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CIRCUMSTELLAR Fe OXIDE FROM THE ACFER 094 CARBONACEOUS CHONDRITE

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ABSTRACT

We report the discovery of a unique Fe- and O-bearing circumstellar grain from the Acfer 094 ungrouped carbonaceous chondrite. The grain has a close-to-solar $^{17}O/^{16}O$ ratio and an $^{18}O/^{16}O$ ratio that is 1.34 times the solar value. Iron isotopic compositions show depletions of 100‰–200‰ in both ^{54}Fe and ^{57}Fe, relative to ^{56}Fe. No evidence of excess ^{60}Ni from the decay of extinct ^{60}Fe was observed. Auger elemental spectra show that the grain is compositionally similar to wüstite (FeO), but may contain a small amount of Mg in addition to Fe and O. The solid solution series magnesiowüstite, (Mg,Fe)O, is predicted to form under nonequilibrium conditions in oxygen-rich asymptotic giant branch (AGB) stars with low mass-loss rates, and Fe-rich magnesiowüstite has been proposed as the carrier of the 19.5 μm feature observed in the spectra of certain low–mass-loss AGB stars that show little silicate emission. Although the isotopic data are ambiguous, these observations argue in favor of an AGB source for this Fe oxide grain.

Subject headings: astrochemistry — nuclear reactions, nucleosynthesis, abundances

1. INTRODUCTION

The field of astrophysics has been revolutionized in the last 20+ yr through the discovery of presolar grains that survived the formation of the solar system and were incorporated into extraterrestrial materials available for laboratory study (e.g., Bernatowicz & Zinner 1997; Zinner 2004). These grains are found, in more or less pristine form, in primitive meteorites and are identified on the basis of their anomalous isotopic compositions. The isotopic composition of any given circumstellar grain reflects the composition of the stellar atmosphere in which it condensed, which in turn is determined by factors including the galactic history of the material from which the star formed, its mass, the nucleosynthetic processes taking place in the star and the amount of mixing that takes place to dredge up material from the star's interior to the envelope where grain formation occurs. Thus, studies of presolar grains have the potential to provide detailed information not only about the grains themselves and their individual histories, but also about processes occurring in their stellar sources during formation, as well as nucleosynthesis and stellar evolution. The predominant presolar phases found in meteorites include nanodiamonds, silicon carbide, graphite, silicon nitride, silicates, and oxides such as corundum, spinel and hibonite (e.g., Zinner 2004).

Acfer 094 is one of the most primitive, i.e., unaltered, carbonaceous chondrites in the meteorite collection and contains high abundances of both presolar silicate and oxide grains (Nguyen et al. 2007). We have been carrying out combined NanoSIMS and Auger Nanoprobe analyses of size-separated fractions of Acfer 094 matrix material in order to investigate the isotopic and elemental compositions of presolar grains present in this material. Here we report on the discovery of a unique grain, 34C-10, that consists essentially only of Fe and O.

