zones of type II supernovae, where neutron capture leads to the production of ^{41}Ca (Woosley and Weaver, 1995). Measurements of calcium isotopic ratios in grains without evidence for ^{44}Ti show excesses in ^{42}Ca, ^{43}Ca, and ^{44}Ca, with ^{43}Ca having the largest excess (Amari *et al.*, 1996; Travaglio *et al.*, 1999). This pattern is best

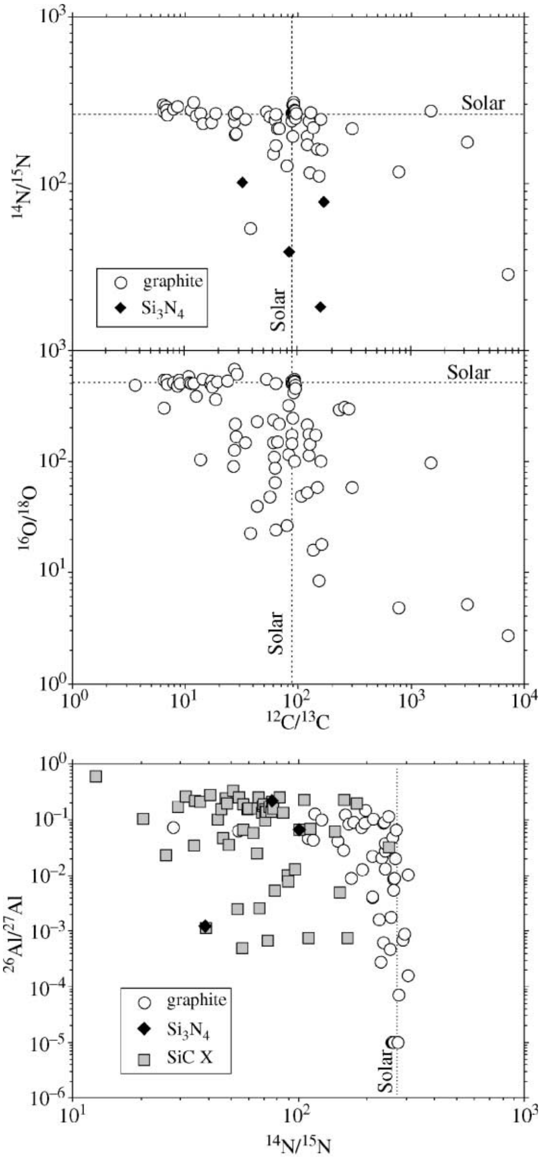

Figure 12 Nitrogen, oxygen, carbon, and aluminum isotopic ratios measured in individual low-density graphite grains. Also shown are data for presolar Si_3N_4 and SiC grains of type X (source Zinner, 1998a).

explained by neutron capture in the He/C and O/C zones (Figure 9) of type II supernovae. In cases where titanium isotopic ratios have been measured (Amari *et al*., 1996; Nittler *et al*., 1996; Travaglio *et al*., 1999), they show large excesses in ^{49}Ti and smaller ones in ^{50}Ti. This pattern also indicates neutron capture and is well matched by predictions for the He/C zone (Amari *et al*., 1996). However, large ^{49}Ti excesses in grains with relatively low (10–100) $^{12}C/^{13}C$ ratios can only be explained if contributions from the decay of ^{49}V are considered (Travaglio *et al*., 1999). Nicolussi *et al*. (1998c) have reported RIMS measurements of zirconium and molybdenum isotopic ratios in graphite grains from the highest density fraction (2.15–2.20 g cm^{-3}), but no other isotopic ratios had been measured. Several grains show s-process patterns for zirconium and molybdenum, similar to those exhibited by mainstream SiC grains, although two grains with a distinct s-process pattern for zirconium have normal molybdenum. Two grains have extreme ^{96}Zr excesses, indicating an SN origin, but the molybdenum isotopes in one are almost normal. Molybdenum, like nitrogen, might have suffered isotopic equilibration in graphite. High-density graphite grains apparently come from AGB stars as previously indicated by the krypton data (Amari *et al*., 1995b) and from supernovae. It remains to be seen whether also LD grains have multiple stellar sources.

