FE-PT ALLOY CAPSULES FOR HIGH-PRESSURE EXPERIMENTS. Hwai-Kuo Chen, David J. Andersen, and Donald H. Lindsley, Department of Earth and Space Sciences, S.U.N.Y., Stony Brook, NY 11794.

Petrologists have long been concerned with choosing containers for phase-equilibrium experiments on iron-bearing samples. Pure iron can be a suitable capsule material (e.g., 1; 2) if it is kept isolated from free oxygen and if the sample being studied may appropriately be in equilibrium with metallic Fe; that is, \( a_{Fe} = 1 \). Molybdenum containers may be suitably inert if the oxygen fugacity is high enough (that is, \( a_{Fe} \) is low enough) to minimize alloying (3). Fe-Pt alloys have been advocated (e.g., 1) for samples that were not saturated with Fe (\( a_{Fe} < 1 \)), because each sample has an intrinsic \( a_{Fe} \) that can be matched by that of a suitably chosen Fe-Pt alloy. Several techniques are used to produce suitable alloys (e.g., 4; 5; 6; 7). Most are limited for experiments at high pressures: Fe-Pt alloys are brittle and tend to fail at some stage of the experiment. A suitable container requires a pure Pt or Fe outer portion for mechanical integrity and an alloyed inner surface for compatibility with the sample. We describe two techniques to produce such containers.

Fe-rich capsules. Flat-bottomed capsules are machined from 99.999% pure Fe rods: 2.39 mm O.D. x 3.56 mm long with a concentric bore 1.70 mm I.D. by 3.05 mm deep. Lids 1.70 mm in diameter and 0.51 mm thick are made from the same stock. For each capsule we cut two disks 1.70 mm across and a rectangle 2.54 mm x 5.33 mm from 0.025 mm Pt foil. One disk is placed at the bottom of the Fe capsule; the rectangular foil is inserted into the capsule in close contact with the inner surface. This assembly is sealed in an evacuated silica-glass tube for annealing. The remaining Pt disk, in contact with one surface of the Fe lid, is sealed in a separate evacuated silica-glass tube. All the materials were then annealed for 7–8 hours at 1300°C. Fig. 1a shows the distribution of Fe and Pt near the inner surfaces of the capsule and lid after annealing. The high iron contents at the inner surfaces are attributed to vapor transport of Fe during annealing. Fig. 1b shows the distribution of Fe and Pt in a similar capsule after a sample with 19 wt.% FeO was run for 1.3 hr at 1342°C and 9.3 kbar. The Fe-rich "spike" at the inner surface has disappeared, and the alloy in contact with the sample is mainly 55 to 60 wt. % Fe. This composition corresponds to \( a_{Fe} = 0.64 \) to 0.71 (8). The total iron content of the sample, expressed as FeO, was virtually unchanged.

Fe-poor capsules. We make Pt capsules from 2.39 mm O.D. tubing: the tubes are cut approximately 2.5 mm longer than the desired final length. One end is crimped and welded or simply folded with opposite walls overlapping; in either case the closure is flattened in a pellet press. This procedure is repeated for the other end after the sample is loaded. Neither method was successful for Fe-plated platinum capsules: either the capsule walls cracked during crimping or folding, or the Fe-rich lining spalled off, exposing the sample to pure Pt. Thus, we lined the inside of a pure Pt capsule with Fe-Pt alloy prepared as follows: Approximately 0.030 mm of Fe was plated onto both sides of a piece of 0.025 mm foil and onto the inside surface of Pt tubing 1.98 mm O.D. x 1.60 mm I.D. x 2.80 mm long. We followed the methods of (9) for plating. The electrode was a piece of Fe wire placed concentrically within the sleeve. Formation of hydrogen bubbles within the sleeve prevented even plating; this problem was overcome by slowly flowing the electrolyte solution through the sleeve. The plated foil and sleeve were sealed in evacuated silica-glass capsules and annealed for several hours at 1300°C to form Fe-Pt alloys. Disks 0.078" (1.98 mm) diameter were punched from the alloyed foil to serve as liners for each end of the capsule. Fig. 2 shows the distribution of Fe in such a capsule after it was run for 1.5 hr at 1459°C and...
FE-PT ALLOY CAPSULES

Chen H.-K. et al.

13.9 kbar with a sample containing 19 wt. % FeO. The Fe "spike" in the capsule wall at 0.2 mm results from vapor transport of Fe from the inner to the outer surface of the sleeve during annealing; some of that Fe then diffused into the outer Pt capsule during the actual experiment. The composition of the capsule (ca. 12 wt % Fe) where it contacts the sample corresponds to an iron activity of approximately 0.03. Curiously, the apparent FeO content of the sample (glass) run in this capsule is only slightly lower than those run in pure Fe and in Fe-rich capsules despite their much higher Fe values. A possible explanation is that a considerable portion of the iron in the Fe-poor capsule is ferric and that the true content of FeO is much less than that indicated by the electron microprobe analysis, which expresses total Fe as FeO. We hope to test this idea by Mossbauer spectroscopy of the glasses. If this explanation is correct, the source of the excess oxygen is unclear; one possibility is oxygen-rich inclusions trapped during the electroplating process.

Conclusions. It is possible to make capsules that can survive piston-cylinder experiments up to at least 20 kbar and which can have a variety of Fe-Pt alloys in contact with the silicate sample. The possibility that oxidizing material may be trapped during electroplating must be further investigated before this method is adopted for runs in closed systems.