

FUSED BEAD ANALYSIS IN DIOGENITE METEORITES. A. W. Beck¹, H. Y. McSween Jr.¹, D. W. Mittlefehldt² and C.-T. A. Lee³, ¹Dept. of Earth and Planetary Sciences, University of Tennessee, Knoxville, TN (abeck3@utk.edu), ²Astromaterials Research Office, NASA Johnson Space Center, Houston, TX, ³Dept. of Earth Science, Rice University, Houston, TX

Introduction: Bulk rock chemistry is an essential dataset in meteoritics and planetary science [1]. A common method used to obtain the bulk chemistry of meteorites is ICP-MS. While the accuracy, precision and low detection limits of this process are advantageous [2], the sample size used for analysis (~70 mg) can be a problem in a field where small and finite samples are the norm.

Fused bead analysis is another bulk rock analytical technique that has been used in meteoritics [3]. This technique involves forming a glass bead from ~10 mg of sample and measuring its chemistry using a defocused beam on a microprobe. Though the ICP-MS has lower detection limits than the microprobe, the fused bead method destroys a much smaller sample of the meteorite. Fused bead analysis was initially designed for samples with near-eutectic compositions and low viscosities. Melts generated of this type homogenize at relatively low temperatures and produce primary melts near the sample's bulk composition [3]. The application of fused bead analysis to samples with non-eutectic melt compositions has not been validated. The purpose of this study is to test if fused bead analysis can accurately determine the bulk rock chemistry of non-eutectic melt composition meteorites. To determine this, we conduct two examinations of the fused bead. First, we compare ICP-MS and fused bead results of the same samples using statistical analysis. Secondly, we inspect the beads for the presence of crystals and chemical heterogeneity. The presence of either of these would indicate incomplete melting and quenching of the bead.

Methods: We used ten diogenite meteorites as a means to evaluate fused bead analysis with non-eutectic melt compositions. Diogenites are Mg-rich orthopyroxenites from the asteroid 4 Vesta [4]. Sample splits of these meteorites were first inspected, then ground and thoroughly mixed. 50-80 mg and 10 mg aliquots of each sample were separated for ICP-MS and fused bead analysis, respectively.

ICP-MS analyses were conducted at Rice University using procedures described by [5]. Fused beads were measured using a CAMECA SX-100 electron microprobe at the University of Tennessee. Approximately forty 10 μm defocused beam analyses were taken per bead. These results were then tested for nor-

mality and averaged into mean elemental concentrations for each sample.

We compared concentrations of Ti, Al, Ca, Mn, and Fe between the two methods using Student's t-test [6]. Si was not compared because Si is volatilized in solution ICP-MS. Mg was not compared due to variable ICP-MS results. If a given element in a particular sample passes the test, this means that the two methods measured the statistically same concentration. In assuming that ICP-MS is correct we can deduce that fused bead analysis is accurate for a given element if the t-test is passed.

Finally, in an effort to detect if the glass beads are crystal-free and to measure any chemical heterogeneities, we made cross sections of three beads. These cross sections were remounted and examined for heterogeneity (i.e. crystal formation) using the electron microprobe.

Results and Discussion: Minor element concentrations had a high t-test success rate, with the exception of relatively low Al values. Note that Al concentrations were compared in only seven of the ten samples, due to non-normally distributed data (Fig 1). Fe, the one major element compared using methods, had a poor t-test success rate.

Sample	Ti	Al	Ca	Mn	Fe
ALH85015	✓	✓	✓	✓	✓
ALHA77256	✓	✗	✓	✓	✗
EET83246	✓	✗	✓	✓	✗
LEW88008	✓	✓	✓	✓	✗
LAP03979	✓	✓	✓	✓	✗
GRA98018	✓	✓	✓	✓	✓
LEW88679	✓	✓	✓	✓	✓
MET01084	✓	✗	✓	✓	✗
MIL03368	✓	✓	✓	✗	✓
PCA02008	✓	✓	✓	✓	✓
Success Rate	100%	57%	100%	90%	50%

Fig. 1. Success of the student's t-test in the ten samples. Gray boxes represent data that did not pass the test for normality and have been omitted.

We plotted the concentrations measured with both methods on a bivariate plot in an effort to detect any systematic variation. These results echo the t-test in showing a very strong 1:1 relationship in the minor element concentrations (Fig 2A). A plot of Fe concentrations reveals a trend much different than the ex-

pected 1:1 correlation between fused bead and ICP-MS (Fig 2B). These results suggest that in diogenite meteorites, fused bead analysis can be used to accurately measure Ca, Mn and Ti. However, Fe (and presumably other major elements) were not accurately measured using this analytical method. Reasons into why this may be the case are explored below.

