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A simple and effective capsule sealing technique for hydrothermal experiments

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Abstract

Capsule sealing has always been a key procedure in hydrothermal experiments to explore the composition and properties of geo-fluids and their influence on various geological processes. Previously reported capsule sealing techniques have primarily focused on either weld-sealing or cold-sealing methods, which have some disadvantages and limitations. Here, we report on a newly developed, simple, and effective capsule sealing technique incorporating operations from the cold-sealing and weld-sealing techniques. The technique includes three steps: first, preparing inner and outer tubes, both with a flat bottom at one end; subsequently, reverse-buckling the tubes to form a preliminary seal; and finally, welding shut the tiny slit at one end of the tubes. The new capsule sealing technique was tested in experiments for fluid inclusion synthesis. Fluid inclusions were successfully synthesized in 10 runs over a range of conditions (800~900 °C, 1~1.5 GPa). Considering the insignificant mass changes recorded and the occurrence of free fluid from the recovered capsules, the new capsule sealing technique was proven to be reliable. The simple and effective capsule sealing technique has the following advantages over the previous techniques. First, the capsule sealing technique is simple, effective, and easy to operate. The technique does not require a capsule body and lid with a complex structure, nor does it require dies or special tools. The critical weld-sealing operation is easier to complete due to the narrow and uniform slit surrounded by more metal, during which loss of volatilization is prevented by the preliminary seal. Second, the capsules can be sealed with uniform thickness and regular shape, prechecked for leakage in an oven, and annealed under high temperature and high pressure with less deformation, which could improve the success rate of experiments. Third, the theoretically required capsule materials can be changed (such as precious metals, alloys, etc.), as can the dimensions required to construct a capsule with the desired size and wall thickness (large volume or thick wall). Thus, sealed capsules are suitable not only for piston cylinders but also for multi-anvil presses and other gas-media or hydrothermal-media apparatus, such as autoclaves and pressure vessels, which means a wider range of temperatures and pressures are accessible and thus more fields of application.

Keywords: Capsule sealing, cold-sealing, weld-sealing, hydrothermal experiments, fluid inclusion synthesis

Introduction

Widely existing fluids in the crust and upper mantle have been implicated in many geological processes at different scales involving material cycles and energy transfer, such as slab subduction, magmatic emplacement, volcanic eruptions, seismic activities, mineralization, and so forth (Hack and Mavrogenes 2006a). Determining the composition and properties of geo-fluids and their influence on various geological processes has always been the goal of geologists. To understand geological fluids, hydrothermal experiments are an important means. One key difficulty in hydrothermal experiments is how to seal liquid samples in capsules and maintain a good seal under high-temperature and high-pressure conditions related to real geological processes. Several capsule weld-sealing and cold-sealing techniques have been invented and successfully applied in hydrothermal experiments (Arndt and Rombach 1976; Audétat and Bali 2010; Ayers et al. 1992; Becker et al. 1983; Brodholt and Wood 1994; Cemič et al. 1990; Hack and Mavrogenes 2006a; Lerchbaumer and Audétat 2012; Manning and Boettcher 1994; Sneeringer and Watson 1985).

In weld-sealing techniques, a capsule charged with fluid and other starting materials are squeezed a few millimeters below its upper end (e.g., a three-corner crimp or milk carton-like fold) between thin jaws of a vice or an ashtray-like lid is inserted concave-outward in the open end and then welded shut under cooling by cold water, dry ice or liquid nitrogen (Brodholt and Wood 1994; Manning and Boettcher 1994; Sneeringer and Watson 1985). In general, the resulting capsules have a high length-to-width ratio and an asymmetrical geometry, which is disadvantageous for piston-cylinder or multi-anvil experiments in which the capsules should be as short and compact as possible to reduce temperature gradients. Furthermore, it is worth mentioning that in addition to

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fluid volatilization loss, welding the capsules shut is relatively difficult due to the thin rims, local large gap and certain capsule materials (such as copper, silver, etc. at least in our laboratory).