2. EXPERIMENTAL

Grain size separates (0.5–1.0 μm) of Acfer 094 matrix material were dispersed onto high-purity Au foil in order to identify presolar grains with the Washington University Cameca NanoSIMS 50 ion microprobe. The NanoSIMS measurements to initially locate isotopically anomalous grains were carried out in raster ion imaging mode by scanning a Cs^+ primary ion beam over areas of either 20×20 μm (512^2 pixels) or 10×10 μm (256^2 pixels) and simultaneously collecting secondary ions of $^{12}C^-$, $^{13}C^-$, $^{16}O^-$, $^{17}O^-$, $^{18}O^-$ (or in some cases $^{16}O^-$, $^{17}O^-$, $^{18}O^-$, $^{28}Si^-$, $^{24}Mg^{16}O^-$), as well as secondary electrons. Following the initial identification of Acfer 094 grain 34C-10 as isotopically anomalous, a second close-up (5×5 μm; 256^2 pixels) measurement was carried out to more precisely define the O isotopic composition of this grain. A detailed description of the analysis procedures and data reduction methods is given by Stadermann et al. (2005). After elemental characterization of grain 34C-10 (see below), we also carried out Fe isotopic analyses of this grain. These measurements were made with an O^- primary beam and were done in combined analysis mode, in which multicollection is combined with magnetic peak jumping. Analyses were carried out in so-called grain mode, in which the 10×10 μm measurement area was initially located and imaged in $^{56}Fe^+$. Grains within this region were then selected and measured by rastering the beam over the individual area of interest. The setup included three magnetic fields in which the following masses were measured; magnetic field 1: $^{28}Si^+$, $^{52}Cr^+$, $^{54}Fe^+$, $^{57}Fe^+$, and $^{59}Co^+$ in detectors 1–5; magnetic field 2: $^{56}Fe^+$, $^{58}Fe^+$, and $^{61}Ni^+$ in detectors 2–4; magnetic field 3: $^{60}Ni^+$ and $^{62}Ni^+$ in detectors 2–3. Although measurements were done at high mass resolution, sufficient to separate $^{56}FeH^+$ from $^{57}Fe^+$, some interferences could not be separated. Specifically, $^{54}Cr^+$ cannot be separated from $^{54}Fe^+$, and $^{58}Ni^+$ cannot be separated from $^{58}Fe^+$. We were able to make a correction for ^{54}Fe amounting to about 10‰ on the standard and 5‰ on grain 34C-10, assuming terrestrial Cr isotopic compositions. However, estimates of ^{58}Ni contributing at mass 58 were more than 80% for all grains measured, and thus, the $^{58}Fe/^{56}Fe$ ratio is highly uncertain. Therefore, only $^{54}Fe/^{56}Fe$ and $^{57}Fe/^{56}Fe$ ratios are reported here. An Fe,Ni metal standard was used for tuning and calibration of the isotopic ratios, and the data were normalized to the average composition of bulk Acfer 094 matrix grains, which are assumed to have solar Fe isotopic ratios. The Fe/Ni ratio was calculated using sensitivity factors determined from oxide and silicate standards measured with the Washington University Cameca 3f ion microprobe.

Subsequent to the identification of the presolar grain in the NanoSIMS, the sample mount was moved to the new PHI 700 Auger Nanoprobe at Washington University for high-resolution

