

**FIB IN THE NANOSIMS.** Ernst Zinner and Frank Gyngard, Laboratory for Space Sciences, Washington University, One Brookings Drive, St. Louis, Mo 63130, USA, ekz@wustl.edu and fmggyngar@wustl.edu.

**Introduction:** In contrast to SiC, only a small fraction of oxide grains in meteorites are of presolar origin and their identification requires isotopic analysis of a large number of grains. The most efficient way to locate presolar oxides is isotopic imaging of tightly packed grain separates or polished meteorite sections [1]. Presolar silicates in interplanetary dust particles and meteorites have been discovered in this way [2, 3]. As discussed by Nguyen et al. [4], one problem of isotopic imaging is that, because of beam overlap onto neighboring grains, isotopic ratios are shifted toward normal values. This is clearly shown in a comparison of O isotopic ratios measured in separated individual spinel grains [5-7] with ratios measured in tightly packed spinels from the CG (grain size 0.45  $\mu\text{m}$ ) and CF (0.15  $\mu\text{m}$ ) fraction of the Murray carbonaceous chondrite [1]. These O isotopic measurements were made in the NanoSIMS with a  $\text{Cs}^+$  primary beam. The situation is even worse for an  $\text{O}^-$  primary beam, required for the analysis of elements such as Mg and Fe, which are detected as positive secondary ions; only very few imaging analyses of these elements have been reported [3, 8]. Even during analysis of distributed grains, it frequently occurs that a presolar grain is located in the close proximity of an isotopically normal grain. Here we report how we achieved uncontaminated isotopic analyses of such grains.

**FIB in the NanoSIMS:** Murray spinel M16 is one of the grains whose O and Mg isotopic compositions have been reported by Zinner et al. [6]. The SEM image at the upper left of Fig. 1, taken after the first O isotopic analysis, shows what we thought to be M16 (a and b) in close proximity of another grain (c). In order to remove grain c we rastered the finely focused primary Cs beam (diameter  $\sim 100$  nm) over this grain and sputtered it away. The SEM picture on the upper right shows the remainder after another set of O and Mg isotopic analyses. At that point it became clear that this remainder actually consisted of two grains. The isotopic images, obtained together with the secondary electron (S.E.) image in the NanoSIMS, show that grain a is the grain with an  $^{17}\text{O}$  excess and  $^{18}\text{O}$  depletion (Fig. 5). While we sputtered away grain b, we discovered that it happened to also be isotopically anomalous, exhibiting an excess in  $^{18}\text{O}$ . (Fig. 5). Finally, we could measure the O and Mg of Murray grain M16 without any dilution by adjacent grains. As seen in Figs. 5 and 6, the final isotopic ratios are much more anomalous than what we had obtained in the initial analyses [6].

Three more examples discussed here are from automatic imaging searches of Murray CG grains reported in a separate abstract [7]. In these measurements, an algorithm identifies candidate grains from a  $20 \times 20 \mu\text{m}^2$   $^{16}\text{O}$  image, whereupon the primary beam is deflected onto individual grains for O isotopic

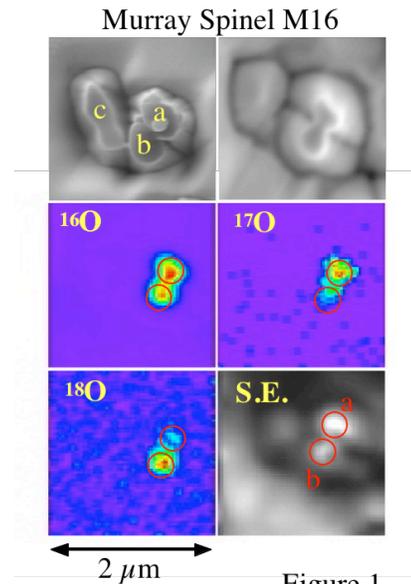


Figure 1

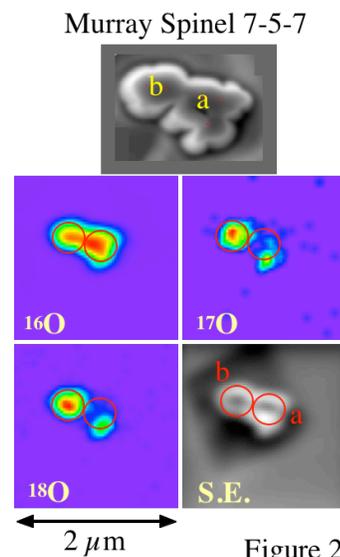
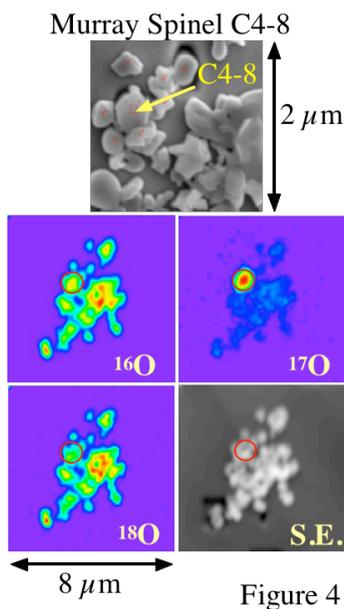
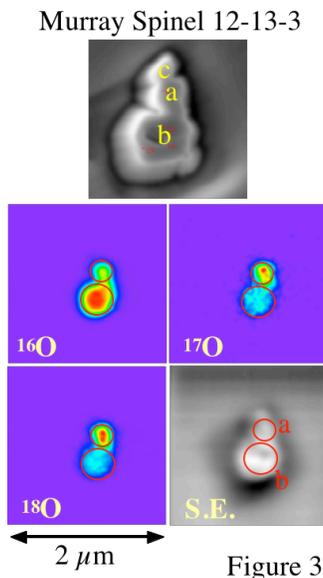