The cold-sealing technique was first reported by Arndt and Rombach (1976) and subsequently modified by Becker et al. (1983) and Cemič et al. (1990). In the technique, a matching lid was cast directly by cold pressurization onto a charged cylindrical thick-walled capsule body machined out of a solid metal rod. To reduce the high cost of preparing thick-walled capsules from noble metal rods, Ayers et al. (1992) developed a technique in which thin noble metal sleeves were hammered into thick-walled (typically 0.5 mm wall thickness) cylinders machined from transition metals. Large volume capsules (23~32 mm in length, 9~15 mm in diameter), which consist of a capsule body and lid with special structures, have been used by Hack and Mavrogenes (2006) and Spandler et al. (2007, 2014). Though the cold-sealing methods mentioned above well avoid potential problems associated with the weld-sealing technique (fluid volatilization loss, difficulties in operation, and arc welder costliness), such cold-sealing methods require the aid of compression from a piston-cylinder apparatus to accomplish complete sealing. Consequently, on the one hand, the cold-sealing capsules cannot be prechecked for leakage to prevent possible failures in follow-up experiments, and on the other hand, when the cold-sealing capsules are pressurized on an apparatus from more than one direction, such as multi-anvil press, the reliability of sealing may be greatly reduced due to the complicated differential stress. Combining the advantages of weld-sealing and cold-sealing, Audétat and Bali (2010) established a method of sealing volatile-rich samples into platinum capsules using a minimum amount of metal. However, the technique is complex, needs to be performed with the help of special dies, and is only suitable for soft noble metals such as platinum or $Pt_{95}Rh_{05}$ alloy.

Here, we describe a simple and effective method in which fluids can be perfectly sealed in copper (or silver, nickel, iron, gold, platinum, alloy, etc.) capsules without fluid loss. The resulting capsules have a regular shape (cylindrical) and uniform thickness and thus fit easily into high-pressure assemblies of piston cylinders and multi-anvils. The sealed capsule could also be used in conventional hydrothermal- and gas-media apparatuses. In contrast to some of the techniques mentioned above, the technique is simple to operate and effective with a high success rate.

