Experimental melt inclusion homogenization in a hydrothermal diamond-anvil cell: a comparison with homogenization at one atmosphere

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ABSTRACT

Melt inclusion (MI) homogenization experiments are essential for determining the pressure–volume–temperature–composition (P–V–T–X) parameters of magma systems. The hydrothermal diamond-anvil cell (HDAC) is currently the only equipment that can exert external pressure on MIs while allowing in situ observation of MI phase changes during heating. The HDAC’s pressure potentially prevents the MI diffusion that, under heating at one atmosphere, produces artificially elevated measurements of phase transition temperatures. It is important to compare the phase transition temperatures measured using HDAC at elevated external pressure with those obtained using conventional equipment at one atmosphere. Such a comparison not only helps assess the reliability of HDAC phase transition temperatures but also helps determine phase transition temperatures that are naturally occurring in MI during the natural history of cooling.

In this study, we homogenized MIs hosted in quartz from the granitic porphyry in the Yixingzhai Au deposit, China, using HDAC at an elevated pressure of ~(140–230) MPa. We compared our experimental results with published data measured using a Linkam TS1500 stage at one atmosphere. The experiments show that the initial melting temperature ($T_{\text{iniM}}$), total melting temperature ($T_{\text{TotM}}$), and total homogenization temperature ($T_{\text{hTot}}$) of the MIs are 695 ± 20 °C, 780 ± 15 °C, and 833 ± 17 °C respectively. These phase transition temperatures are as much as 374 °C lower than the corresponding values measured at one atmosphere using the
Linkam stage. Moreover, the temperatures measured using HDAC agree with actual values estimated using the linear extrapolation method based on correlations of MI size with phase transition temperatures measured using the Linkam stage. Based on the experimental HDAC results, we estimate that MIs in the Yixingzhai Au deposit were trapped at ~140 Ma and contained ~2 wt% H$_2$O. These figures are consistent with previously estimated emplacement pressures and H$_2$O contents of granitic magmas in granitic porphyry-type Cu–Au deposits. These features demonstrate that MI-homogenization experiments using HDAC at suitably elevated pressures can yield reliable naturally occurring phase transition temperatures in MI during the melt cooling process.

**Keywords:** melt inclusions, hydrothermal diamond-anvil cell, Linkam heating stage, homogenization experiment, H$_2$O content

**INTRODUCTION**

Melt inclusions (MIs) are naturally occurring drops of magma trapped among crystallizing magmatic mineral grains (e.g., olivine, pyroxene, feldspar, hornblende, quartz, and garnet). MIs occur in both eruptive and intrusive rocks (Halter et al. 2004; Lowenstern 1995). Once trapped at high pressure–temperature ($P$–$T$) conditions, MIs are sealed in the relatively incompressible host minerals, preserving physicochemical information about the surrounding magma medium during the crystallization of the
host minerals (Smirnov et al. 2003; Webster and Thomas 2006). Post-entrapment, crystallization or devitrification can occur when the $P$–$T$ of the ambient environment decrease, producing compositional heterogeneity in any given inclusion. Therefore, in order to determine the pressure–volume–temperature–composition ($P$–$V$–$T$–$X$) evolutionary trajectories of MIs during the crystallization of a melt, as well as MI compositions, MIs must be experimentally homogenized for analysis of various kinds: electron microprobe analysis, secondary ion mass spectrometer, and Fourier-transform infrared spectroscopy (Roedder 1979; Schiano 2003; Thomas 2000).

