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EXPERIMENTAL SEGREGATION OF IRON-NICKEL METAL, IRON-SULFIDE, AND OLIVINE IN A THERMAL GRADIENT: PRELIMINARY RESULTS. Stephen R. Jurewicz and J.H. Jones, Mail Code SN4, NASA Johnson Space Center, Houston, Texas 77058

INTRODUCTION: Speculation about the possible mechanisms for core formation in small asteroids raises more questions than answers. Petrologic evidence from iron meteorites, pallasites, and astronomical observations of M asteroids suggests that many small bodies were capable of core formation. Recent work by Taylor [1] reviews the geochemical evidence and examines the possible physical/mechanical constraints on segregation processes. Taylor's evaluation suggests that extensive silicate partial melting (preferably 50 vol.% or greater) is required before metal can segregate from the surrounding silicate and form a metal core. The arguments for large degrees of silicate partial melting are two-fold: 1) elemental trends in iron meteorites require that the metal was at its liquidus, and 2) experimental observations of metal/sulfide inclusions in partially molten silicate meteorites show that the metal/sulfide tends to form spherules in the liquid silicate due to surface tension effects (Takahashi [2], Walker and Agee [3], Dickinson and Lofgren [4]). Taylor points out that for these metal spherules to sink through a silicate mush, high degrees of silicate partial melting are required to lower the silicate yield strength.

Although some qualitative experimental data exists, little is actually known about the behavior of metals and liquid sulfides dispersed in silicate systems. In addition, we have been impressed with the ability of cumulative olivine to expel trapped liquid when placed in a thermal gradient (Walker et al. [5]). Consequently, we undertook to: 1) experimentally evaluate the potential for metal/sulfide/silicate segregation in a thermal gradient; and 2) obtain quantitative data of the wetting parameters of metal-sulfide melts among silicate grains.

EXPERIMENTAL: San Carlos olivine was selected as the silicate starting material because of its abundance and because olivine is the dominant silicate mineral in planetary mantles. Iron and nickel metal powders were used for the metal fraction and powdered pyrite was used for the sulfide. The starting composition was selected to be 60 wt.% olivine (76.1 vol.%), 25 wt.% Fe (13.3 vol.%), 5 wt.% Ni (2.3 vol.%), and 10 wt% FeS₂ (8.3 vol.%). Thus, the total volume fraction of metal + sulfide in the system was 24%. Both olivine and pyrite were ground to fine powders before being blended in an agate mortar with the iron and nickel powders until uniform.

The starting mix was loaded into a fired pyrophyllite capsule with a 10mm long by 2mm diameter sample chamber. The capsule was then loaded into a piston cylinder assembly such that the top of the capsule was next to the control thermocouple. Based on thermal gradient measurements of the piston cylinder assembly employed, we estimate the bottom of the charge to be about 100-150 degrees colder than the thermocouple reading. The assembly was loaded into the piston-cylinder apparatus and run at 10kb pressure and 1200°C for 8 days. Runs were quenched to near room temperature within 10 seconds. After quenching, the runs were sectioned parallel to the long axis and polished for microstructural analysis. Chemical compositions were obtained using standard WDS analysis techniques on the CAMECA microprobe at JSC.

RESULTS: Figure 1 is a reflected-light photograph of the 8 day run. Figure 1 only shows the bottom 4mm of the charge. The total length of the sample after the run is 6-7mm. The abrupt change in reflectivity in the center of the photograph marks the boundary between two distinct regions of the charge. The higher reflectivity lower region is composed of Fe-Ni-metal and olivine and the upper darker region is composed of Fe-Ni sulfide and olivine. The segregation of sulfide and metal into the two regions is complete. The bright band at the bottom of the charge is Fe-Ni-metal.

Microstructural examination shows that the uppermost portion of the sulfide-olivine region appears to have approached textural equilibrium (as shown in the SEM photograph in Fig. 2), and contains about 10 vol.% sulfide by visual estimate. The volume fraction of sulfide increases down the thermal gradient and near the sulfide/metal interface the volume fraction of sulfide is estimated at 15-20%. On the olivine/metal side of the boundary, the Fe-Ni-metal fraction is estimated at 30-40 vol.%. The volume fraction of metal in the rest of olivine/metal region varies between 30-50 vol.%. Thus, the metal + sulfide component at the hot end of the charge has decreased, whereas the metal fraction at the cold end has increased.

The equilibrated-looking texture of the uppermost olivine/sulfide region permitted the measurement of wetting angles between the sulfide (liquid at run temperature) and the olivine. One hundred angles were measured and processed as described in Jurewicz and Jurewicz [6]. The olivine/sulfide liquid wetting angle was

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determined to be 92°. This wetting angle value implies that a critical melt fraction must be exceeded to establish interconnectivity.

DISCUSSION: Our experiments demonstrate essentially complete metal/sulfide segregation in a thermal gradient and the beginnings of olivine/metal-sulfide segregation in a period of 8 days at relatively moderate temperature. This segregation occurred with 0% silicate partial melting and < 40% (by weight) melting of the metal+sulfide assemblage. Consequently, the total volume fraction of melting within the system is constrained to be $< 13 \pm 3\%$. It is especially interesting to note that sulfide did not simply melt and migrate to the top of the charge, but all the solid Fe-Ni metal moved to the bottom. Obviously, this is a complicated experiment and the exact segregation mechanisms and their respective contributions are debatable. Gravitational segregation is difficult to invoke because only about 13 vol.% of the sample is liquid at run conditions. However, the observation that the Fe-metal moves to the cold end of the charge is consistent with the results of Jones and Walker [7] for thermal diffusion of Fe in metal-sulfide systems. Given that diffusivities in liquid metals are generally on the order of 1×10^{-5} cm²/sec [8], thermal diffusion cannot be ruled out. On the time scale of our experiments, the diffusive scale length is approximately 2.5 cm, considerably longer than the experimental charge. Our tentative conclusion is that solid metal dissolved at the hot end and precipitated at the cold end. Given the inevitability of boundary layers and thermal gradients in natural systems, we believe that this mechanism should be considered in discussions of core formation.

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