In order to obtain better constraints on theoretical models of SN nucleosynthesis, Travaglio *et al*. (1999) tried to match the isotopic compositions of LD graphite grains by performing mixing calculations of different type II SN layers (Woosley and Weaver, 1995). While the results reproduce the principal isotopic signatures of the grains, there remain several problems. The models do not produce enough ^{15}N and yield too low $^{29}Si/^{30}Si$ ratios. The models also cannot explain the magnitude of $^{26}Al/^{27}Al$, especially if SiC X grains are also considered, and give the wrong sign in the correlation of this ratio with the $^{14}N/^{15}N$ ratio. Furthermore, large neutron-capture effects observed in calcium and titanium can be only achieved in a mix with $O > C$. Clayton *et al*. (1999) proposed a kinetic condensation model that allows formation of graphite in the high-radiation environment of SN ejecta even when $O > C$, which relaxes the chemical constraint on mixing. However, it remains to be seen whether SiC and Si_3N_4 can also form under oxidizing conditions. Additional information about the formation environment of presolar graphite is, in principle, provided by the presence of indigenous polycyclic aromatic hydrocarbons (PAHs) (Messenger *et al*., 1998). PAHs with anomalous carbon ratios show different mass envelopes, which indicate different formation conditions.

A few graphite grains appear to come from novae. Laser extraction GMS of single grains show that, like SiC grains, only a small fraction contains evidence for Ne-E. Two of these grains have $^{20}Ne/^{22}Ne$ ratios that are lower than ratios predicted to result from helium burning in any known stellar sources, implying decay of ^{22}Na (Nichols *et al*., 2003). Furthermore, their ^{22}Ne is not accompanied by ^{4}He, expected if neon was implanted. The $^{12}C/^{13}C$ of these two grains are 4 and 10, in the range of SiC grains with a putative nova origin. Another graphite grain with $^{12}C/^{13}C = 8.5$ has a large ^{30}Si excess of 760‰ (Amari *et al*., 2001a).

In summary, although a few graphite grains have the isotopic signature expected for condensates from AGB stars (isotopically heavy carbon and light nitrogen, enhanced abundances of s-process elements) and fewer still those of nova grains, the majority seems to have an SN origin (see also Figure 6). This remains an unsolved puzzle because stars that produce SiC are also expected to form graphite and the apparent underabundance of graphite from AGB stars points to deficiencies in the current understanding of the condensation of carbonaceous phases in carbon-rich stellar atmospheres and the survival of different grain types in the ISM.

1.02.9 OXIDE GRAINS

In contrast to the carbonaceous presolar phases, presolar oxide grains apparently do not carry any "exotic" noble gas component. They have been identified by ion microprobe oxygen isotopic measurements of single grains from acid residues free of silicates. In contrast to SiC, essentially all of which is of presolar origin, most oxide grains found in meteorites formed in the solar system and only a small fraction is presolar. The oxygen isotopic compositions of the presolar oxide minerals identified so far are plotted in Figure 13. They include 198 corundum or likely corundum grains (Huss *et al.*, 1994; Hutcheon *et al.*, 1994; Nittler *et al.*, 1994, 1997, 1998; Nittler and Alexander, 1999; Strebel *et al.*, 1996; Choi *et al.*, 1998, 1999; Krestina *et al.*, 2002), 41 spinel grains (Nittler *et al.*, 1997; Choi *et al.*, 1998; Zinner *et al.*, 2003), and 5 hibonite grains (Choi *et al.*, 1999; Krestina *et al.*, 2002). These numbers, however, cannot be used to infer relative abundances of these mineral phases. Most presolar corundum and hibonite grains are ≥ 1 μm. In contrast, most presolar spinels were found by NanoSIMS analysis of grains down to 0.15 μm in diameter. At this size, the abundance of presolar spinel among all spinel grains is $\sim 3\%$, whereas it is $\leq 0.5\%$ among >1 μm spinel grains. Furthermore, searches for presolar oxide grains have been made in different types of residues, some containing spinel, others not. Another complication is that more than half of all presolar corundum grains have been found by automatic direct $^{18}O/^{16}O$ imaging searches in the ion microprobe (Nittler *et al.*, 1997), a method that does not detect grains with anomalies in the $^{17}O/^{16}O$ ratio but with close-to-normal $^{18}O/^{16}O$. The oxygen isotopic distribution of corundum in Figure 13, therefore, does not reflect the true distribution. NanoSIMS oxygen isotopic raster imaging of tightly packed submicron grains from the Murray CM2 chondrite led to the identification of an additional 252 presolar spinel and 32 presolar corundum grains (Nguyen *et al.*, 2003).