Several heating techniques have been used to successfully homogenize MI at one atmosphere pressure, either in a microscope-mounted stage (e.g., Linkam stage; Esposito et al. 2012; Fedele et al. 2003; Lowenstern 1994; Magakyan et al. 1993; Reyf 1997) or tube furnace (Raia et al. 2000; Stockstill et al. 2005; Thomas and Webster 2000; Webster et al. 1997; Yang and Bodnar 1994). These heating techniques work well for MI with low concentrations of volatiles, such as MIs hosted in volcanic rocks (Bodnar and Student, 2006; Cannatelli et al. 2016). Using these techniques, the measured homogenization temperatures ($T_h$s) and behavior are affected by the heating rate, inclusion size, and volatiles content (Audétat and Lowenstern 2014; Bodnar and Student, 2006; Danyushevsky et al. 2002; Lowenstern 1994; Qin et al. 1992; Student and Bodnar 1999, 2004; Thomas 1994). For example, large inclusion size and long heating time will increase the likelihood that the inclusion composition (e.g., $H_2O$) change as a result of component diffusion out of (or into) the inclusion, and then
increase the $T_h$ deviation from the correct value (Massare et al. 2002; Severs et al. 2007; Thomas 1994). Consequently, the kinetic experiments of volatile diffusion inside MIs are required to assess the $T_h$s of MIs heated at one atmosphere pressure, as described by Danyushevsky et al. (2002) and Thomas (1994). Moreover, if the MIs contain significant amounts of volatiles (e.g., H$_2$O), such techniques are hard to homogenize MIs as they are commonly decrepitated before $T_h$ is reached, owing to the high internal pressures generated during heating (Audétat and Lowenstern 2014; Bodnar and Student 2006). Therefore, MIs enriched with volatiles were usually heated under an elevated confining pressure with high-pressure (cold-sealed or internally heated) vessels (Anderson et al. 2000; Severs et al. 2007; Skirius et al. 1990; Student and Bodnar 2004; Thomas et al. 2003), or in a piston cylinder apparatus (Bartoli et al. 2011; Cesare et al. 2011; Ferrero et al. 2012). Using the pressurized equipment, MIs are heated incrementally and then quenched after each heating step for observing and obtaining the stable phase transition temperatures (Thomas and Davidson 2016b). This technology does not allow MIs to be monitored in situ during heating, the phase transition temperatures could be measured with some errors (Student and Bodnar 2004); it could also cause MIs to be overheated and then increase the chance of MI decrepitation in some cases, especially for the larger inclusions (greater than approximately 30–50 µm) (Bodnar and Student 2006).

HDAC was designed to study the properties of fluids at pressures up to 2.5 GPa and temperatures ranging from −190 to 1200 °C (Bassett et al. 1993; Li et al. 2016,
Thomas et al. (2006) suggested HDAC be used in MI-homogenization experiments, to provide the opportunity to observe phase transitions \textit{in situ} during MI heating under elevated appropriate external pressures. Later, Li and Li (2014) and Li and Chou (2017) established the MI-homogenization experimental method using HDAC. However, more experiments are essential for evaluating whether the phase transition temperatures observed using HDAC experiments reflect those naturally occurring in MIs during the natural history of cooling. Therefore, in this study, we used HDAC to homogenize MIs in quartz from the Yixingzhai granite porphyry-type Au deposit, Shanxi Province, China, in order to compare the homogenization results of MIs heated in a Linkam heating stage at one atmosphere as performed by Wang (2014). The results of this study illustrate the evident effect of the external pressure on the measured phase transition temperature within a given MI while demonstrating the reliability of phase transition temperatures measured using HDAC.

**FEATURES OF MELT INCLUSIONS IN THE YIXINZHAI GOLD DEPOSIT**

The Yixingzhai Au deposit is a representative porphyry-type gold deposit in northern Shanxi Province, China, located at the intersection of the Taihang and Yanshan orogenic belts, on the northern margin of the North China Block. The Yixingzhai Au deposit consists mainly of quartz porphyry and its associated cryptoexplosive breccia. Among these rocks, quartz porphyry is the main metallogenic rock, bearing a typical porphyritic texture (Wang 2014). In this study, we selected MIs hosted in the quartz phenocrysts of quartz porphyry from the Yixingzhai
Au deposit for use in MI-homogenization experiments. In the quartz porphyry, quartz occurs as 2–3 mm phenocrysts of subhedral shape (Fig. 1), hosting numerous MIs (Fig. 2). These MIs exhibit the characteristics of primary inclusions: typically elliptical in shape, 10–25 μm in size, occurring in isolation or in MI assemblages (MIAs) consisting of MIs with uniform shape and composition (Fig. 2a). The MIs are commonly dark to opaque in transmitted light, owing to the presence of abundant crystallites formed during MI devitrification. As a result, aqueous phases were too obscure to be observed by optical microscopy at room temperature. The MIs are composed primarily of feldspar and quartz as identified by Raman spectroscopy (Wang 2014). Additionally, some MIs coexist with CO₂–H₂O fluid inclusions (Fig. 2b).