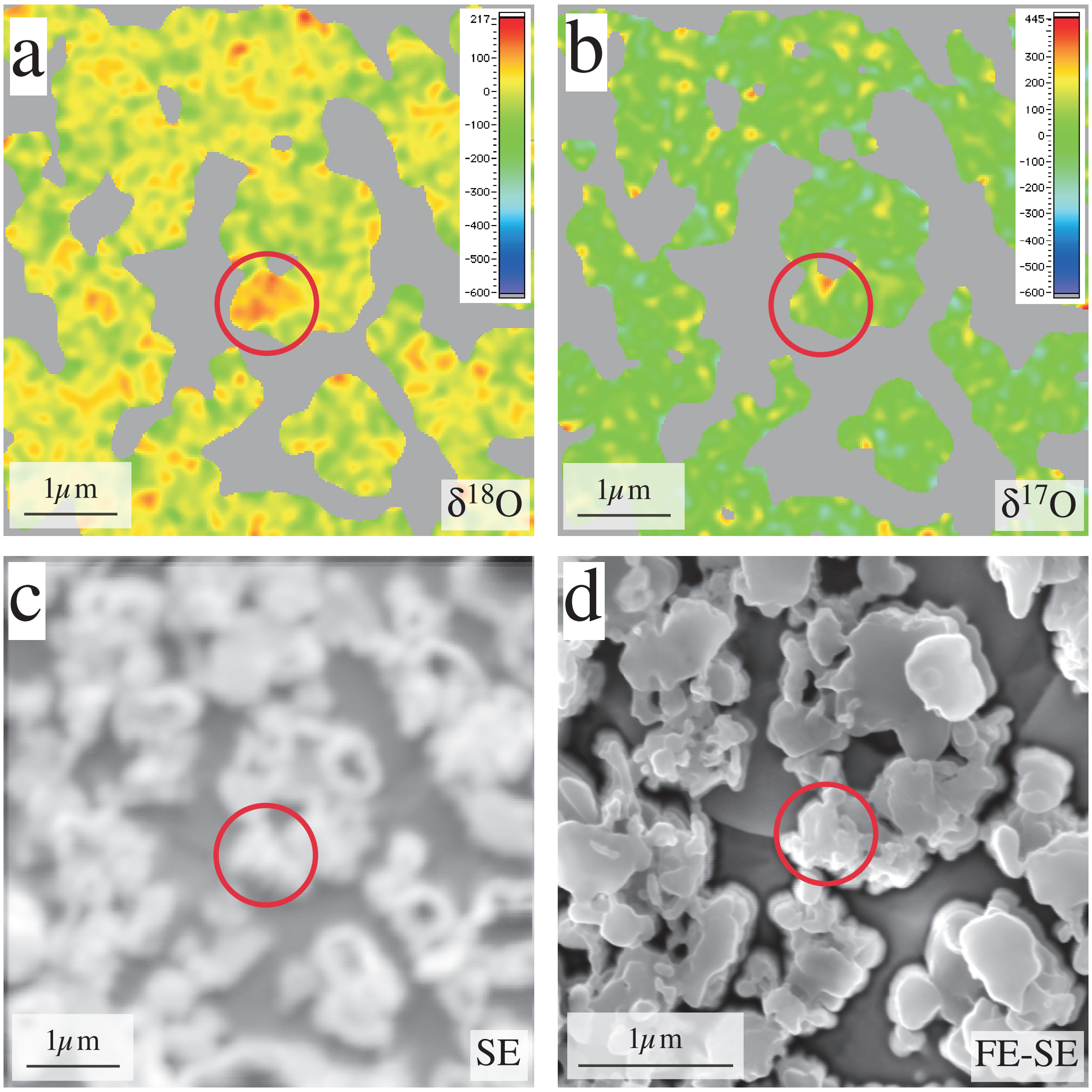

FIG. 1.—False color and gray-scale images of the area containing grain 34C-10: (*a*) $\delta^{18}O$ (permil) map; (*b*) $\delta^{17}O$ (permil) map; (*c*) NanoSIMS secondary electron (SE) image; and (*d*) Auger Nanoprobe field emission secondary electron (FE-SE) image. Note that the field of view of (*d*) is different from that of (*a*)–(*c*).

imaging of the grain and in situ elemental measurements. Following brief sputter cleaning with a 2 kV 1 μA Ar^+ ion beam to remove atmospheric surface contamination, Auger elemental spectra from 100 to 2100 eV were obtained with a 10 kV 10 nA primary electron beam with a diameter of $\sim$20 nm. Auger spectra are typically differentiated, using a seven-point smoothing and Savitsky-Golay differentiation routine, prior to peak identification and quantification. For grain 34C-10, quantification was carried out using pure element sensitivity factors (Childs et al. 1995), and a magnetite standard was measured in order to evaluate the relative proportions of Fe and O in the grain. Previous work on silicate standards has shown that sputtering in the NanoSIMS does not lead to any compositional artifacts in the Auger measurements (unpublished data from our laboratory). High-resolution (3×3 μm; 256^2 pixels) maps of selected elements (Fe, Mg, Si, O, K, S, and C) were also acquired of the area containing the grain.

3. RESULTS

Figure 1 shows oxygen isotopic images ($\delta^{18}O$ and $\delta^{17}O$, displaying permil [‰] deviations from normal) of the area containing grain 34C-10, together with secondary electron images from the NanoSIMS and Auger Nanoprobe. The oxygen isotopic composition of this grain is compared with other presolar grains from Acfer 094 in Figure 2. The anomalous grain has essentially normal $^{17}O/^{16}O$, but has an $^{18}O/^{16}O$ ratio that is elevated by 34% ($^{17}O/^{16}O = 4.12 \pm 0.14 \times 10^{-4}$; $^{18}O/^{16}O = 2.68 \pm 0.04 \times 10^{-3}$). Based on this composition, it can be classified as a group 4 grain (e.g., Nittler et al., 1997). The grain is about 400 nm across, with a roughly triangular shape, and the morphology suggests that it may be an aggregate of several smaller subgrains (Fig. 1*d*).

Auger elemental maps show that the grain consists essentially only of Fe and O (Fig. 3). Some of the point spectra taken on the grain (Fig. 4) indicate that small amounts ($\sim$2 atomic percent [at.%]) of Mg may be present, but no Si, Al, or Ca were detected. Comparison of these spectra with those of a magnetite (Fe_3O_4) standard shows that grain 34C-10 has a higher proportion of Fe to O than magnetite (atomic Fe $\pm$ Mg : O $\sim$ 1.1 vs. 0.75) and is compositionally similar to wüstite (FeO). Wüstite together with periclase (MgO) forms the solid solution series magnesiowüstite ($Mg_xFe_{1-x}O$).

Following Auger characterization, we returned grain 34C-10 to the NanoSIMS for Fe isotopic measurements. Figure 5 shows the Fe isotopic composition of the presolar grain compared to those of adjacent grains from the Acfer 094 sample mount.

