Milk cartons and ash cans: two unconventional welding techniques

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Abstract

Two previously unpublished methods of sealing experimental materials in noble metal containers for high P-T investigations provide advantages over conventional techniques. The “milk carton” closure allows the use of maximum diameter capsules in small reaction vessels and involves only a single straight-line weld. The “ash can” closure accommodates cylindrical samples with little void space in the container, thereby minimizing sample deformation at pressure. Both are also suitable for use in experiments involving very large amounts of volatiles.

Introduction

Noble metal tubing is still the material of general choice for fabrication of sample containers in high pressure/high temperature experimentation. In many applications, the shape and/or volume of the sealed tube (capsule) is of secondary importance, so no special attention to the method of closure is required. There are, however, two general circumstances under which capsule shape can be important: (1) when maximum volume for a given reaction vessel diameter is required; and (2) when preservation of sample geometry in a solid-media, piston-cylinder assembly is critical to the success of the experiment.

We have investigated a variety of crimping and welding techniques and have developed two methods that we find very useful. Although we are not aware of any precedents, it is likely that other workers have used these or similar methods, and we make no pretense of being the first or sole discoverers.

The milk carton

The main advantage of this technique is that there are no bulges in the capsule wall (due to the closing crimp) that extend beyond the outside diameter of the metal tubing. For this reason, the maximum diameter capsule can be used in small vessels such as cold-seal bombs or piston-cylinder assemblies. An additional advantage over the 3-corner crimp sometimes used for similar purposes is that the weld is a single straight line and involves a relatively large amount of metal.

For best results, the tubing should be annealed by heating to an orange glow in a burner flame prior to capsule fabrication. This makes the metal more pliable and less likely to split during crimping. The first crimp is made by squeezing together two opposite points on the end of the capsule using roundnose pliers. About 2–3 mm of the tube length should be closed so that the end of the capsule looks somewhat like a figure eight (Fig. 1). Flat nosed pliers are then used to close the tube from opposite points perpendicular to the original crimp direction. It is advisable at this point to file the top of the crimp to expose the entire folded circumference of the tube before welding. This ensures that a proper weld will close all possible openings. Loss of sample or volatiles during welding can be determined by weighing the capsule after filing and again after welding. The capsule is then sealed in the carbon arc.

If the capsule contains volatiles, or if the weld must be made very close to the enclosed sample, it is advantageous to cool most of the capsule during welding. In most laboratories, this is accomplished by partial immersion in H2O, liquid N2 or dry ice. We find that liquid H2O is usually an adequate coolant, especially if the capsule is approximately 80% immersed in the water by suspension from a copper wire cradle attached to the welder vise. As with conventional sealing techniques, further protection against volatile loss can be obtained by lengthening the crimp, thus moving the heated part of the capsule away from the sample.

The ash can

This technique is an outgrowth of the solid-plug sealing method described by Watson (1979), but has the advantage of being less expensive because it requires far less precious metal. The weld was developed primarily for tracer studies of diffusion in melts, for which a cylindrical sample with a tight-fitting container is often required. It also proves useful, however, when large amounts of volatiles must be sealed in a cylindrical capsule (e.g., to fit tightly in a piston-cylinder assembly).

The ash can involves no crimping, but does require
prefabrication of capsule end pieces, which are actually shallow cups whose outside diameter is the same as the inside diameter of the capsule tubing. We make these pieces by pressing annealed disks (punched from metal sheet) through a hole of appropriate diameter in a brass plate (Fig. 2). This procedure turns up the edges of the disks to form the cup-like end pieces, which are inserted concave-outward in a length of tubing to form a capsule (Fig. 3). A weld around each rim seals the container.

We have successfully used this closing technique to encapsulate glass cylinders with very little void space within the container (this promotes preservation of glass sample geometry in the high-pressure apparatus), and also to prepare sealed containers virtually full of liquid H₂O. Loss of contained water during the final weld in this latter application can be minimized by partially filling the end cup with cooling water.

The two capsule closing methods described above have worked well with a variety of reaction vessels and sample materials, including volatiles. Although these methods are not foolproof, reasonable care in their execution nearly eliminates failed runs due to capsule leakage and/or sample loss during welding.

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References


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