**EXPERIMENTAL METHODS**

In the homogenization experiments of MIs using HDAC, granitic porphyry wafers hosting MIs were cut from the same granitic porphyry samples used for heating experiments in the Linkam stage by Wang (2014) and then double-polished. Large, isolated, regularly-shaped MIs were selected for HDAC homogenization experiments (HDAC-VT, Li et al. 2016). The HDAC-VT sample chamber consists of a hole (diameter 1.0 mm) at the center of a Re gasket (diameter 3.0 mm, thickness 0.25 mm) placed between the two diamond-anvil faces (diameter 1.6 mm). Sample temperatures in the sample chamber were measured via two calibrated K-type thermocouples with their temperature sensing tips attached separately to the two diamonds. The
thermocouples were calibrated using the triple point of H$_2$O (0.01 °C) and the melting points of NaNO$_3$ (306.8 °C) and NaCl (800.5 °C). The reported temperatures were precise to ± 0.5 °C and accurate to ±1.5 °C for those above 380 °C.

In the experiments, a quartz wafer piece hosting MIs, deionized water, and vapor bubble(s) were sealed in the sample chamber through the compression of the two diamond anvils of the HDAC (Fig. 3). The wafer piece was <1.0 mm in length and width, and ~0.15 mm in thickness, so that the wafer piece would not break while sealing the sample chamber. During heating, the initial heating rate was 5 °C/min up to a temperature of 400 °C. A rate of 1 °C/min was then maintained until total MI-homogenization. Following these heating steps, a MI-homogenization experiment took 6–8 hours. In addition, at temperatures exceeding 200 °C, a continuous flow of a mixed gas (98% Ar and 2% H$_2$) outside of the HDAC sample chamber was kept to protect the HDAC’s diamond anvils and resistance heating wires from oxidation.

During heating, the pressure in the HDAC chamber was calculated from the known bulk H$_2$O density and temperature in the chamber using the equation of state of Wagner and Pruß (2002). The bulk H$_2$O density was determined by the temperature ($T_{\text{hDC}}$) at which the vapor bubble disappeared in the HDAC sample chamber. $T_{\text{hDC}}$ in each experiment was measured in the heating process and again during reheating after generation of a vapor phase in the cooling process; the latter measurement was used to calculate the H$_2$O density according to the equation of state of H$_2$O (Wagner and Pruß 2002). In this paper, we adjusted the $T_{\text{hDC}}$ to 370–373 °C in each case, by shrinking
the sample chamber or leaking a certain amount of $\text{H}_2\text{O}$ from the sample chamber by tightening or loosening the HDAC pressurizing screws as needed. Accordingly, the $\text{H}_2\text{O}$ loaded in the sample chamber exhibited a density of 451.43–398.68 kg/m$^3$, and pressures of ~200 MPa were applied at 800 °C to the MIs in the sample chamber. Such pressure roughly balanced with the internal pressure of MIs, since estimated metallogenic pressures of porphyry deposits are about 100–200 MPa (Cline 1995; Richards 2011). Hence, MI volume stretching was minimized, and MI decrepitation was prevented during heating.

Wang (2014) performed MI-homogenization experiments at one atmosphere in a Linkam TS1500 heating stage, referring to the heating process described by Esposito et al. (2012). In the experiments, the heating stage was calibrated using the melting temperature of NaCl (800.5 °C). The difference between the known melting temperature of the calibration standard and the measured temperature was always <5 °C. In Wang’s experiments, MIs hosted in quartz wafer (~0.2 mm in thickness) were observed through a ZEISS Axioskop 50× optical microscope. Below 500 °C, a heating rate of 5 °C/min was maintained; after holding at 500 °C for half an hour, MIs continued to be heated at a rate of 1 °C/min until total MI-homogenization.

In the MI-homogenization experiments, the heating rate was critical for obtaining exact temperatures (Danyushevsky et al. 2002). A high heating rate could result in the melting process lagging behind, yielding measured transition temperatures higher than the true values. On the other hand, a low heating rate could
lead to re-equilibration (i.e., equilibration at conditions different from those at the moment of trapping) of a MI with its host, or even diffusion of volatiles such as H$_2$O (Severs et al. 2007). In the present paper, the heating rate of 1 °C/min ensured thermal
equilibrium inside each MI while preventing the diffusion of volatiles during heating, according to kinetic experiments performed by Danyushevsky et al. (2002) and Student and Bodnar (1999).