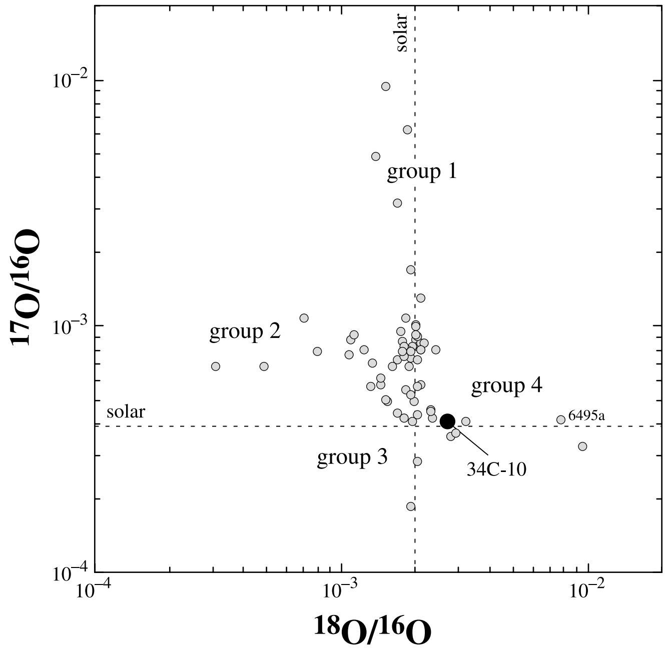

FIG. 2.—Oxygen three-isotope plot showing the composition of grain 34C-10 (*black*) compared to other presolar silicate and oxide grains from Acfer 094 (*gray*). Also indicated are the compositional regions of the four groups of oxide grains defined by Nittler et al. (1997). Acfer 094 data are from Nguyen & Zinner (2004), Mostefaoui & Hoppe (2004), Nagashima et al. (2004), Hoppe et al. (2005), Vollmer et al. (2006), Bland et al. (2007), Nguyen et al. (2007), and unpublished data from our laboratory.

Grain 34C-10 has an anomalous Fe isotopic composition, with depletions in ^{54}Fe and ^{57}Fe, relative to ^{56}Fe ($\delta^{54}Fe = -110‰ \pm 20‰$; $\delta^{57}Fe = -130‰ \pm 30‰$). However, the size of the area rastered over the grain ($\sim 1 \times 1\ \mu m$) was significantly larger than the size of grain 34C-10 and contained a significant amount of Fe from adjacent isotopically normal grains. Based on the Fe elemental image and the size of our grain, we estimate that approximately half of the Fe in the analysis comes from grain 34C-10. Thus, the true composition of this grain is about a factor of 2 more anomalous ($\delta^{54}Fe = -215‰$; $\delta^{57}Fe = -255‰$) than the actual measured composition (e.g., Fig. 5).

The Ni isotopes (^{60}Ni, ^{61}Ni, and ^{62}Ni) were measured along with the Fe isotopes. Grain 34C-10 has Ni isotopic ratios that are normal within the relatively large errors ($\delta^{60}Ni/^{61}Ni = +300‰ \pm 360‰$; $\delta^{60}Ni/^{62}Ni = -15‰ \pm 150‰$). The measured Fe/Ni ratio is quite low ($\sim$31), but because approximately half of the Fe from this analysis comes from outside of the grain, we do not have a good constraint on the true Fe/Ni ratio of grain 34C-10 itself. The Auger spectra show that Ni abundances in the grain are below the detection limits of about 2–3 at.%, but unfortunately this does not provide strong constraints on the Fe/Ni ratio of the grain. However, we note that within the limits provided by our data, there is no evidence of excess ^{60}Ni that might indicate the former presence of ^{60}Fe.

4. DISCUSSION

Although several other Fe-rich presolar oxide grains, specifically chromite, have been found (Nittler et al. 2005), to our knowledge this is the first observation of a circumstellar FeO grain. Below we discuss the possible origin of this grain in more detail.

4.1. *Stellar Formation of Magnesiowüstite*

The expected condensation behaviors of Fe and Mg oxides have only recently been studied. Ferrarotti & Gail (2003) carried out thermodynamic calculations in order to determine the stability of Fe and Mg oxides in the outflows of oxygen-rich asymptotic giant branch (AGB) and red giant stars. Their calculations show that the solid solution series, magnesiowüstite, is a likely condensate and is predicted to form under nonequilibrium conditions