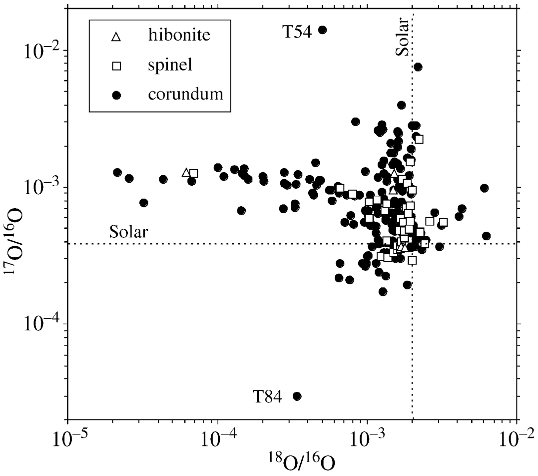

Figure 13 Oxygen isotopic ratios in individual oxide grains (sources Nittler *et al.*, 1997, 1998; Chai *et al.*, 1998, 1999; Krestina *et al.*, 2002, unpublished data; Zinner *et al.*, 2003).

Nittler *et al.* (1997) have classified presolar oxide grains into four different groups according to their oxygen isotopic ratios. Grains with $^{17}O/^{16}O >$ solar (3.82×10^{-4}) and $0.001 < {}^{18}O/^{16}O <$ solar (2.01×10^{-3}), comprising group 1, have oxygen isotopic ratios similar to those observed in RG and AGB stars (Harris and Lambert, 1984; Harris *et al.*, 1987; Smith and Lambert, 1990), indicating such an origin also for the grains. These compositions can be explained by hydrogen burning in the core of low-to-intermediate-mass stars followed by mixing of core material into the envelope during the first dredge-up (also second dredge-up in low-metallicity stars with $M > 3M_{\odot}$) (Boothroyd *et al.*, 1994; Boothroyd and Sackmann, 1999). Variations in $^{17}O/^{16}O$ ratios mainly correspond to differences in stellar mass, while those in $^{18}O/^{16}O$ can be explained by assuming that stars with different metallicities contributed oxide grains to the solar system. According to galactic chemical evolution models, $^{17}O/^{16}O$ and $^{18}O/^{16}O$ ratios are expected to increase as a function of stellar metallicity (Timmes *et al.*, 1995). Grains with depletions in both ^{17}O and ^{18}O (group 3) could thus come from low-mass stars (producing only small ^{17}O enrichments) with lower than solar metallicity (originally having lower than solar $^{17}O/^{16}O$ and $^{18}O/^{16}O$ ratios). The oxygen isotopic ratios of group 3 grains have been used to obtain an estimate of the age of the galaxy (Nittler and Cowsik, 1997). Group 2 grains have ^{17}O excesses and large ^{18}O depletions $(^{18}O/^{16}O < 0.001)$. Such depletions cannot be produced by the first and second dredge-up but have been successfully explained by extra mixing (cool bottom processing) of low-mass $(M < 1.65M_{\odot})$ stars during the AGB phase that circulates material from the envelope through regions close to the hydrogen-burning shell (Wasserburg *et al.*, 1995; Denissenkov and Weiss, 1996; Nollett *et al.*, 2003). Group 4 grains have both ^{17}O and ^{18}O excesses. If they originated from AGB stars they could either come from low-mass stars, in which ^{18}O produced by helium burning of ^{14}N during early pulses was mixed into the envelope by third dredge-up (Boothroyd and Sackmann, 1988) or from stars with high metallicity. More likely for the grains with the largest ^{18}O excesses is an SN origin as suggested by Choi *et al.* (1998) if ^{18}O-rich material from the He/C zone can be admixed to material from oxygen-rich zones.