**EXPERIMENTAL RESULTS**

Table 1 provides the experimental microthermometry results for the MIs in quartz phenocrysts in granitic porphyry from the Yixingzhai Au deposit using HDAC. In the MI heating process, the solid phases in each MI began to melt as the temperature reached the MI’s initial melting temperature ($T_{iniM}$). During further heating, the MI’s remaining solid phase seemingly melted into a molten state at the total melting temperature ($T_{TotM}$) of the solid phases (Fig. 4), with the possibility that at $T_{TotM}$ few fine-feldspar crystals (not visible) could remain in contact with the vapor bubble or on the MI wall as suggested by Student and Bodnar (2004). This vapor phase coexisted with the melt phase in each MI until the vapor phase totally dissolved into the melt, yielding a total homogenization temperature ($T_{hTot}$) of the MI. After being totally homogenized, each MI occupied a larger volume than before heating, transformed from an irregular or negative crystal shape to a round or elliptical shape (Fig. 4). This transformation indicated that the crystallized phases on the MI wall were melted, in keeping with the known characteristics of MIs before and after
homogenization in igneous rocks (Frezzotti 2001). Additionally, the heating results of MIs performed using a Linkam stage by Wang (2014) are listed in Table 2, and the phase transition process of one representative MI during heating is shown in Fig. 5.

As shown in Figs. 4 and 5, the phase transition sequence and behavior of MI in homogenization experiments using a Linkam TS1500 stage were qualitatively similar to those obtained using HDAC. However, close comparison reveals the robust effects of elevated external pressure on MIs’ measured phase transition temperatures (Tables 1 and 2). $T_{\text{IniM}}$, $T_{\text{TotM}}$, and $T_{\text{hTot}}$ as measured using the Linkam stage were 719–826 °C, 845–1046 °C, and 943–1190 °C, respectively (Table 3; Fig. 6). These values were up to 374 °C higher than the corresponding values obtained using HDAC: 675–720 °C, 760–791 °C, and 816–850 °C, respectively (Table 3; Fig. 7).

**DISCUSSION**

**Phase transition temperatures of melt inclusions estimated from homogenization experiments with Linkam stage**

As shown in Tables 1, 2, and 3, the MI-homogenization results using HDAC and using the Linkam stage are substantially different. Such differences can be ascribed to the external pressure applied to the MIs during heating, as the external pressure can prevent decrepitation and volatile diffusion or leakage (Thomas and Davidson 2016a). When the MIs were heated at one atmosphere, homogenization was slow due to high melt viscosity and a correspondingly low diffusivity for escaping volatiles (Hurai et al. 2015). Consequently, the measured MI $T_{\text{hTot}}$ using the Linkam stage is significantly
greater than the equilibrium temperature at which the aqueous phase is separated from
the melt inside MIs during cooling at depth.

Generally, MIs of small size possess little absolute wall area for volatile diffusion
during the heating process, as volatiles mainly escape through either preexisting or
strain-induced dislocations and decrepitation cracks (Hurai et al. 2015; Severs et al.
2007), as well as along microcracks that form at the quartz α–β transition at ~573 °C
at one atmosphere pressure (Severs et al. 2007). Usually, in hours, the diffusion of
H₂O to or from MIs can occur during heating (Cannatelli et al. 2016; Chen et al., 2011;
Massare et al. 2002; Portnyagin et al. 2007), particularly when the MIs are hosted in a
thin mineral wafer rather than relatively large mineral grains in the heating
experiments. Hence, at a constant heating rate, MI-homogenization temperatures are
controlled by diffusion as a function of inclusion volume and the duration of the
heating experiment as described by Thomas (1994). As a consequence, this can be
seen as a correlation between inclusion size and phase transition temperature as was
found in homogenization experiments using the Linkam stage, even though MIs were
heated in several hours (Student and Bodnar 1999). This phenomenon has also been
observed in MI-homogenization experiments using a furnace. In these experiments,
MIIs were heated to a constant temperature higher than the solidus for the given melt
composition and then quenched to analyze possible evidence of homogenization
(Thomas 1994; Thomas and Klemm 1997). In such quenching homogenization
experiments with a constant 20-hour heating duration, the phase transition
temperatures of MIs and the corresponding diameters of MIs showed a clear linear relationship (Thomas 1994). This positive correlation was used to infer the true phase transition temperature at a theoretical inclusion size of zero, for which no diffusion kinetics would bias the measurement (Hurai et al. 2015; Student and Bondar 1999).