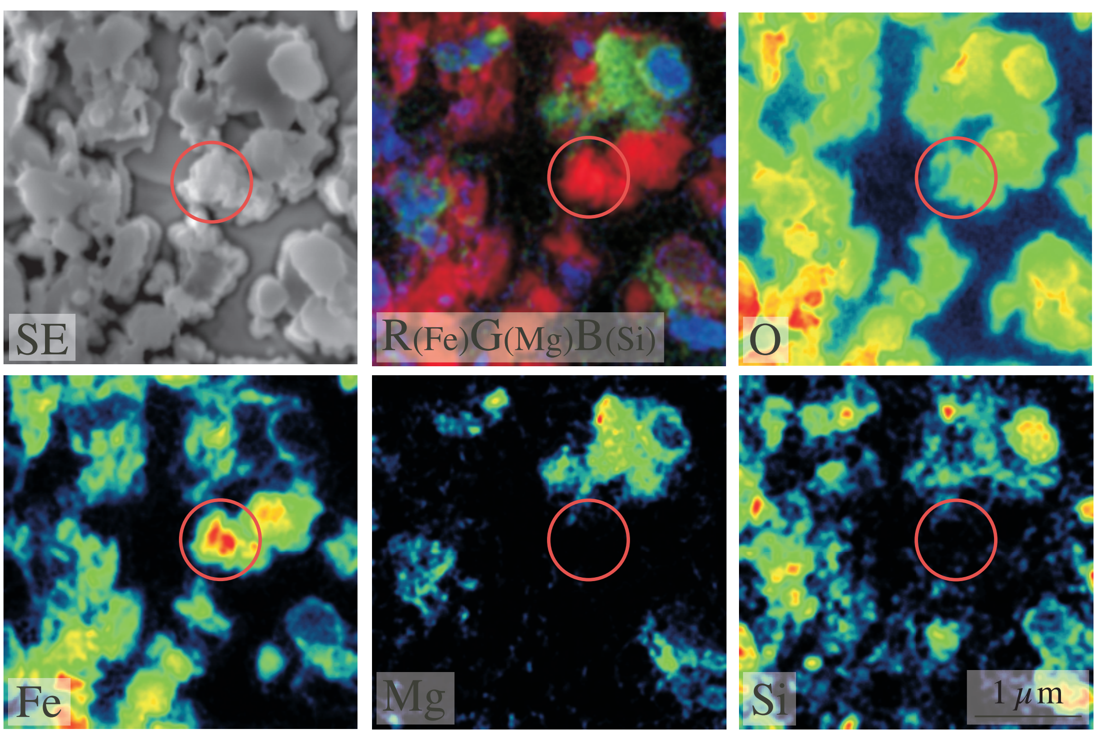

FIG. 3.—Auger elemental and secondary electron images of grain 34C-10 and the surrounding area on the sample mount. The RGB image shows a composite of the three elements, Fe (*red*), Mg (*green*), and Si (*blue*), whose images are displayed in the bottom panels of the figure.

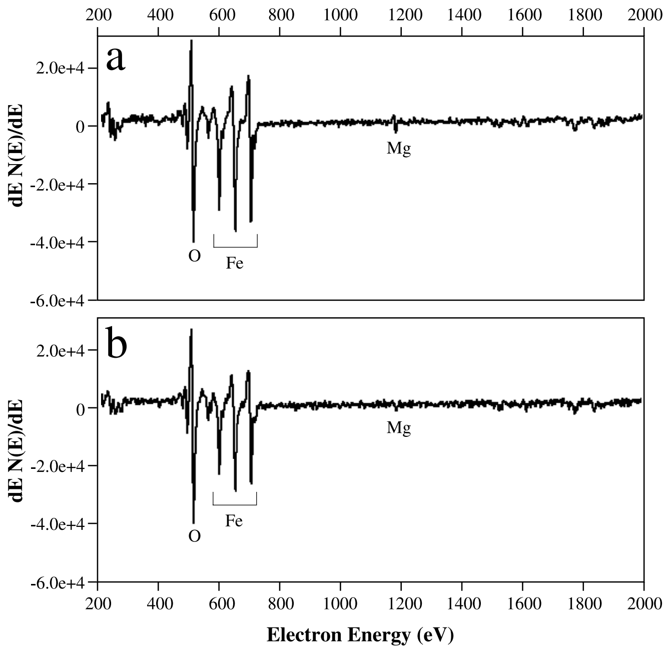

FIG. 4.—Differentiated Auger electron energy spectra of 34C-10: (*a*) spectrum with O, Fe and Mg; (*b*) spectrum with only O and Fe.