Methods

Figure 1 illustrates the simple and effective capsule sealing technique. The technique includes three steps. First, capsule preparation. The capsule is mainly composed of two cylindrical tubes (indicated in Figs. 1a and 1b, inner and outer tubes with a flat bottom at one end), which can be machined from a solid metal rod or fashioned by a punch process. The former approach is suitable for base metals or transition metals that are relatively cheap and hard, such as copper, nickel, iron, and so forth, while the latter is preferred for noble metals owing to their softness and expense. The punch process requires a set of special dies, consisting mainly of a base with two round holes and two matching levers. Figure 2 roughly shows the special dies for fabricating gold capsule with an outer diameter (O.D.) in 5.0 mm, inner diameter (I.D.) in 4.6 mm. A cylindrical gold slug (5.0 mm O.D., 3.0 mm height) is placed into the round hole (5.00 mm diameter) pre-coated with lubricating oil on the base, and the matching pressure lever (4.80 mm diameter) fixed to a manual hydraulic press is laid on the gold slug with the central axes of both aligned. With the manual hydraulic press, the lever is gradually pressed into the gold slug and a gold outer tube with the desired size and shape similar to that in Figure 1a can be produced after final shaping. The gold inner tube can also be fabricated with the same method. The detailed dimensions of the inner and outer tubes, which can also be scaled down to adapt to different assemblies, are shown in Figure 1a and 1b, respectively. The inner tubes (e.g., 11.0 mm O.D., 10.0 mm I.D., 16.0 mm length) and outer tubes (e.g., 12.0 mm O.D., 11.0 mm I.D., 17.0 mm length) both have a flat bottom at one end (1.0 mm in thickness). Machining accuracy is preferably controlled within 0.02 mm. In testing experiments, the tubes for copper capsules were processed by ourselves on the lathe in our lab and those for silver capsules were customized in bulk with the punch process. After the tubes are properly prepared, starting materials with a certain amount of fluid or only solids rich in volatiles are successively charged into the inner tube as fully and tightly as possible. Then the outer tube is capped on the inner tube and subsequently pressed down with a hammer or hydraulic press until the inside of the bottom of the outer tube is in tight contact with the open end of the inner tube (illustrated in Fig. 1c). By virtue of a reverse-buckling operation, a preliminary seal between the fluid-bearing inner tube and outer tube is produced (indicated in Fig. 1f), which can prevent loss of fluid via volatilization during the following weld-sealing. Third, the tiny slit between the outer and the inner tubes is shut at one end by standard arc welding (indicated in Figure 1d). The circular slit is generally very narrow (<0.1 mm in width) and is surrounded by a large mass metal (indicated in Fig. 1e), which makes the weld-sealing much easier. The fact that the welding goes smoothly without hissing, sudden bursts, or difficult-to-close holes demonstrates that no fluid from the capsules is lost during this process (Audétat and Bali 2010). Based on the three steps above, the capsules are finally sealed and maintain a regular shape (cylindrical) and uniform thickness (1 mm). Our tests in which the capsules were weighed between the production of the preliminary seal and the final welding showed that the loss of volatiles and metal during welding is negligible (see Table 1). The sealed capsules were also prechecked for leakage by holding in a 110 °C vacuum drying oven for more than 10 h. All capsules passed the inspection with a mass change of less than 1 mg (see Table 1). In fact, one important reason why such a good seal can be achieved by this technique is that the sealing effectiveness lies not only on the welded seam at one end of the capsule, as in other sealing methods, but also on the larger sealing area formed between the walls of the inner and outer tubes under high pressure (see Fig. 1f, the walls were completely fused together in the testing experiments). To date, capsules constructed from pure Cu and pure Ag have been employed successfully in experiments for fluid inclusion synthesis. The sealing effectiveness is essentially not changed, though copper or silver could interfere with oxygen fugacity or diffuse into the run products to some extent (Audétat et al. 2018). Aspects such as oxygen fugacity or diffusion could also be utilized or avoided by choosing suitable capsule materials according to the experimental requirements. Theoretically, the capsule sealing technique could be used with any metal and is especially suitable for experiments with a small fluid/solid mass ratio. If more fluid is incorporated, there is a risk that the charged fluid may overflow during the reverse-buckling operation. To avoid fluid overflow, the inner tube containing massive fluid could be frozen before the reverse-buckling operation. Another problem associated with the sealing method is that some air may be entrapped in the capsule; compacting solid charges as tightly as possible when loading the inner tube would minimize the disturbance.

Hydrothermal experiments: Synthesis of fluid inclusions

Experimental synthesis of fluid inclusions has been widely used in many fields of earth science for several decades (Doppler et al. 2013; Hack and Mavrogenes 2006b; Spandler et al. 2007, 2014; Tasy et al. 2016; Zhou et al. 2016). In experiments, the fluid and host mineral must be completely sealed in the capsule, until a certain amount of fluid is captured by the host mineral during fracture healing and (or) overgrowth of crystal surfaces. Thus, to examine a new capsule sealing technique, the experimental synthesis of fluid inclusion is not only a good test but also an important practical field of application (e.g., Hack and Mavrogenes 2006a).

Experimental methods

The simple and effective technique described above was tested in 10 consecutive multi-anvil high-temperature and high-pressure experiments for fluid inclusion synthesis (details are shown in Table 1). The traditional fracture healing method for fluid inclusion synthesis was used in the test experiments (Sterner and Bodnar 1984).