Bead cross-sections were made in an attempt to detect heterogeneities in the bead composition and to look for quench crystals in the glass. The three bead cross-sections that were examined revealed a strong heterogeneity in Fe distribution within the bead, shown as light green in Fig 3. Within any give bead, the average range of FeO was 16.1-17.4 wt.%. Abundant, Mg-rich quench crystals were also found (seen in Fig 3 as blue, circular shaped objects). No significant heterogeneities in minor element abundances were detected.

The uneven distribution of Fe found in the bead cross-sections is the likely cause for the failure of the Fe t-tests. This, along with the presence of Mg-rich crystals, indicates that fused bead analysis cannot be used to accurately determine major element abundances (i.e. Mg and Si) in diogenite meteorites. It is likely that these problems would also arise if this method was applied to other, non-eutectic melt samples.

Conclusions: Fused bead analysis did not accurately determine bulk Fe concentrations in diogenite meteorites. This failure is the result of incomplete Fe homogenization in the glass. This, coupled with the presence of Mg-rich quench crystals, suggests that fused beads would produce inaccurate results for other major elements. The occurrence of these crystals and variation in Fe also implies that minor element distribution may be heterogeneous, though the sensitivity of the microprobe does not detect this. Even though most minor elements passed the t-tests, we are skeptical that fused bead analysis can be used to determine accurate bulk minor element concentrations in diogenites.

References: [1] McSween H. Y. *et al.* (2003) *JGR*, **108**, 5135. [2] Thomas R. J. (2008) *A Practical Guide to ICP-MS*, CRC Publishing. [3] Brown R. W. (1977) *Geochim. Cosmochim. Acta*, **41**, 435-438. [4] Mittlefehldt D. W. *et al.* (1998) *Planetary Materials, RiM*, **36**, Ch. 4. [5] Lee C.-T. A. *et al.* (2007) *Geochim. Cosmochim. Acta.*, **71**, 481-496. [6] Harris D. C. (2007) *Quantitative Chemical Analysis*, W. H. Freeman & Co.

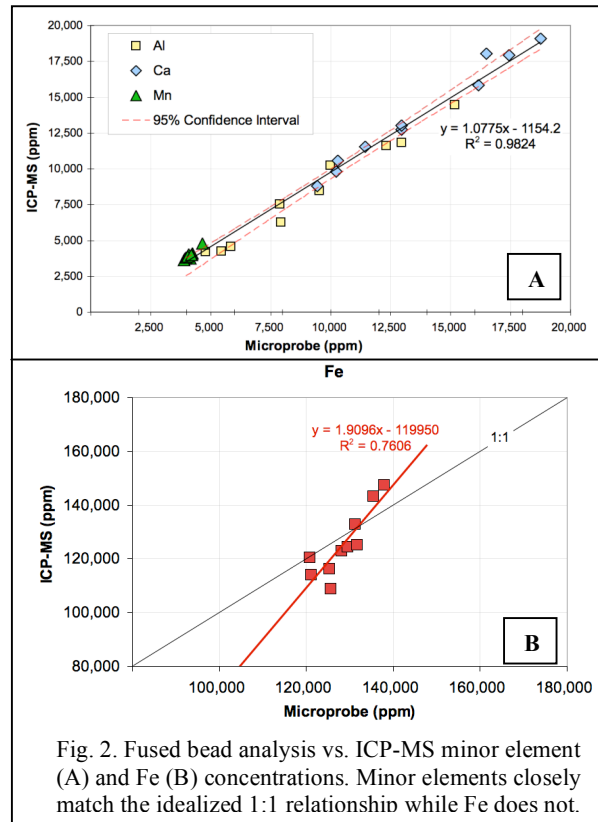


Fig. 2. Fused bead analysis vs. ICP-MS minor element (A) and Fe (B) concentrations. Minor elements closely match the idealized 1:1 relationship while Fe does not.

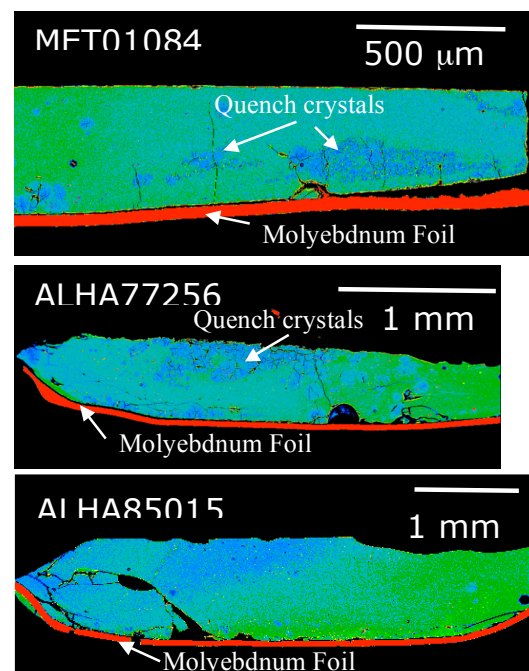


Fig. 3. Backscatter electron images of three bead cross-sections showing a heterogeneous distribution of Fe (light green) and the presence of quench crystals.