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$^{40}\text{Ar}/^{39}\text{Ar}$ dating of very fine-grained samples: An encapsulated-vial procedure to overcome the problem of ^{39}Ar recoil loss

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(Received November 12, 1991; revised and accepted March 18, 1992)

ABSTRACT

Foland, K.A., Hubacher, F.A. and Arehart, G.B., 1992. $^{40}\text{Ar}/^{39}\text{Ar}$ dating of very fine-grained samples: An encapsulated-vial procedure to overcome the problem of ^{39}Ar recoil loss. *Chem. Geol. (Isot. Geosci. Sect.)*, 102: 269–276.

$^{40}\text{Ar}/^{39}\text{Ar}$ dating of very fine-grained materials is compromised by the loss of ^{39}Ar by recoil, and also sometimes other Ar isotopes, during irradiation. An encapsulated sample $^{40}\text{Ar}/^{39}\text{Ar}$ procedure is described which overcomes this problem and which produces a K–Ar equivalent date. Measurements indicate that the procedure yields reliable and reproducible results. It offers the potential for dating small amounts of material of many types, for example very fine-grained authigenic sheet silicates from rock cores, which may suffer Ar loss during irradiation.

1. Introduction

It is well established that separated very fine-grained minerals, such as authigenic glauconite and sericite, will lose ^{39}Ar as a result of recoil during neutron irradiation for $^{40}\text{Ar}/^{39}\text{Ar}$ dating (see, e.g., Halliday, 1978; Foland et al., 1984; Hess and Lippolt, 1986). The loss of ^{39}Ar occurs because the recoil energy following production by the n,p transmutation is sufficient to displace the atom $\sim 0.08 \mu\text{m}$ on the average (Turner and Cadogan, 1974), which is a significant distance relative to the physical sizes of small crystals.

Even elementary models are sufficient to demonstrate the magnitude of the possible loss (see Fig. 1); for example, using the formulae

of Foland and Xu (1990) and the recoil range relations of Turner and Cadogan (1974), the recoil of ^{39}Ar out of a $1\text{-}\mu\text{m}$ -thick plate-like grain is $\sim 8\%$ of that produced. While some or even the majority of this ^{39}Ar may recoil into, and be trapped by, an adjacent grain, the loss of ^{39}Ar from very small grains is inevitable and becomes significant as grain size decreases. The problem becomes acute for fine materials such as glauconite where mica platelets may have thicknesses on the order of $\leq 0.1 \mu\text{m}$ (Odin and Matter, 1981; Foland et al., 1984); recoil losses as much as 30% have been documented for such samples (Foland et al., 1984; Hess and Lippolt, 1986).

Another manner in which Ar may be lost during irradiation is from phases, such as alteration products or poorly crystalline material, which are inherently unstable especially if elevated temperatures are attained during neutron bombardment. Hess and Lippolt (1986)

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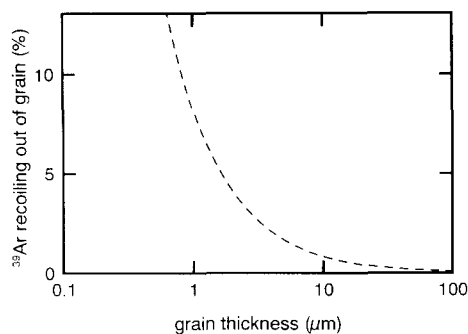


Fig. 1. Percentage of ^{39}Ar which is predicted to recoil out of plate-like grains as a function of grain thickness. Calculation using the geometric formula of Foland and Xu (1990) and the recoil-range relationships of Turner and Cadogan (1974).

report significant ^{39}Ar losses from biotite mineral separates with a low-K bulk concentration and which is attributed to losses from alteration phases. Alteration phases may contain significant ^{39}Ar as a result of recoil capture from adjacent high-K grains. Hess and Lippolt (1986) found that glauconite lost radiogenic ^{40}Ar , demonstrating that some materials may also lose significant amounts of other Ar components, for example radiogenic ^{40}Ar , during irradiation.

Both of these effects can produce incorrect dates. An erroneous $^{40}\text{Ar}/^{39}\text{Ar}$ date will result unless there is a provision for "catching" and reintegrating the ^{39}Ar lost by recoil into the age calculation.