Fluid (pure water or solution with 15wt% NaCl), pre-cracked labradorite core and metabasalt powder (Loss on ignition of the whole rock is up to 8.58 wt%) were loaded into Cu or Ag capsules of three sizes: 5.0 mm I.D., 7.0 mm O.D., 12.0 mm outer length; 6.0 mm I.D., 8.0 mm O.D., 12.0 mm outer length; and 10.0 mm I.D., 12.0 mm O.D., 17.0 mm outer length. The amount of loaded fluid ranged from 12 μL to 16 μL in the smaller capsules and was approximately 100 μL in the larger

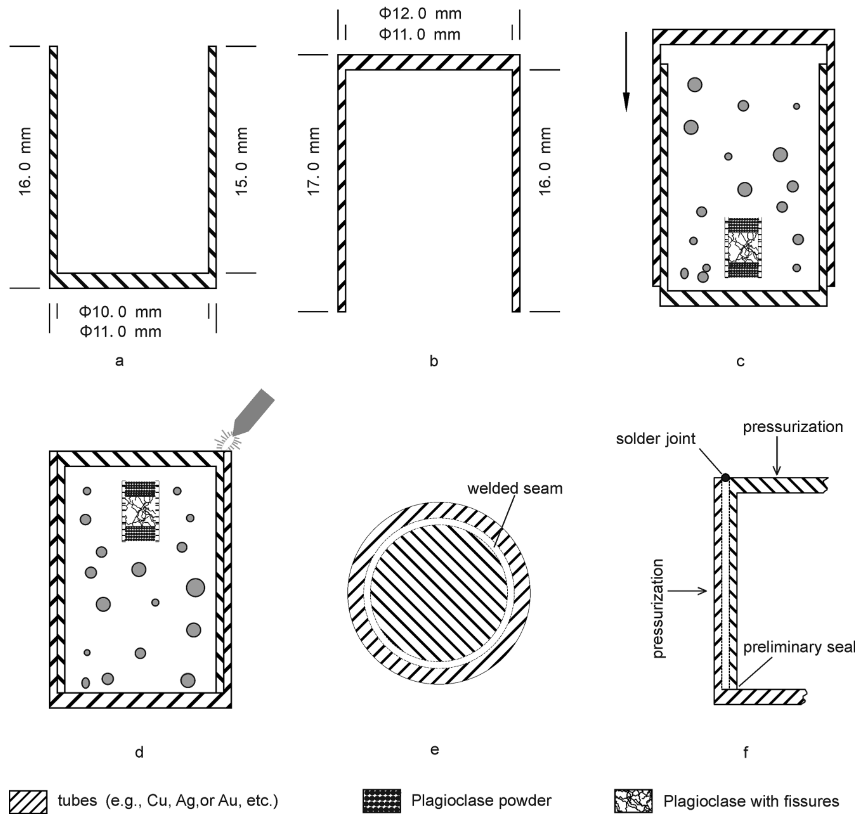

Figure 1. Schematic of the simple and effective capsule sealing technique. (**a**) Cross-sectional view of the inner tube. (**b**) Cross-sectional view of the outer tube. (**c**) Reverse-buckling operation between outer and charged inner tubes. (**d**) The final weld-sealing. (**e**) Top view of the upper part of the capsule (not proportional). The dashed line outlines the circular welded seam with the width less than 0.1 mm. (**f**) Schematic of the capsule seal accomplished by both of the cold-sealing and the weld-sealing (not proportional). The reverse-buckling operation makes a preliminary seal at the lower part first, then the welding shut operation makes the weld-sealing at the upper part by continuous solder joints and the rising confining pressure finally contributes to a solid seal between walls of the inner and outer tubes

capsules. The sealed copper (or silver) capsule was fitted into assemblies consisting of a boron nitride cell, a graphite heater, pyrophyllite inserts and a pyrophyllite cube (indicated in Fig. 3). The temperature was controlled using type-K thermocouples and a Eurotherm temperature controller with an uncertainty of ±5 °C. The pressure was converted from the load and was accurate to 0.1 GPa (Ren and Li 2018; Shan et al. 2007). All the runs were carried out by pressurization to the designed pressure first and then heating to the desired temperature. The pressurization rate was less than 1 GPa/h, and the heating rate was 20 °C/min. The runtime was chosen to be between 48 and 72 h (Table 1). Quenching was performed at a cooling rate of 50 °C/min with synchronous pressure decline. After quenching, the capsules were recovered, cleaned, pierced with a scalpel and further made into doubly polished thin wafers. It is noted that the capsules were weighed with an electronic scale (with accuracy in 0.1 mg) after being loaded, welded shut, prechecked, and annealed, respectively. Additionally, in two runs (D4 and D7) no additional fluids were added into the capsules and the capsules were not welded shut according to the third step either. So fluid inclusions could only be formed by capturing the fluid from de-volatilization of the metabasalt and the effectiveness of cold-sealing based on the first two steps could be tested in the two runs.