The only grain that has the typical isotopic signature expected for SN condensates, namely, a large ^{16}O excess, is grain T84 (Figure 13) (Nittler *et al.*, 1998). All oxygen-rich zones (O/C, O/Ne, O/Si—see Figure 9) are dominated by ^{16}O (Woosley and Weaver, 1995; Thielemann *et al.*, 1996; Rauscher *et al.*, 2002). The paucity of such grains, whose abundance is expected to dominate that of carbonaceous phases with an SN origin, remains an unsolved mystery. It has been suggested that oxide grains from supernovae are smaller than those from red giant stars, but recent measurements of submicron grains have not uncovered any additional oxides with large ^{16}O excesses (Zinner *et al.*, 2003; Nguyen *et al.*, 2003). Another grain that does not fit into the four groups is T54 (Nittler *et al.*, 1997). This grain could come from a star with $>5M_\odot$ that experienced hot bottom burning, a condition during which the convective envelope extends into the hydrogen-burning shell (Boothroyd *et al.*, 1995; Lattanzio *et al.*, 1997).

Some but not all grains in the four groups show evidence for initial ^{26}Al (Nittler *et al.*, 1997; Choi *et al.*, 1998, 1999; Krestina *et al.*, 2002). Because ^{26}Al is produced in the hydrogen-burning shell (Forestini *et al.*, 1991), dredge-up of material during the TP AGB phase is required, and grains without ^{26}Al must have formed before their parent stars reached this evolutionary stage. Initial ^{26}Al/^{27}Al ratios are highest in group 2 grains (Nittler *et al.*, 1997; Choi *et al.*, 1998), which is explained if these grains formed in the later stages of the AGB phase when more ^{18}O had been destroyed by cool bottom processing and more ^{26}Al dredged up (Choi *et al.*, 1998). Titanium isotopic ratios have been determined in a few presolar corundum grains (Choi *et al.*, 1998). The observed ^{50}Ti excesses agree with those predicted to result from neutron capture in AGB stars. The depletions in all titanium isotopes relative to ^{48}Ti found in one grain indicate that, just as for SiC grains, galactic evolution affects the isotopic compositions of the parent stars of oxide grains. One hibonite grain was found to have a large ^{41}K excess, corresponding to a ^{41}Ca/^{40}Ca ratio of 1.5×10^{-4}, within the range of values predicted for the envelope of AGB stars (Wasserburg *et al.*, 1994).

To date, all attempts to identify presolar silicates in primitive meteorites have been unsuccessful (Nittler *et al.*, 1997; Messenger and Bernatowicz, 2000). However, Messenger *et al.* (2003) have discovered presolar grains, including silicates, in IDPs. These grains are 0.2–1 μm in size and the discovery was made possible by the high spatial resolution of the NanoSIMS. It remains to be seen whether meteorites also contain presolar silicates in this size range (previous searches have been made mostly on ≥1 μm grains) or whether such grains were preserved only in IDPs.

