

Simple cold-weld sealing of noble-metal tubes

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Abstract

A pinch-off device, normally used for pinching off vacuum exhaust tubing of copper in refrigeration systems, is a convenient tool for sealing noble-metal tubing for pressure-temperature (including hydrothermal) experimentation. The principal advantages of this sealing technique are that it is readily mastered, extremely easy, fast, inexpensive, and reliable to use. It can also be used for sealing tubing such as Al and Cu, which are not so readily welded. The disadvantages are that the seals are somewhat less robust than welds, and more tubing is required for a given charge when fluid expansion is significant during the run or a gas phase remains at the end of the run.

Sealing noble-metal tubes using an arc welder or gas torch for pressure-temperature experiments is an art which is not readily mastered. The cold-weld sealing technique by a pinch-off device gives reliable seals for users with no prior experience and requires no special technique or art. The pinch-off device used is model No. POD-375, which can be obtained from CHA Industries, 1215 Chrysler Drive, Menlo Park, California 94025. This device is normally used for pinching off vacuum exhaust tubing (0 to 3/8" OD \times 0.049 wall) of fully annealed OFHC copper. The jaws of this pinch-off device are hardened, ground, high-speed tool steel (Rockwell C60 to 65). This device requires only minimal maintenance. Periodic cleaning and lubrication with a spray-on lubricant such as WD 40 will ensure long life. Extensive use of the pinch-off device, however, may necessitate jaw replacement.

The initial preparation of gold tubing follows conventional procedure which includes cutting the tubing to length, cleaning, annealing at 500 to 700°C for about 2 hours, and marking identification numbers with a scribe. Sealing of the tubes is accomplished by a pinch-off device instead of arc welding. One end of the tube is crimped by holding the capsule between the jaws of a pinch-off device as shown in Figure 1. Crimping or pinching the tube results in a cold-weld sealing of this end. It is necessary to have clean, smooth and parallel jaws and clean tubes to produce a hermetically perfect cold weld of tube walls.

When the device is new, it will cut cleanly through the gold tubing. As it wears, a thin web of metal is left which is readily broken by flexing several times. Sealing tubes with the pinch-off device produces ends which are clean and thin. The crimp-sealed tubes must be handled with care to avoid disrupting the seal. The capsules can now be loaded with the solid sample by a funnel, the liquid by a syringe. The open end of the tube must be cleaned before sealing. Rolled Kimwipes or pipecleaners are well suited for this. The top of the loaded gold tube is then crimped or pinched off with the pinch-off device.

Since this device does not use heat, it can be used without further precautions for sealing capsules containing water or other compounds which evaporate or decompose at elevated temperatures. This is one of the main advantages of this technique compared to the arc welding technique. It also offers the possibility of sealing samples at cryogenic temperatures. Likewise, tubing not normally readily welded (Al, Ni, and Cu, for example) can be sealed. This allows the use of these metals as less expensive alternatives to noble-metal capsules, or to eliminate the reaction between the capsule and its contents (*e.g.* use copper tubing for the Cu-CuO oxygen buffer, thus avoiding the alloying of the Cu with the Ag or Au capsule).

The cold-weld sealing technique of gold capsules described here was used with great success on hundreds of gold capsules (with 5 mm ID and 0.005 wall thickness) heated at several temperatures (mostly at

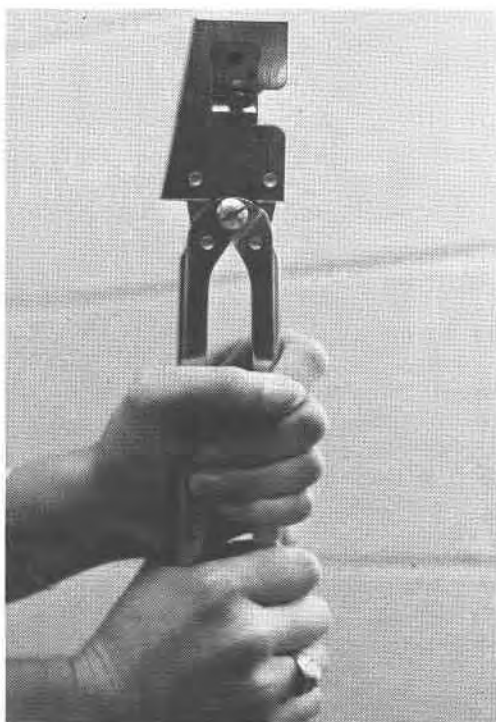


Fig. 1. Crimping of gold tube between the jaws of a pinch-off device.

100, 200, and 300°C and some at 400 to 700°C) and pressures (300 to 1000 bars) in small conventional cold-seal vessels as well as large modified Bridgeman seal autoclaves. However, our experience has been

that the seals produced by the pinch-off device are less robust (a failure rate of about 3 percent) than those obtained by welding (a failure rate of about 1 percent). As a test of this observation, six capsules were partly filled with water; three were sealed with the pinch-off device, and three with a H_2-O_2 torch. The capsules were then run on a thermogravimetric analyzer. The "pinched" capsules began to leak at 125 to 145°C and had expanded slightly in response to the internal pressure of 2 to 3 atm. The welded capsules failed at 190 to 225°C or 12 to 24 atm. In fact the best welds held and the tubing failed catastrophically. Since the seal produced by a pinch-off device is less robust, it is necessary to provide a larger volume if gases are released during the treatment. For example, if CO_2 is evolved during the experiment, the capsule sealed by a pinch-off device would have to have about four times the free volume of a capsule sealed by H_2-O_2 torch to remain sealed when the external pressure is removed. Thus, the cold-weld technique using a pinch-off device requires somewhat longer tubing if gases are released.

In summary, the pinch-off device is an extremely simple and rapid means for sealing noble-metal tubes reliably for pressure-temperature experiments. There is also the possibility of using a wider range of tubing materials for pressure-temperature work.

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