Similarly, we use data measured at one atmosphere in a Linkam stage by Wang (2014) to extrapolate the relationship between \( T_{htot} \) and diameter to an infinitesimally-small MI (Fig. 6). These data show a good linear correlation of \( T_{htot}(\degree C) = 11.748d + 825 \), where \( d \) represents MI diameter in \( \mu m \), as shown in Fig. 6. Similarly, the correlations of \( T_{TotM} \) and \( T_{IniM} \) with MI size can roughly be expressed by \( T_{TotM}(\degree C) = 9.1928d + 760 \) and \( T_{IniM}(\degree C) = 3.3436d + 690 \), respectively. These latter correlations exhibited relatively low correlation coefficients, possibly because \( T_{TotM} \) and \( T_{IniM} \) were difficult to determine with high precision through observation. These correlations, all positive, indicate that these MIs hosted in quartz from porphyry samples were trapped from uniform magma at a similar temperature, as indicated in experiments by Student and Bodnar (1999). Moreover, the slopes in the linear equations of \( T_{htot} \), \( T_{TotM} \), and \( T_{IniM} \) vs. MI diameter decreased in that sequence (Fig. 6), indicating the effects of MI volatile diffusion on phase transition temperatures during heating. Based on these positive correlations, we extrapolated linearly to determine the \( T_{htot} \), \( T_{TotM} \), and \( T_{IniM} \) of a hypothetical, infinitesimally small MI (diameter = 0 \( \mu m \)). These estimates were \( \sim 825 \degree C \), \( \sim 760 \degree C \), and \( \sim 690 \degree C \), respectively, among which the extrapolated \( T_{htot} \) (825 ± 8 \( \degree C \)) exhibits relatively small error, closely approximating the actual \( T_{htot} \).
Phase transition temperatures of melt inclusions measured using hydrothermal diamond-anvil cell

Granitic porphyry deposits typically form at pressures of 100–200 MPa (Cline 1995; Richards 2011), so such pressures could be assumed for the studied MIs. After the homogenization of fluid phases during heating, however, the internal pressure would have increased along the fluid isochores, potentially reaching a higher pressure than the MI trapped pressure prior to homogenization (Fig. 8; Student and Bodnar 1996). Therefore, in our homogenization experiments of MIs using HDAC, external pressures of ~(140–230) MPa were exerted on the MIs after initial melting of the solid phase (Table 1). The phase transition temperatures of MIs as measured using HDAC ($T_{hTot} = 833 \pm 17 ^\circ C$, $T_{TotM} = 780 \pm 15 ^\circ C$, and $T_{IniM} = 695 \pm 20 ^\circ C$; Fig. 7a) were identical, within error, to the temperature ranges estimated from the linear correlations of phase transition temperatures measured at one atmosphere with MI size as shown in Fig. 6. Moreover, $T_{hTot}$, $T_{TotM}$ and $T_{IniM}$ did not show the effects of external pressures and MI size on measured phase transition temperatures (Fig. 7). This finding indicates that no significant diffusion that occurred in MIs heated at one atmosphere using the Linkam stage occurred during heating using HDAC.

The MI-homogenization process showed that the bubble finally dissolved into a hydrous melt phase, indicating that the MIs homogenized into H$_2$O-saturated melt. Moreover, as shown in Fig. 2b, the coexistence of MIs and FIs indicates that silicate MIs were trapped from H$_2$O-saturated melt (Student and Bodnar 1999), from which
aqueous fluids exsolved. Therefore, the $T_{\text{hTotS}}$ represent the trapping temperatures of MIs. Wang et al. (2010) used the whole-rock zirconium saturation thermometer to estimate the average crystallization temperature of the Dexing granodiorite porphyry rock mass to be $790 \pm 50 \, ^\circ\text{C}$. Student and Bodnar (2004) homogenized the MIs hosted in quartz phenocrysts of quartz latite from Red Mountain, Arizona, USA, under pressure in a cold-seal pressure vessel at $810–835 \, ^\circ\text{C}$. These temperatures are compatible with the present homogenization temperature results for MIs in the Yixingzhai Au deposit using HDAC.