As a result of such problems, normal application of the $^{40}\text{Ar}/^{39}\text{Ar}$ technique is not recommended for separated very fine-grained minerals. Furthermore, the $^{40}\text{Ar}/^{39}\text{Ar}$ technique yields little additional information for such hydrous materials such as glauconite because they decompose at rather low temperatures during vacuum heating for Ar extraction (e.g., Evernden et al., 1960; Foland et al., 1984) and therefore generally render incremental-heating data meaningless. Conventional K-Ar, on the other hand, requires that a sample be available in sufficient quantity and homogeneity to provide suitable splits for separate K and Ar analyses. In counterpoint, how-

ever, the $^{40}\text{Ar}/^{39}\text{Ar}$ technique does have the distinct advantage in terms of sample size because only mg-order quantities are needed and because the heterogeneity problem is obviated. With the sensitivity of modern mass spectrometers, it is typically not sample size which is limiting but rather sample heterogeneities and recoil.

In a study of the timing of mineralization of sediment-hosted disseminated gold deposits (Arehart et al., 1992), we used Ar techniques to date associated "alteration sericite" from sedimentary and igneous host rocks. Facing the difficult, or in some instances impossible, task of obtaining separates appropriate for K-Ar analyses, we investigated a $^{40}\text{Ar}/^{39}\text{Ar}$ approach which quantitatively captures recoiled ^{39}Ar and any ^{40}Ar lost during irradiation. We describe here the procedure found quite satisfactory for determining a K-Ar equivalent date of such material.

2. Procedure

The objective is to capture quantitatively all Ar lost during irradiation and to combine it with that in the sample to produce a single mixture containing all Ar components. This avoids the separate measurements of Ar quantities if, for example, the Ar lost during irradiation and that still contained in the specimen were measured separately.

In brief, our encapsulation procedure is as follows: (1) mineral samples are placed in small vials of silica glass; (2) the vials are attached to a vacuum system, evacuated, and sealed under vacuum; (3) the sealed vials are irradiated along with mineral standards for J -value determination; (4) after irradiation, the sealed vials are heated in a vacuum extraction line to melt the sample and cause failure of the glass vial; (5) Ar lost by the sample during irradiation and captured in the sealed vial, along with Ar evolved from the sample during heating is released into the vacuum system and

mixed; and (6) Ar is processed by the customary procedures.

The encapsulation arrangement is illustrated schematically in Fig. 2. Samples are encapsulated in vials fashioned from high-purity and bubble-free silica glass tubing of 3.4-mm ID and 5-mm OD. Suprasil[®] tubing (Heraeus-Amersil, Inc.) which we use for instrumental neutron activation analysis is used for this. Short lengths (~50 mm) are cleaned by HNO₃ acid washing and then one end is closed to make a flat "bottom". Grains of sample are then loaded into these vials making sure that none adheres to the sides. A small amount of silica glass wool is placed on top of the mineral sample. The glass wool serves to keep the sample in place, to prevent movement of the sample from the bottom during initial evacuation, and to reflect heat during subsequent glass blowing at the top of the vial. The vial is then attached to a high-vacuum line and necked at the top. Typically, 6–12 vials are treated at the same time. The line and samples are baked out overnight at ~100°C to achieve a pressure of $\lesssim 3 \cdot 10^{-8}$ Torr. Each vial is sealed at the neck and removed from the system. During all glass blowing steps, samples are kept cool with water.

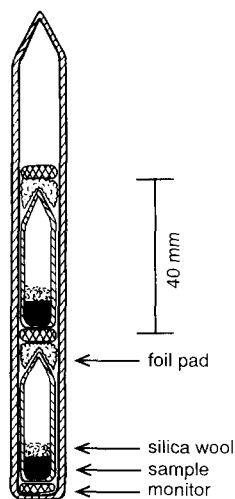


Fig. 2. Schematic illustration of the irradiation arrangement. Two encapsulated vials are shown within a single irradiation tube.

Our experience is that heating of the sample by conduction is not a problem but that radiative transfer can heat the top of the sample to undesirable levels; hence, the glass wool is important to maintaining sample integrity. The overall length of the sealed vial is ~35 mm. The top seal, done under vacuum, is critical and can pose a problem if necked too finely during removal. Several early experiments suffered loss of vacuum integrity due to breakage of tips which were too delicate.