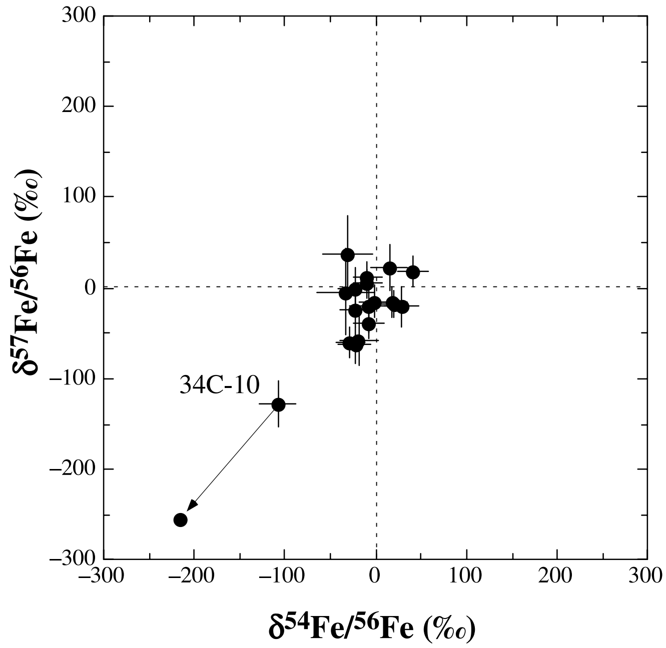

FIG. 5.—Iron three-isotope plot (δ^{54}Fe/^{56}Fe vs. δ^{57}Fe/^{56}Fe) showing the composition of grain 34C-10 compared to other adjacent silicate grains from Acfer 094. Arrow indicates the approximate true composition of the grain, based on estimates of the amount of contamination with isotopically normal Fe; see text for details. Errors are 1 σ.

in the stellar outflows of oxygen-rich AGB stars with low mass-loss rates ($M \leq 4 \times 10^{-6}\ M_\odot\ \mathrm{yr}^{-1}$). At higher mass-loss rates, its condensation is suppressed by the early condensation of silicates. The proportion of Fe incorporated into magnesiowüstite is dependent on temperature. Initially, pure periclase condenses, but as temperatures decrease, large amounts of Fe can be incorporated, with the solid solution containing up to 40% FeO at temperatures below 850 K (Ferrarotti & Gail 2003). This is far less Fe than is present in our grain, which is virtually pure FeO. However, Ferrarotti & Gail (2003) note that the amount of Fe calculated to be present in magnesiowüstite depends on the evaporation coefficients for MgO and FeO, which are not well known.

Other factors could also explain the high Fe content of our grain. Among silicate grains, spectral observations of amorphous grains, specifically their higher absorptivity in the near infrared, indicates that they are more Fe-rich than crystalline silicates (Molster et al. 2002). Indeed, Nguyen et al. (2007) found a significant amount of Fe in an amorphous presolar silicate grain from Acfer 094 characterized by transmission electron microscopy. Thus, it is possible that our Fe-oxide grain is amorphous and contains a higher amount of Fe than expected for nonequilibrium condensation of crystalline magnesiowüstite. Unfortunately, although our Auger analyses provide compositional information, they do not allow us to determine whether or not grain 34C-10 is crystalline or amorphous. Diffusion of Fe into magnesiowüstite could also account for the elevated Fe/Mg ratio compared to predicted compositions. This could occur either in the stellar outflow, shortly after formation of the grain, or possibly as an alteration process on the Acfer 094 parent body. However, if significant introduction of Fe into the grain took place on the parent body, this would require the initial Fe isotopic composition of 34C-10 to have been even more anomalous than the composition determined here. In addition, alteration resulting in a high degree of Fe diffusion into the grain might also be expected to lead to re-equilibration of the oxygen isotopes. Thus, overall it seems more likely that the isotopic (and elemental) compositions observed represent those of the grain at the time of its initial formation in the stellar environment.

In addition, there is some spectral evidence for the presence of magnesiowüstite with high Fe/Mg ratios in the outflows of low-mass AGB stars. Several studies have suggested that FeO could be the carrier of a broad ~20 μm emission feature seen in some oxygen-rich dust shells (e.g., Malfait et al. 1999; Demyk et al. 1999), and Henning et al. (1995) showed that the addition of FeO will broaden the 18 μm band of amorphous silicates and shift it to longer wavelengths. Posch et al. (2002) took the optical constants of Henning et al. (1995) for FeO and MgO and used them to evaluate which solid solution composition in the magnesiowüstite series can account for the 19.5 μm feature observed in the spectra of certain high-temperature, low–mass-loss AGB stars that show little silicate emission. Their results show that this feature is best reproduced with a magnesiowüstite composition of $Mg_{0.1}Fe_{0.9}O$, similar to the very Fe-rich composition of grain 34C-10. The overall emission spectra of the stars studied by Posch et al. (2002) could be effectively modeled as consisting of a mixture of 73% amorphous Al_2O_3 and 27% $Mg_{0.1}Fe_{0.9}O$, indicating that magnesiowüstite could be a significant component of the dust shells of such stars.