In summary, the MI-homogenization temperatures measured in HDAC experiments at suitably elevated external pressures could be close to the actual values, and can be used to indicate the $P–V–T–X$ properties of MIs. Moreover, the HDAC experiments directly yield concrete values, in comparison with the linear extrapolation method required for experiments performed using the Linkam stage at one atmosphere (e.g., Fig. 6). This distinction is particularly important from a practical perspective because the linear extrapolation method is extremely time-consuming and can be applied only to samples with numerous primary inclusions originating at the same temperature with a uniform chemical composition, phase composition, and water content. Decrepitation cracks and the entrapment of a heterogeneous melt-fluid mixture make the use of this method impossible (Hurai et al. 2015).

**Entrapment conditions of melt inclusions inferred from homogenization**
Based on the phase changes in granitic melt inclusions suggested by Student and Bodnar (1996) and Hurai et al. (2015), the P–T trajectory of the MIs during heating is described in Fig. 8. With increasing temperature, the fluid phases inside MI homogenize into liquid or vapor (point A in Fig. 8), although such a process was not clearly observed in the studied MIs. Subsequently, the P–T trajectory of the MI moves along the fluid isochore, and the incipient melting of solid phases occurs when the isochore intersects the fluid-saturated solidus of the granitic magma at the MI’s $T_{\text{ IniM}}$ (point B in Fig. 8). With the onset of melting, the fluid phase volume decreases as the fluid phase gradually dissolves in the melt (range C between points B and D in Fig. 8) until total melting of solid phases at the MI’s $T_{\text{ TotM}}$. Finally, fluid is totally dissolved into the melt phase at point D in Fig. 8, corresponding to $T_{\text{ hTot}}$ on the H$_2$O-saturated liquidus. The $T_{\text{ hTot}}$ and the corresponding MI homogenization pressure represent the MI trapping P–T conditions as MIs were trapped from H$_2$O-saturated granitic melt.

As shown in Fig. 8, the MI trapping pressure can be roughly determined by the intersection of the isochore of the fluid phase within the MI and the water-saturated solidus curve for granite, as suggested by Audétat and Pettke (2003), Audétat et al. (2008), and Yang and Bodnar (1994). Accordingly, we roughly estimated the trapping pressure of MIs in the Yixingzhai granitic porphyry to be ~140 MPa, based on the H$_2$O-saturated granite melt solidus and the ~695 °C $T_{\text{ IniM}}$ of MIs (Fig. 8). This figure agrees with the reported 100–200 MPa estimates of the metallogenic pressure of
granitic porphyry-type Cu–Au deposits (Cline 1995; Richards 2011). Moreover, MI
H$_2$O content can be estimated to be ~2 wt% based on the estimated MI trapping
pressure (~140 Ma) and $T_{hTot}$ (~836 °C) (Fig. 8), since $T_{hTot}$ corresponds to the
liquidus of the H$_2$O-saturated granite melt at the MI trapping pressure (Li et al. 2017;
Poutiainen and Scherbakova 1998). Such H$_2$O content should be considered as the
minimum value, because the presence of additional volatiles (e.g., F, CO$_2$) would also
affect the liquidus temperature (King and Holloway 2002; Manning 1981). Moreover,
the 2 wt% H$_2$O content of MIs is consistent with that (1.6–3.6 wt%) of comenditic
pumice and pyroclastic flow as obtained by Li et al (2006), as well as with the 2.1–2.8
wt% H$_2$O contents in granitic magma that produced the skarn-porphyry Cu–Fe–Au
deposit in Tieshan, China (Zhou et al. 2020).