Our normal irradiation procedures have been described previously (Foland et al., 1984, 1989). Normally, foil-wrapped samples and monitors are loaded and then sealed in a 6-mm-ID by 8-mm-OD by ~8-cm-long silica glass tube with as many as 14 such tubes irradiated at one time. For the encapsulation technique, sealed vials containing samples are loaded in the larger irradiation tubes with foil-wrapped monitors on each side for the *J*-value determination by interpolation to the physical position of the encapsulated unknown. Two sealed vials are easily accommodated in each irradiation tube. Some Al foil is used to cushion and provide protection for the fragile top seal of the encapsulation vial (see Fig. 2). Otherwise, there is no difference from normal irradiation procedures.

After irradiation, encapsulation vials are placed in a side arm of a vacuum fusion system for analysis. A vial is dropped into the ~22-cm-long, 1.4-cm-ID crucible of a double-vacuum furnace which is essentially the same as that described by Staudacher et al. (1978). The crucible temperature is raised over a period of ~20 min to ~1300°C which is maintained for 20 min. As temperature is increased, decomposition of hydrous sample inside the vial causes a progressive pressure increase which, coupled with softening of the silica glass, results in rupturing of the vial. The sample is eventually melted so that all Ar is released into the vacuum chamber and mixed. Very little of the silica glass is fused in the process. The liberated gases are processed with the same pro-

TABLE 1

⁴⁰Ar/³⁹Ar analytical data and calculated ages

T ^(*) (°C)	⁴⁰ Ar/ ³⁹ Ar ^(**2)	³⁸ Ar/ ³⁹ Ar ^(**2)	³⁷ Ar/ ³⁹ Ar ^(**2) (×100)	³⁶ Ar/ ³⁹ Ar ^(**2) (×100)	F ^(**3)	³⁹ Ar ^(**4) (%)	⁴⁰ Ar ^(**5) (%)	K/Ca ^(**6)	K/C ^(**7)	Age ^{**8} (Ma)
GL-O (<i>glauconite standard</i>):										
Run 39A7 (<i>J</i> =0.007835, 0.1148 g):										
E-Fuse	7.719	0.0295	5.125	0.3329	6.708	100	86.91	10.2	291	92.4±0.4**9
Run 39A8 (<i>J</i> =0.007995, 0.0531 g):										
E-fuse	7.698	0.0901	5.349	0.3602	6.608	100	85.84	9.77	66.5	92.9±0.2**9
Run 39C7 (<i>J</i> =0.007800, 0.0157 g):										
E-fuse	9.943	0.1162	5.169	1.024	6.891	100	69.31	10.1	50.5	94.5±1.1
MON-4 (<i>biotite, intralaboratory standard</i>):										
Run 39F7 (<i>J</i> =0.007688, 0.0439 g):										
E-Fuse	10.71	0.0558	4.013	0.5488	9.057	100	84.60	13.0	119	121.4±0.4
MCC-3 (<i>sericite, <2 μm</i>):										
Run 41D8 (<i>J</i> =0.007787, 0.0287 g):										
E-Fuse	21.98	3.974	614.6	2.681	14.92	100	67.56	0.09	1.31	198.4±1.6
SED-3 (<i>sericite, <2 μm</i>):										
Run 41E10 (<i>J</i> =0.007843, 0.0298 g):										
E-Fuse	20.40	1.312	12.27	4.854	6.140	100	30.10	4.26	4.04	84.9±2.0
Run 41B7 (<i>J</i> =0.007846, 0.0264 g):										
E-Fuse	20.24	1.194	10.59	4.881	5.885	100	29.08	4.93	4.45	81.4±1.5**9
SED-4 (<i>sericite, <2 μm</i>):										
Run 41C7 (<i>J</i> =0.007696, 0.0271 g):										
E-Fuse	11.15	1.854	45.83	1.022	8.650	100	77.36	1.10	2.83	116.4±0.4**9
Run 41E9 (<i>J</i> =0.007645, 0.0253 g):										
E-Fuse	11.63	1.859	46.68	1.046	8.700	100	74.77	1.12	2.83	116.2±0.6

SED-2 (sericite, <2 μm):