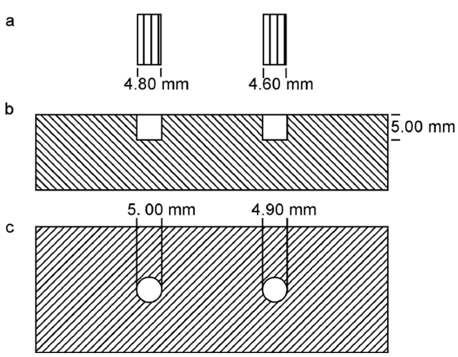

Figure 2. Drawing of the dies used for the punch process. (**a**) The matching pressure levers. (**b**) Front view of the die. (**c**) Top view of the die. Note the precision of the dies is preferably controlled in 0.01 mm, and the specific dimensions of the dies can be adjusted according to the desired size of the capsule.

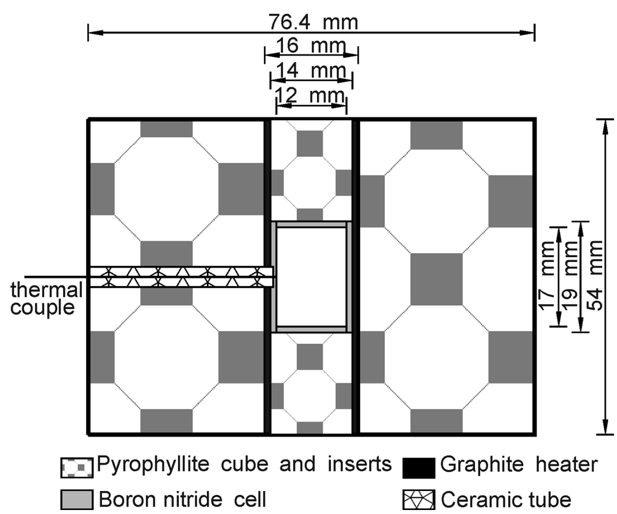

Figure 3. Assembly used for fluid inclusion synthesis in testing experiments.

Table 1. Details of experimental runs

Run no.	Capsule type (mm)	Mass of starting materials added: Metabasalt (mg)	Labradorite (mg)	Fluid (mg)	Charged capsule weight (g)	ΔM_1 (mg)	ΔM_2 (mg)	ΔM_3 (mg)	Pressure (GPa)	Temperature (°C)	Duration (h)
D4	5/7/12 Cu	285.8	37.2	/	2.4750	/	/	−0.5	1.0	900	48
D7	5/7/12 Cu	266.2	42.5	/	2.5320	/	/	−0.7	1.0	800	48
D9	5/7/12 Cu	230.8	35.3	16.0[a]	2.5193	−0.3	−0.3	−1.2	1.0	800	48
D10	10/12/17 Cu	2333.2	47.6	100.0[a]	8.3281	−0.4	−0.1	−2.1	1.2	900	48
D11	10/12/17 Ag	2431.7	44.4	100.0[a]	10.3265	−0.5	0.0	−3.2	1.5	800	72
D12	5/7/12 Cu	334.0	47.9	12.0[a]	2.5794	−0.4	−0.3	−0.7	1.0	900	48
D13	5/7/12 Cu	334.2	31.0	12.0[b]	2.6179	−0.5	+0.1	−0.9	1.0	900	48
D15	6/8/12 Cu	545.4	42.4	12.0[b]	3.1535	−0.6	+0.1	−0.8	1.0	900	48
D16	6/8/12 Cu	511.2	46.7	12.0[b]	2.9757	−0.2	−0.2	−1.3	1.0	900	72
D17	6/8/12 Cu	586.8	47.5	12.0[b]	3.1930	−0.5	−0.2	−0.8	1.0	800	48