4.2. *Oxygen Isotopic Composition of 34C-10*

The oxygen isotopic composition of grain 34C-10 is ^{18}O-rich (Fig. 2), and it can thus be identified as a group 4 grain, based on the classification scheme of Nittler et al. (1997). The origin of group 4 grains is somewhat enigmatic and proposed sources for these grains have included formation in Type II supernovae (e.g., Choi et al. 1998; Messenger et al. 2005; Bland et al. 2007) or AGB stars (Nittler et al. 1997; Mostefaoui & Hoppe 2004).

As discussed by Choi et al. (1998) and Messenger et al. (2005), large enrichments in ^{18}O may be produced in Type II supernovae in the He/C shell during He burning of ^{14}N, the product of previous H burning in the CNO cycle. This region is C-rich, but only a very small amount of material (<0.1% based on the 15 $M_\odot$ supernova model of Rauscher et al. [2002]) from this zone would need to be mixed with O-rich material from the envelope in order to reproduce the oxygen isotopic anomaly observed in grain 34C-10.

Alternatively, there are several possible scenarios for an AGB origin of grain 34C-10. One possibility that is commonly invoked for group 4 grains (e.g., Nittler et al. 1997; Nguyen et al. 2007) is that they originated in high-metallicity low-mass AGB stars, and their oxygen isotopic compositions simply reflect the initial ratios of their parent stars. However, enrichment in both heavy isotopes is expected in this case, whereas grain 34C-10 shows an excess only in ^{18}O. Another possibility, originally suggested by Nittler et al. (1997), is that dredge-up of ^{18}O in thermally pulsing AGB stars (the third dredge-up) can result in ^{18}O excesses in the envelopes of certain low-mass AGB stars. ^{18}O is both created and destroyed during He burning; dredge-up after early thermal pulses before most ^{18}O is destroyed could result in elevated ^{18}O/^{16}O in the envelope (Boothroyd & Sackman 1988). However, although this mechanism was invoked to explain the elevated ^{18}O/^{16}O ratio of the barium star HD 101013 (Harris et al. 1985), observations of most AGB stars that have experienced third dredge-up episodes do not show large ^{18}O excesses (Nittler et al. 1997). Moreover, recent calculations suggest that the third dredge-up is not expected to occur in the first few pulses when ^{18}O is still likely to be present (Stancliffe et al. 2005). Finally, it is possible that grain 34C-10 may have formed in an AGB or red giant star that simply had oxygen isotopic ratios that were slightly different from solar. The isotopic anomaly we observe in this grain is not very large (^{18}O/^{16}O $= 1.34 \times$ the solar value), and it is possible that small effects such as this could arise from local heterogeneities in the interstellar medium (e.g., Nittler et al. 2007).

4.3. *Iron Isotopic Composition of 34C-10*

The Fe isotopic composition measured in grain 34C-10 shows deficits in both ^{54}Fe and ^{57}Fe relative to ^{56}Fe (Fig. 5). Relatively few circumstellar grains have been measured for Fe isotopes. Among presolar silicates, one grain measured by Mostefaoui & Hoppe (2004) has a somewhat higher than solar ^{54}Fe/^{56}Fe ratio (δ^{54}Fe/^{56}Fe $= 116‰ \pm 44‰$); other Fe isotopes were not measured in this grain. The oxygen isotopic composition of this grain is also ^{18}O-enriched (i.e., group 4), although the enrichment is significantly larger than that observed in 34C-10 (Fig. 2, "6495a"). These authors argued for an origin of this grain from an AGB star with somewhat higher than solar metallicity, based on its higher than solar ^{54}Fe/^{56}Fe and ^{18}O/^{16}O ratios.