Run 39H8 (J=0.007780, 0.0966 g):										
E-Fuse	9.567	0.0935	2.162	0.5237	7.991	100	83.53	24.2	64.1	108.8 ± 0.3*9
Run 39K9 (J=0.007850, 0.0906 g):										
E-Fuse	9.707	0.896	2.284	0.5451	8.068	100	83.12	22.9	67.3	110.8 ± 0.6
Run 39E9 (J=0.007955, 0.1007 g):										
Fuse	12.24	0.0137	2.280	0.6713	10.23	100	83.55	22.9	3,560	141.1 ± 1.1
Run 40I6 (J=0.007933, 0.2483 g):										
325	13.92	0.0186	1.657	2.917	5.267	4.54	37.86	31.5	2,440	73.9 ± 2.4
400	13.37	0.0143	4.959	1.404	9.188	6.17	68.76	10.5	>5,000	126.9 ± 1.2
475	11.56	0.0122	11.81	0.5025	10.05	16.22	86.98	4.42	>5,000	138.4 ± 0.3
550	11.69	0.0126	3.486	0.0800	11.42	40.83	97.75	15.0	3,560	156.5 ± 0.3
625	11.06	0.0110	4.244	0.0369	10.92	26.06	98.77	12.3	>5,000	149.9 ± 0.3
700	8.590	0.0157	9.392	0.1314	8.178	3.91	95.23	5.56	1,170	113.4 ± 1.1
Fuse	3.184	0.0352	3.332	0.7629	0.904	2.27	58.45	15.7	230	12.9 ± 0.7
Sum	11.39	0.0131	5.442	0.3641	10.29	100	90.33	9.92	3,650	141.5
GSD-1 (sericite 20–40 μm):										
Run 39F8 (J=0.007875, 0.0970 g):										
E-Fuse	13.33	0.0530	7.107	0.7964	10.95	100	82.15	7.35	129	149.2 ± 0.4
Run 39E8 (J=0.007962, 0.0988 g):										
Fuse	10.76	0.0171	0.0796	0.0910	10.47	100	92.27	6.57	883	144.5 ± 0.7

*¹T=temperature. Fuse=sample fusion with normal procedure; E-Fuse= fusion with encapsulated vial technique.
 *²The isotope ratios given are not corrected for Ca, K and Cl derived Ar isotopic interferences but, ³⁷Ar is corrected for decay using a half-life of 35.1 days. The ratios are corrected for line blanks of atmospheric Ar composition. The line blanks are approximately: 1 · 10⁻¹⁴ mol ⁴⁰Ar for T < 1200 °C and 2 · 10⁻¹⁴ mol ⁴⁰Ar for fusion.
 *³F is the ratio of radiogenic ⁴⁰Ar to K-derived ³⁹Ar. It is corrected for atmospheric Ar and interference using the following constants: (⁴⁰Ar/³⁶Ar)_{air} = 295.5; (³⁶Ar/³⁹Ar)_K = 0.0110; (³⁸Ar/³⁷Ar)_{Ca} = 1.40 · 10⁻⁴; (³⁶Ar/³⁸Ar)_{Cl} = 2.02 · 10⁻⁶ per day after irradiation; (³⁹Ar/³⁷Ar)_{Ca} = 6.76 · 10⁻⁴ run Nos. starting 29 and 40, or = 7.88 · 10⁻⁴ run No.'s starting 41; (³⁶Ar/³⁷Ar)_{Ca} = 2.69 · 10⁻⁴ run No.'s starting 39 and 40, or = 2.77 · 10⁻⁴ run No.'s starting 41; (⁴⁰Ar/³⁹Ar)_K = 0.0314 run No.'s starting 39 and 40; = 0.0322 run No.'s starting 41.
 *⁴Relative percent of the total ³⁹Ar released by fraction.
 *⁵Percent of the total ⁴⁰Ar in the fraction that is radiogenic.
 *⁶Weight ratio calculated using the relationship: K/Ca = 0.523 (³⁹Ar_K/³⁷Ar_{Ca}).
 *⁷Weight ratio calculated using the relationship: K/Cl = 5.220 (³⁹Ar_K/³⁸Ar_{Cl}).
 *⁸Ages calculated with a total decay constant of 5.543 · 10⁻¹⁰ a⁻¹. Uncertainties are quoted at the 1σ level. For increments of the step-heating analysis, the uncertainties do not include J-value uncertainty. All other age uncertainties reflect a relative uncertainty of ± 0.25% in J but an overall systematic uncertainty of ± 1% applies to all ages.
 *⁹Sample "popped" during heating and date is a minimum

cedural steps used for a normal $^{40}\text{Ar}/^{39}\text{Ar}$ fusion analysis.