Figure 2

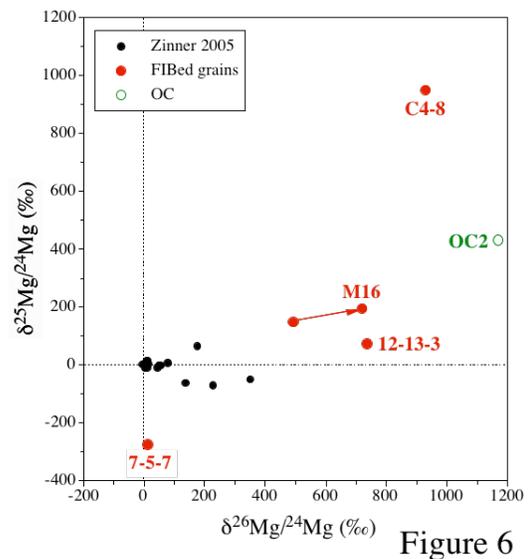
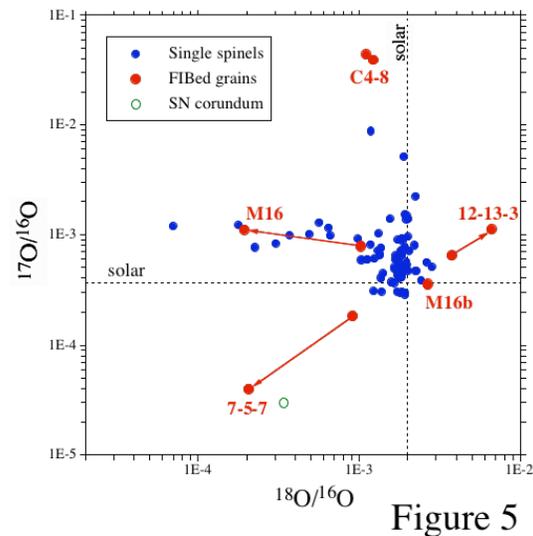
analysis. Fig. 2 shows a case of a grain with depletions in both  $^{17}\text{O}$  and  $^{18}\text{O}$ . The SEM picture taken after the automatic O analysis reveals two adjacent grains. Subsequently obtained O isotopic images show that grain a is anomalous. After removal of grain b and the small attachment below grain a by Cs-beam sputtering, the O isotopic composition of the “naked” spinel grain Murray 7-5-7 has a large  $^{16}\text{O}$  excess (Fig. 5) and plots in close to a corundum grain, for which Nittler identified an origin in a Type II supernova [9]. Its Mg isotopic ratios shows a large depletion in  $^{25}\text{Mg}$  (Fig. 6).

Murray spinel grain 12-13-3 was found by the automatic grain search to have excesses in both  $^{17}\text{O}$  and  $^{18}\text{O}$ . The SEM picture in Fig. 3 in combination with



subsequently obtained O isotopic images reveals it as a fairly small grain of  $\sim 200$  nm (grain a) between a much larger grain (b) and an even smaller grain (c). After FIB removal of these adjacent grains, the true  $^{17}\text{O}$  and  $^{18}\text{O}$  excesses of grain 12-13-3 are much larger, with O isotopic ratios being more than three times solar (Fig. 5). This grain has a large  $^{26}\text{Mg}$  excess but a fairly normal  $^{25}\text{Mg}/^{24}\text{Mg}$  ratio (Fig. 6).

Grain C4-8 is a special case. This grain was located in a pile of other Murray CG grains (Fig. 4) and was therefore not identified as an individual grain by the automatic grain algorithm. However, it stuck out in the O isotopic image because of its enormous  $^{17}\text{O}$  excess, and we analyzed it manually. Because of its large size, FIB removal of the surrounding grains did not result in a large shift of its O isotopic ratios, but we could not have



obtained an uncontaminated Mg isotopic analysis without it. The  $^{17}\text{O}$  excess in grain C4-8 (Fig. 5) is the largest observed to date in any oxide grain. The grain has large  $^{25}\text{Mg}$  and  $^{26}\text{Mg}$  excesses, with the  $^{26}\text{Mg}/^{24}\text{Mg}$  ratios approaching that of grain OC2 [10], but with a  $^{26}\text{Mg}$  excess more than twice that of OC2 (Fig. 6).

The astrophysical implications of the O and Mg isotopic compositions of the grains of this study are discussed in more detail in the accompanying abstract by Gyngard et al. [7]. The new data will be a challenge to theoretical models of stellar nucleosynthesis.

**References:** [1] Nguyen A. et al. (2003) *PASA*, 20, 382-388. [2] Messenger S. et al. (2003) *Science*, 300, 105-108. [3] Nguyen A. N. and Zinner E. (2004) *Science*, 303, 1496-1499. [4] Nguyen A. N. et al. (2007) *ApJ*, 656, 1223-1240. [5] Zinner E. et al. (2003) *GCA*, 67, 5083-5095. [6] Zinner E. et al. (2005) *GCA*, 69, 4149-4165. [7] Gyngard F. et al. (2009) *This conference*. [8] Floss C. et al. (2008) *ApJ*, 672, 1266-1271. [9] Nittler L. R. et al. (1998) *Nature*, 393, 222. [10] Lugaro M. et al. (2007) *A&A*, 461, 657-669.