Notes: Capsule type includes capsule material and dimension. "5/7/12 Cu" indicates that the resulting capsule was constructed by copper with an inner diameter in 5 mm, outer diameter in 7 mm, and outer length in 12 mm. The fluid marked with the superscript "a" is pure water and that with the superscript "b" is the brine solution with 15 wt% NaCl. "ΔM_1" indicates the weight change of the capsule after being welded shut relative to that of the charged capsule; "ΔM_2" shows the weight change of the capsule after being prechecked relative to that of the welded capsule; "ΔM_3" reveals the weight change of the capsule after being annealed relative to that of the pre-checked capsule. All weight data in Table 1 were determined using an average of five times of weighing results with an error of ±0.1 mg.

Results and discussion

Capsule weights and their changes after being welded shut, prechecked, and annealed are shown in Table 1. Weights of capsules from the 8 runs decrease slightly after the weld-sealing (ΔM_1), with the maximum decrease up to 0.6 mg from the run D15, which indicates that the loss of volatiles and metal during welding is negligible. After pre-inspection, the changes of the capsule weight (ΔM_2) relative to that after welding are less than 0.3 mg and also not significantly affected by the mass of the fluid and size of the capsule. After quenching, all the recovered capsules remained nicely cylindrical but were shortened by 5~10% during the experiment. The capsule weights are all slightly lower than those after prechecked (indicated as ΔM_3 in Table 1) and the change seems to be related to the annealing time and the amount of fluid charged into the capsules. It is inferred that the measured mass loss of the recovered capsules may be caused by the diffusion of hydrogen produced by water decomposition (Ayers et al. 1992; Katsuta and McLellan 1979; Magnusson and Frisk 2017) out of the capsule. Nevertheless, tiny mass changes (ΔM_2 and ΔM_3) showed that the capsules were kept well sealed during the pre-inspection and annealing stage. When the capsules from the 10 runs were opened, a fluid phase bubbled freely from the piercing point with hissing at the same time. Under a microscope, a large number of fluid inclusions were found in thin wafers from the 10 runs, of which 4 runs (D4, D9, D10, and D11) were the most typical for the synthesized fluid inclusions (see Fig. 4).

Based on the evidence above, it could be claimed that a perfect capsule sealing was achieved, and the capsule sealing technique is feasible for high-temperature and high-pressure experiments. If the capsules were not kept completely sealed during the runtime, fluid or volatiles in the capsule would escape instantaneously and thoroughly under high pressure, and fluid inclusions would not be