**IMPLICATIONS**

Our study showed that MI-homogenization experiments using heating stages
(e.g., the Linkam series stages) at one atmosphere generally yield MI phase transition
temperatures significantly higher than the actual occurring temperatures during the
melt natural cooling process due to volatile diffusion or leakage from MIs during
heating. Therefore, temperatures measured at one atmosphere require detailed
interpretation such as the linear extrapolation method described above to account for
volatile diffusion before they can be used to research the $P$–$V$–$T$–$X$ properties of MIs.
However, the extrapolation method is extremely time-consuming and is available only
for assemblages of MIs trapped from a uniform magma at a single temperature.
In our study, HDAC was used to exert an appropriate external pressure on MIs to balance the internal pressure of the MI during heating, effectively preventing volatile-rich MI decrepitation and volatile diffusion while allowing the homogenization process to be monitored continuously in situ. As a result, in MI-homogenization experiments using HDAC, measured MI phase transition temperatures were not artificially elevated but rather coincided with naturally occurred phase transition temperatures during cooling process, with measured phase transition temperature not affected by MI size. As a result, the measured temperatures were suitable for direct use in calculating the $P–V–T–X$ properties of magma such as the $\text{H}_2\text{O}$ content in granitic magma. Moreover, the new type of HDAC used (HDAC-VT type) was more stable than previous versions, and was able to run under high $P–T$ conditions for a long time under the control of a computer, so that HDAC is no longer laborious for MI-homogenization. Moreover, a cooling system was designed for HDAC (Li et al. 2020), which benefits for the fast quenching of MIs samples in homogenization experiments. Therefore, we recommend the further adoption of the HDAC as a tool for MI-homogenization experiments based on the advantages demonstrated in this study.

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FIGURE CAPTIONS

Figure 1. Petrography of quartz porphyry in the Yixingzhai Au deposit, Shanxi Province, China. Quartz porphyry sample was shown in (a) with a marker of quartz (Qtz) phenocryst. Microscopic photos in the cross-polarized light of quartz porphyry are shown in (b, c, and d) in which quartz (Qtz) phenocryst, groundmass (Gm, mainly composing of microcrystalline quartz and feldspar), and melt inclusions (MIs) are marked.

Figure 2. Photographs of fluid inclusions (FIs), melt inclusions (MIs), and a MI assemblage (MIA) in quartz phenocrysts from the granitic porphyry in the Yixingzhai Au deposit, China.

Figure 3. Photograph of the hydrothermal diamond-anvil cell sample chamber containing a quartz wafer, deionized water, and vapor bubbles at 25 °C.

Figure 4. Images showing the homogenization process of one melt inclusion (No. 11 in Table 1) in a quartz phenocryst of the quartz porphyry from the Yixingzhai Au deposit using the hydrothermal diamond-anvil cell. The solid phases inside the MI were initially melted at ~682 °C and totally melted at ~783 °C. After the total melting of solid phases, a vapor phase clearly coexisted with the melt until the complete dissolution of the vapor phase into the melt at the total homogenization temperature (835 °C).
Figure 5. Images showing the homogenization process of one melt inclusion (No. 3 in Table 2) in a quartz phenocryst of the quartz porphyry from the Yixingzhai Au deposit using the Linkam TS1500 stage at one atmosphere of pressure (Wang 2014). The MI-homogenization process here is similar to that observed in the hydrothermal diamond-anvil cell as shown in Fig. 4. The initial and total melting temperatures of solid phases within the MI were at ~780 °C and ~990 °C, respectively, and the vapor phase totally dissolved into the melt at 1144 °C.

Figure 6. Diagram showing the positive correlations of the initial melting temperature ($T_{\text{IniM}}$; diamonds) and the total melting temperature ($T_{\text{TotM}}$; dots) of the solid phases, and the total homogenization temperature ($T_{\text{hTot}}$; squares) of melt inclusions (MIs) with MI diameter ($d$) in homogenization experiments performed at one atmosphere using the Linkam TS1500 heating stage.

Figure 7. Diagram showing the correlations of the initial melting temperature ($T_{\text{IniM}}$; diamonds) and the total melting temperature ($T_{\text{TotM}}$; dots) of the solid phases, and the total homogenization temperature ($T_{\text{hTot}}$; squares) of melt inclusions (MIs) with (a) external pressure on MIs and (b) MI diameter in homogenization experiments performed using the hydrothermal diamond-anvil cell.
Figure 8. Schematic diagram showing the $P$–$T$ path (bold black line) of melt inclusions (MIs) in homogenization experiments using the hydrothermal diamond-anvil cell. The range of external pressures exerted on MIs during heating is shown as a gray region, determined by the vapor bubble disappearance temperatures ($T_{\text{HDAC}}$) in the HDAC sample chamber. The $\text{H}_2\text{O}$-saturated solidus curve and liquidus curves are taken from Holtz et al. (2001). Points A, B, and D correspond to the $P$–$T$ conditions of heating states a, b, and d shown in the inset. Range C between points B and D indicates the possible $P$–$T$ path in the solid phase-melting process during heating. Given the ~695 °C initial melt temperature of MIs and the fluid-saturated granitic magma solidus, the entrapment pressure of the MIs was estimated to be ~140 MPa, and the $\text{H}_2\text{O}$ content in MIs was estimated to be ~2 wt% according to the $\text{H}_2\text{O}$-saturated magma liquidus under the entrapment $P$–$T$ conditions (~140 MPa, ~833 °C) of the MIs.
TABLE 1. Microthermometry experimental results for melt inclusions (MIs) in quartz phenocrysts from the Yixingzhai Au deposit, China, using the hydrothermal diamond-anvil cell (HDAC-VT) heating stage.