Depending upon the heating routine as well as the nature and amount of encapsulated sample, a vial may fail abruptly and violently (it "pops") during heating at relatively low temperatures. Although this popping occurs well inside the all-metal crucible, there is the potential for loss of sample from the crucible. We have experienced this problem several times particularly during the early stages of procedure development. Loss of a significant fraction of sample can, of course, result in a $^{40}\text{Ar}/^{39}\text{Ar}$ date which is too young *if* both the ^{39}Ar recoil loss is large and the sample loss occurs before the solid grains are degassed. This occurs because radiogenic ^{40}Ar is disproportionately lost with loss of solid, incompletely degassed sample.

3. Results and discussion

The results for normal $^{40}\text{Ar}/^{39}\text{Ar}$ and encapsulated-vial fusion measurements along with incremental-heating results for one sericite (SED-2) which loses large amounts of ^{39}Ar by recoil are given in Table 1. These results are compared with K–Ar ages in Table 2.

For the encapsulated-fusion analyses in the

TABLE 2

Comparison of ages determined by the described $^{40}\text{Ar}/^{39}\text{Ar}$ encapsulation procedure and normal $^{40}\text{Ar}/^{39}\text{Ar}$ and K–Ar procedures

Sample	Age (Ma)	
	K/Ar	$^{40}\text{Ar}/^{39}\text{Ar}$
		normal, total gas
GL-O, glauconite	$95.3 \pm 1.3^{*1}$	132^{*1} 94.5
MON-4, biotite	n.d.	121.7 121.4
MCC-3, sericite	$195 \pm 5^{*2}$	n.d. 198
SED-2, sericite	$116 \pm 2^{*2}$	141 111
GSD-1, sericite	144 ± 2	144 149

n.d. = not determined.

*1 From Foland et al. (1984); *2 from Arhart et al. (1992).

cases where vial "popping" and sample loss occurred, the apparent ages are slightly lower than would be anticipated (see Tables 1 and 2). In one case (sericite sample SED-4), there is no difference. Where popping occurs, the date is suspect and considered a minimum. However, the effect is in no instance more than a few percent.

The popping problem seems to be most acute for larger samples. Presumably this reflects a higher internal capsule pressure which is attained at relatively lower temperatures with these hydrous samples before the sample is completely degassed. It also suggests that small sample sizes (e.g., 10 mg) may be more desirable.

Analysis of biotite sample MON-4 provides an important test of the technique. This sample yields 121.4 ± 0.4 Ma in agreement with its accepted $^{40}\text{Ar}/^{39}\text{Ar}$ age of 121.7 Ma (Foland et al., 1989). Indeed, this biotite was used as the fluence monitor for the irradiation so that agreement is not only expected but required for a workable procedure.

The analysis of the GL-O glauconite inter-laboratory standard (Odin et al., 1982) gives 94.5 ± 1.1 Ma which is in good agreement with its K–Ar date. This result is an especially significant test because it is well established that GL-O suffers ^{39}Ar recoil loss of $\sim 30\%$ and produces erroneous old dates for precisely the same irradiation procedure but without encapsulation (Foland et al., 1984).

The comparison of established Ar date vs. encapsulated $^{40}\text{Ar}/^{39}\text{Ar}$ date (Table 2) demonstrates the reliability of the procedure. Apparent differences of $\sim 4\%$ between K–Ar and encapsulated-vial $^{40}\text{Ar}/^{39}\text{Ar}$ dates are observed in two cases (SED-2 and GSD-1 sericites), but one is older and one younger. These differences themselves are potentially explicable by heterogeneities. In fact, the encapsulation date for MCC-3 sericite is preferable to the K–Ar date because sample heterogeneities compromise the K–Ar analysis.