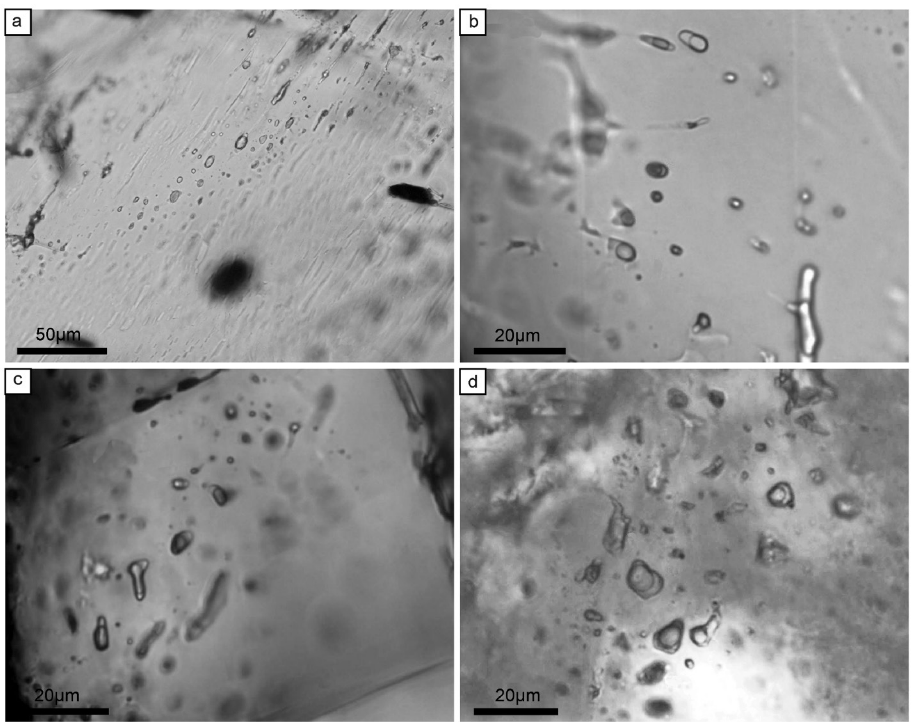

Figure 4. Transmitted-light photomicrographs of the synthesized fluid inclusion in typical runs. (**a**). Two-phase fluid inclusions from experiment D4 (1 GPa, 900 °C). (**b**). Two-phase fluid inclusions from experiment D9 (1 GPa, 800 °C). (**c**). Two-phase fluid inclusions from experiment D10 (1.2 GPa, 900 °C). (**d**). Two-phase fluid inclusions from experiment D11 (1.5 GPa, 800 °C).

synthesized. Another assumption, that inclusions may be formed before the fluid loss, can be excluded by the occurrence of free fluid from the recovered capsules. Furthermore, ΔM_3 was less than 1 mg and fluid inclusions were successfully synthesized in the two runs (D4 and D7), which demonstrated that the capsules were also sealed perfectly only by cold-sealing on the basis of the first two steps. In fact, it is an important reason why such a good seal can be achieved by this technique. The sealing effectiveness lies not only on the welding seam at one end of the capsule, as in other sealing methods, but also on the larger sealing area formed between the walls of the inner and outer tubes due to the cold-sealing (the walls were completely fused together). Furthermore, the sealed capsule has a uniform thickness (1 mm) and a regular shape (cylindrical), which can reduce the risk of capsule rupture caused by deformation.

Implications

This simple and effective capsule sealing technique has been proven to be reliable by the successful experimental synthesis of plagioclase hosted fluid inclusions. In comparison with cold-sealing or weld-sealing, the proposed technique has the following advantages. First, the capsule sealing technique is simple, effective, and easy to operate. The technique does not require a capsule body and lid with a complex structure, nor does it require dies or special tools. The critical weld-sealing operation is easier to complete due to the narrow and uniform slit surrounded by more metal, during which loss of volatilization is prevented by the preliminary seal. Second, the capsules can be sealed with uniform thickness and regular shape, prechecked for leakage in an oven, and annealed under high temperature and high pressure with less deformation, which could improve the success rate of experiments. Third, the theoretically required capsule materials can be changed (such as precious metals, alloys, etc.), as can the dimensions required to construct a capsule with the desired size and wall thickness (large volume or thick wall). Thus, the sealed capsule is suitable not only for piston cylinders but also for multi-anvil presses and other gas-media or hydrothermal-media apparatuses, such as autoclaves and pressure vessels, which means a wider range of temperatures and pressures and thus more fields of application.

This technique could be used in most high-temperature and high-pressure experiments currently reported with fluids, such as experiments involving fluid inclusion synthesis (as verified in this study), mineral solubility determination, fluid-rock reactions related to phase relations, element solubility, diffusion, partition, and fractionation (Bakker and Doppler 2016; Ballhaus et al. 1994; Doppler et al. 2013; Hack and Mavrogenes 2006b; Loucks and Mavrogenes 1999; Simon et al. 2007; Spandler et al. 2007, 2014; Tasy et al. 2016; Zhou et al. 2016).

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