Iron isotopes have also been measured in both mainstream and type X SiC grains (Marhas et al. 2004, 2007). Mainstream SiC grains, which are thought to have an AGB origin, mostly have normal Fe isotopic compositions, within the relatively large errors (Marhas et al. 2004, 2007), unlike our grain. However, four mainstream grains measured by Marhas et al. (2007) are depleted in ^{57}Fe with solar ^{54}Fe/^{56}Fe. SiC X grains from supernovae exhibit a variety of Fe isotopic compositions (Davis et al. 2002; Marhas et al. 2004, 2007), but typically show enrichments in ^{57}Fe and ^{58}Fe (Davis et al. 2002); Marhas et al. (2007) also found several grains with depleted ^{57}Fe/^{56}Fe or depleted ^{54}Fe/^{56}Fe. However, no SiC X grains measured to date exhibit depletions in both ^{54}Fe and ^{57}Fe like grain 34C-10 studied here.

Partial mixing of different zones in a supernova is typically required to account for the isotopic compositions of supernova grains (e.g., Amari et al. 1992; Choi et al. 1998; Travaglio et al. 1999; Messenger et al. 2005). As noted above, in order to reproduce only the O isotopic composition of grain 34C-10 in a 15 $M_\odot$ supernova, the bulk of the grain must have formed in the outer envelope with only a very small contribution from the He/C zone. Iron isotopic compositions (^{54}Fe/^{56}Fe and ^{57}Fe/^{56}Fe) in the envelope are solar, however, and thus, mixing with a zone depleted in ^{54}Fe and ^{57}Fe is required in order to reproduce the Fe isotopic composition of grain 34C-10. Depletions in ^{54}Fe are observed primarily in the O/Ne and O/C zones, but these zones have elevated ^{57}Fe/^{56}Fe ratios. The only region exhibiting depletions in both ^{54}Fe and ^{57}Fe is a small pocket of the O/Si zone. Substantial mixing with this pocket (~20%–40%, depending on whether the measured or more anomalous, inferred Fe isotopic composition is modeled) is required in order to reproduce the Fe isotopic composition of grain 34C-10. Based on this modeling, the lack of evidence for ^{60}Fe in grain 34C-10 is not unexpected, since ^{60}Fe is produced primarily in the O/Ne and O/C zones, which are not contributing to the formation of the grain. However, mixing with the O/Si zone leads to depleted ^{18}O/^{16}O and ^{17}O/^{16}O compositions, because this zone is strongly enriched in ^{16}O. Addition of more material from the He/C zone can compensate for the depleted ^{18}O/^{16}O, but the ^{17}O/^{16}O ratio remains significantly underestimated compared to the measured composition of grain 34C-10. One scenario that might circumvent this problem would be condensation of the grain as Fe metal in the O/Si pocket, followed by oxidation to FeO in the envelope. However, the large amount of O produced in this zone argues against the formation of such a reduced phase.

We can also consider an AGB origin to try to understand the Fe isotopic composition of our grain. In AGB stars both ^{54}Fe and ^{56}Fe are largely consumed by neutron captures in the He intershell, and thus, mixing of intershell material has little effect on the ^{54}Fe/^{56}Fe ratio in the envelope (Davis et al. 2002). Therefore, grains condensing in the envelopes around AGB stars are expected to have ^{54}Fe/^{56}Fe ratios that reflect the initial ratios of the stars. In contrast, *s*-process enhancements are expected for ^{57}Fe and ^{58}Fe, which are produced in the intershell largely via ^{22}Ne$(\alpha, n)^{25}$Mg on Fe from H-burning ashes (Davis et al. 2002). These are dredged up into the envelope, resulting in enhanced ^{57}Fe/^{56}Fe and ^{58}Fe/^{56}Fe. If grain 34C-10 has an AGB source, the depletion in ^{54}Fe/^{56}Fe would suggest that it originated in a star with initial Fe isotope ratios that were lower than solar. However, the depletion in ^{57}Fe/^{56}Fe remains enigmatic.

5. CONCLUSIONS

The isotopic data for 34C-10 do not unequivocally argue for either a supernova or an AGB origin of this grain. Indeed, the data do not seem to fit well to expected compositions from either type of stellar source. On balance, we currently favor an origin in an AGB star, largely because there seems to be some astronomical evidence for the presence of magnesiowüstite in certain AGB stars. As we noted above, theoretical modeling of the predicted conditions for the condensation of magnesiowüstite shows that this phase can form under nonequilibrium conditions in the stellar outflows of oxygen-rich AGB stars. In addition, this phase, with a composition very similar to that of the grain studied here, can account for the spectral features of a particular class of AGB stars that show little evidence for silicate formation (Posch et al. 2002). However, the isotopic data, in particular, the depletion in ^{57}Fe/^{56}Fe, are not consistent with current model predictions.

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