1.02.10 DIAMOND

Although diamond is the most abundant presolar grain species (~500 ppm) and was the first to be isolated (Lewis *et al.*, 1987), it remains the least understood. The only presolar isotopic signatures (indicating an SN origin) are those of Xe-HL and tellurium (Richter *et al.*, 1998), to a marginal extent also those of strontium and barium (Lewis *et al.*, 1991). However, the carbon isotopic composition of bulk diamonds is essentially the same as that of the solar system (Russell *et al.*, 1991, 1996) and diamonds are too small (the average size is ~2.6 nm—hence nanodiamonds) to be analyzed as single grains. At present, it is not known whether or not this normal carbon isotopic composition is the result of averaging over grains that have large carbon isotopic anomalies, and whether all nanodiamonds are of presolar origin. Nitrogen shows a ^{15}N depletion of 343‰, but isotopically light nitrogen is produced by the CN cycle in all stars and is therefore not very diagnostic. More recent measurements have shown that the nitrogen isotopic ratio of Jupiter (Owen *et al.*, 2001) is very similar to that of the nanodiamonds, which therefore is not necessarily a presolar signature. Furthermore, the concentration of Xe-HL is such that only one diamond grain in a million contains a xenon atom. As of early 2000s, all attempts to separate different, isotopically distinct, components among nanodiamonds have met with only limited success. Stepped pyrolysis indicates that nitrogen and the noble gas components Xe-HL and Ar-HL are decoupled, with nitrogen being released at lower temperature (Verchovsky *et al.*, 1993a,b), and it is likely that nitrogen and the exotic gases are located in different carriers. A solar origin of a large fraction of the nanodiamonds remains a distinct possibility (Dai *et al.*, 2002).

The light and heavy isotope enrichment in Xe-HL has been interpreted as being due to the p- and r-process, and thus requires an SN origin (Heymann and Dziczkaniec, 1979, 1980; Clayton, 1989). In one model, Xe-H is made by a short neutron burst, with neutron densities intermediate between those characteristic for the r- and s-processes (Clayton, 1989; Howard *et al.*, 1992). Ott (1996) kept the standard r-process but proposed that xenon is separated from iodine and tellurium precursors on a timescale of a few hours after their production. Measurements of tellurium isotopes in nanodiamonds show almost complete absence of the isotopes ^{120}Te, $^{122-126}$Te, and a slight excess of ^{128}Te relative to ^{130}Te (Richter *et al.*, 1998). This pattern agrees much better with a standard r-process and early element separation than with the neutron burst model. Clayton and co-workers (Clayton, 1989; Clayton *et al.*, 1995) have also tried to attribute the diamonds and their carbon and nitrogen isotopic compositions to a type II supernova. This requires mixing of contributions from different SN zones. In contrast, Jørgensen (1988) proposed that diamond and Xe-HL were produced by different members of a binary system of low-mass ($1-2M_\odot$) stars, diamond in the winds of one member, a carbon star, while Xe-HL by the other, which exploded as a type Ia supernova. However, at present we do not have an unambiguous identification of the origin of the Xe-HL and tellurium, and of the diamonds (in case they have a different origin).

1.02.11 CONCLUSION AND FUTURE PROSPECTS

The study of presolar grains has provided a wealth of information on galactic evolution, stellar nucleosynthesis, physical properties of stellar atmospheres, and conditions in the solar nebula and on meteoritic parent bodies. However, there are still many features that are not well understood with existing models of nucleosynthesis and stellar evolution and stellar structure. Examples are the carbon and nitrogen isotopic compositions of SiC A + B grains, ^{15}N and ^{29}Si excesses in SN grains, and the paucity of oxide grains from supernovae. The grain data, especially correlated isotopic ratios of many elements, thus provide a challenge to nuclear astrophysicist in tightening constraints on theoretical models.

Continuing instrumental developments allow us to make new and more measurements on the grains and likely lead to new discoveries. For example, the NanoSIMS features high spatial resolution and sensitivity, making isotopic analysis of small grains possible, and this capability has already resulted in the discovery of presolar silicate grains in IDPs (Messenger *et al.*, 2003) and the identification of a large number of presolar spinel grains (Zinner *et al.*, 2003; Nguyen *et al.*, 2003). The NanoSIMS will also make it possible to analyze internal grains that have been studied in detail in the TEM (Stadermann *et al.*, 2003). Another example is the application of RIMS to grain studies. As the number of elements that can be analyzed is being expanded (e.g., to the rare-earth elements), unexpected discoveries such as the molybdenum isotopic patterns in SiC X grains (Pellin *et al.*, 1999) will probably result. RIMS measurements can also be made on grains, such as graphites, for which the isotopic ratios of many elements are measured with the ion microprobe.

It is clear that the discovery of presolar grains and their detailed study have opened a new and fruitful field of astrophysical research.

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