While not posing deleterious effects on the

determined dates, it is clear that small amounts of Ar are introduced with the procedure. Some atmospheric Ar is introduced and small amounts of ^{37}Ar and ^{38}Ar are also introduced. This is reflected by decreases in apparent K/Ca and K/Cl ratios for encapsulated samples, particularly for samples with low Ca and Cl. For example, the MON-4 biotite has K/Ca and K/Cl of 24 and 500, respectively, which are reduced to ~ 13 and ~ 120 , respectively, for the encapsulated analysis. Undoubtedly, these effects result from release from the vial of ^{37}Ar and ^{38}Ar produced, respectively, from Ca and Cl impurities in silica glass. It seems logical that any bubbles introduced to the encapsulation vial during glass blowing may introduce atmospheric Ar but this effect has not been investigated.

The incremental-heating data for SED-2 sericite ($<2\text{-}\mu\text{m}$ size fraction) are illustrated in Fig. 3 because they have important implications in two regards. First, it is apparent from the results that SED-2 suffers a serious ^{39}Ar recoil loss of almost 20%. The incremental-heating spectrum is clearly a meaningless endeavor. The objective in this context is to stress this point which should be obvious from previous work on glauconites which behave similarly.

A second point addressed by these stepwise-heating data is the potential for revealing Ar

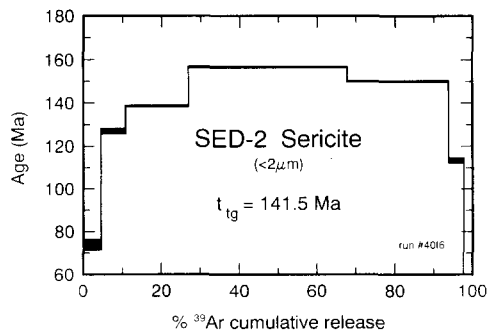


Fig. 3. Standard $^{40}\text{Ar}/^{39}\text{Ar}$ age spectrum for SED-2 sericite. The K-Ar age of this sample is 116 Ma which is much less than the age implied by normal $^{40}\text{Ar}/^{39}\text{Ar}$ procedures. This sample loses $\sim 20\%$ of its ^{39}Ar by recoil and thus the spectrum has no age significance.

gradients which may be present in such grains. Because the recoil loss of ^{39}Ar is substantial, one can infer that ^{39}Ar is not uniformly distributed within individual grains. Specifically, it is expected that the $^{40}\text{Ar}/^{39}\text{Ar}$ ratio is highest at the grain margins and lowest in the interiors reflecting greater ^{39}Ar recoil loss from grain margins. If incremental heating progressively released Ar from grain margins toward the interiors as envisioned conceptually for the $^{40}\text{Ar}/^{39}\text{Ar}$ stepwise-heating technique (Turner, 1968), the $^{40}\text{Ar}/^{39}\text{Ar}$ ratio (and apparent age) should decrease with progressive Ar release. Such a pattern is clearly not observed for this sericite. Nor probably should such ideal behavior for any hydrous minerals be expected because they have conclusively been demonstrated to undergo morphological and mineralogical changes and dehydration during vacuum heating (e.g., Giletti, 1974; Gaber et al., 1988).

The variations in the apparent age for stepwise-heating SED-2 must reflect $^{40}\text{Ar}/^{39}\text{Ar}$ heterogeneities. These are most logically due to ^{39}Ar redistribution, for example from high- to low-K regions or grains, during irradiation. The spectrum is simply a consequence of how and when various of these regions decompose. In short, the results further demonstrate that normal $^{40}\text{Ar}/^{39}\text{Ar}$ procedures for fine-grained minerals can produce misleading and incorrect ages.

In summary, the results demonstrate a reliable approach to Ar dating of small, fine-grained specimens which are intractable with normal procedures. Possible errors from sample loss by popping should be monitored to ensure a reliable integration of all Ar. The encapsulated vial approach has considerable potential for very fine-grained or other material where recoil or other Ar loss is a problem, such as authigenic minerals or tiny samples which may be dictated by low abundance in a sidewall core. While other approaches or $^{40}\text{Ar}/^{39}\text{Ar}$ adaptations are possible, one of which is described by Hess and Lippolt (1986), the one

outlined here is simple and requires only equipment available in most Ar laboratories. In addition, the procedure should be adaptable to laser fusion techniques.

Acknowledgements

The authors thank colleague A. Heimann for suggestions on an early version of the paper and G.S. Odin for very helpful review comments. This work was supported in part by a Geological Society of America Research Grant to G.B.A.

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