<table>
<thead>
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<th>Exp. Nos.</th>
<th>Size (μm)</th>
<th>$T_{\text{hDC}}$ (°C)</th>
<th>$P_{\text{IniM}}$ (MPa)</th>
<th>$T_{\text{IniM}}$ (°C)</th>
<th>$P_{\text{TotM}}$ (MPa)</th>
<th>$T_{\text{TotM}}$ (°C)</th>
<th>$P_{\text{hTot}}$ (MPa)</th>
<th>$T_{\text{hTot}}$ (°C)</th>
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$T_{\text{hDC}}$—vapor bubble disappearance temperature in the HDAC sample chamber; $T_{\text{IniM}}$—MI’s solid phase initial melting temperature; $T_{\text{TotM}}$—MI’s solid phase total melting temperature; $T_{\text{hTot}}$—total homogenization temperature of MI; $P_{\text{IniM}}$, $P_{\text{TotM}}$, and $P_{\text{hTot}}$—corresponding pressures in the HDAC-VT sample chamber at points $T_{\text{IniM}}$, $T_{\text{TotM}}$, and $T_{\text{hTot}}$, respectively, calculated by employing the equation of state of H$_2$O provided by Wagner and Prüß (2002).
TABLE 2. Microthermometry experimental results of melt inclusions (MIs) in quartz phenocrysts from the Yixingzhai Au deposit, China, using the Linkam TS1500 heating stage (Wang 2014).

<table>
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<th>Exp. Nos.</th>
<th>Size (μm)</th>
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<th>$T_{\text{TotM}}$ (°C)</th>
<th>$T_{\text{hTot}}$ (°C)</th>
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Exp. Nos.—experimental numbers; $T_{\text{IniM}}$—MI’s solid phase initial melting temperature; $T_{\text{TotM}}$—MI’s solid phase total melting temperature; $T_{\text{hTot}}$—total homogenization temperature of MI.
TABLE 3. Ranges of phase transition temperatures from microthermometry experiments on melt inclusions (MIs) from the Yixingzhai Au deposit, China, using the Linkam TS1500 heating stage and the hydrothermal diamond-anvil cell (HDAC-VT).

<table>
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<tr>
<th>Heating stage</th>
<th>$T_{\text{IniM}}$ (°C)</th>
<th>$T_{\text{TotM}}$ (°C)</th>
<th>$T_{\text{hTot}}$ (°C)</th>
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<tr>
<td>HDAC-VT</td>
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<td>Linkam TS1500</td>
<td>719–826</td>
<td>845–1046</td>
<td>943–1190</td>
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$T_{\text{IniM}}$—MI’s solid phase initial melting temperature; $T_{\text{TotM}}$—MI’s solid phase total melting temperature; $T_{\text{hTot}}$—total homogenization temperature of MI.
\[ T_{hTot} = 11.748 \, d + 825 \]
\[ R^2 = 0.98 \]

\[ T_{TotM} = 9.1928 \, d + 760 \]
\[ R^2 = 0.82 \]

\[ T_{IniM} = 3.3436 \, d + 690 \]
\[ R^2 = 0.50 \]
Fig. 8

Glass/crystallite

a-Fluid was homogenized to liquid or vapor
b-Initial melting of solid phases (at ~695 °C)
c-Total melting of solid phases (at ~780 °C)
d-Homogenization (at ~833 °C)

~140 MPa
External pressures exerted on Mls during heating for $T_{hdc}$s (370-373 °C)

$H_2O$-saturated granite solidus

Liquid-vapor curve of $H_2O$