Heating and freezing experiments on aqueous fluid inclusions in anhydrite: Recognition and effects of stretching and the low-temperature formation of gypsum ☆

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Abstract

Aqueous fluid inclusions in anhydrite that are frozen during microthermometry commonly develop new daughter crystals. Heating these inclusions induces the new daughter crystals to decompose. Repeated cycles of crystal growth and decomposition may result in morphological changes of the fluid inclusions. This phenomenon of new mineral growth, presumed to represent the low-temperature hydrate, gypsum, has been recognized for over 20 years. Nevertheless, its occurrence and its significance to the determination of inclusion salinities are not widely appreciated. Similarly, the potential of inclusions in anhydrite to stretch when overheated is commonly assumed, yet tests demonstrating the phenomenon have not been reported. Changes in the ice melting temperature of aqueous inclusions in anhydrite before and after gypsum formation were documented, showing that hydration of the host anhydrite may significantly lower the ice melting temperature and increase the salinity of the residual inclusion fluid. Heating inclusions to induce gypsum to decompose results in the release of bound water, restoring the inclusions to their original salinity. The homogenization temperatures of inclusions that have undergone these phenomena are generally unchanged, indicating that the inclusions' specific volumes are unchanged. However, heating aqueous inclusions in anhydrite more than ten degrees or so above their homogenization temperature commonly results in stretching. Therefore, those who study aqueous inclusions in anhydrite must avoid the determination of ice melting temperatures if new gypsum daughter crystals are present, and must determine homogenization temperatures sequentially with rising temperature to avoid overheating and stretching.
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1. Introduction

Anhydrite [CaSO$_4$] is a common mineral that crystallizes, among other settings, in hydrothermal

ore deposits and geothermal systems. Hydrothermal anhydrite can be a good host for primary and secondary fluid inclusions. It is also potentially a problematic host because of (1) its relatively high, and typically retrograde, solubility in most aqueous solutions; (2) its softness and excellent cleavage, which could allow inclusions to stretch and leak; and (3) its propensity for reaction with aqueous solution to form the hydrate, gypsum [$CaSO_4 \cdot 2H_2O$].

Previous workers have been aware of the potential problems associated with interpreting heating and freezing data from anhydrite-hosted inclusions (e.g., Roedder, 1984, p. 295). Tivey et al. (1998), in particular, noted the possible increase in fluid inclusion salinities both as a result of anhydrite dissolution during cooling from the trapping temperature and as a result of hydration of anhydrite to gypsum. Nevertheless, the consequences of anhydrite hydration to gypsum, and the ease with which inclusions in anhydrite stretch upon overheating, have not been evaluated experimentally and may not be widely appreciated. In this paper, heating and freezing data are reported which demonstrate that the low-temperature hydration of anhydrite to gypsum can dramatically lower the measured ice melting temperature, leading to erroneously high salinity estimates. Furthermore, heating the inclusions above their homogenization temperature may cause them to stretch, making them unacceptable for subsequent heating measurements.

2. Background

2.1. Fluid inclusions in anhydrite

Aqueous fluid inclusions are common in anhydrite. Shikazono et al. (1983) studied them in nodular anhydrite from the Kuroko ore deposits, and Roedder (1984) noted that many porphyry copper deposits contain anhydrite with inclusions. Some active geothermal systems contain inclusions in vein anhydrite, such as the Hohi geothermal area, Japan (Sasada et al., 1986), the Sabatini volcanic district, Italy (Belkin et al., 1988) and the Kakkonda system in Japan (Muramatsu et al., 2000). Active deep-sea hydrothermal systems with inclusion-bearing anhydrite have been studied by Le Bel and Oudin

(1982), Ramboz et al. (1988), Tivey et al. (1998), Petersen et al. (1998, 2000), Lécuyer et al. (1999), Bortnikov et al. (2004) and Vanko et al. (2004), and shallow submarine anhydrite was studied recently from offshore Iceland (Kuhn et al., 2003). Aqueous inclusions have also been studied from anhydrite associated with a post-impact hydrothermal system located within the Chicxulub meteorite impact crater (Gonzales-Partida et al., 2000).

2.2. Previous reports of changes during microthermometry

In his monograph on fluid inclusions, Roedder (1984) showed photographs of a primary anhydrite-hosted inclusion from the Hohi geothermal field in Japan that grew new daughter crystals of gypsum(?) after freezing runs (Taguchi, 1981, cited in Roedder, 1984; and also reported by Sasada, 1986). At that time, Roedder wrote, similar behavior had not been observed during other studies. He suggested a possible reason — many anhydrite-hosted inclusions come from porphyry copper deposits, where the fluids are typically very saline, which might inhibit the reaction of anhydrite to gypsum (Roedder, 1984, p. 295).

More examples of low-temperature induced daughter crystals have been reported recently. Tivey et al. (1998) studied hydrothermal anhydrite cored from the TAG sulfide mound near the mid-Atlantic ridge by the Ocean Drilling Program. They reported that 12 h or more after freezing runs, inclusions commonly developed fine-grained translucent daughter crystals that disappeared if the inclusions were heated to 108–120 °C. Lécuyer et al. (1999), Muramatsu et al. (2000) and Vanko et al. (2004) reported similar behavior. The present paper extends the observations of Vanko et al. (2004), and presents several microthermometric experiments carried out to help understand them.

3. Methods and materials

Doubly-polished plates of delicate anhydrite veins were prepared by vacuum impregnation of small hand specimens with <20 ml of epoxy. Larger volumes of epoxy (e.g., >50 ml) as well as thermoplastic cements

should be avoided, as many epoxies cure exothermically and inclusion-damaging temperatures in excess of 100 °C may result (Harvey Belkin, personal communication, 2004). Grinding and polishing should be done gently so as not to produce deep damage below the surface. The plates, measuring about 50–150 μm thick, were studied in a USGS-type gas-flow heating and freezing stage manufactured by Fluid, Inc. Temperature was measured by means of a thermocouple placed on the sample; the thermocouple measurements were calibrated using quartz with synthetic fluid inclusions having known phase changes at −56.6, 0.0, and +374 °C. Uncertainties in the reported temperature measurements are approximately ±0.1 °C for freezing runs and ±1 °C for heating measurements.

The specimens obtained for this study are from the PACMANUS deep-sea hydrothermal field located in the eastern Manus Basin, Papua New Guinea—a back-arc basin hydrothermal setting (Binns and Scott, 1993; Binns et al., 1996). They were obtained by rotary coring during Leg 193 of the Ocean Drilling Program (Binns et al., 2002). Leg 193 targeted several hydrothermally active sites along Pual Ridge, a Recent andesite–dacite–rhyodacite ridge (Paulick et al., 2004) situated where back-arc extension is propagating into the island arc crust of the New Ireland archipelago (Taylor et al., 1994). Extensive alteration of the primary igneous rocks has resulted in complex secondary mineral assemblages which, greatly simplified, vary from argillic alteration dominated by smectite–chlorite–quartz, to acid-sulfate alteration dominated by anhydrite–pyrophyllite–pyrite–quartz (Binns et al., 2002; Roberts et al., 2003; Bach et al., 2003; Lackschewitz et al., 2004). Many of the cores contain a late vein network filled with anhydrite plus accessory pyrite. These veins, measuring from less than a millimeter to several centimeters in width, commonly have complex pyrophyllite–silica halos, and the anhydrite crystals themselves are complexly zoned, with crude zones of cloudy and clear anhydrite in transmitted light, and multiple finely divided growth zones visible by cathodoluminescence.

The current seafloor hydrothermal activity at PACMANUS ranges from warm diffuse flow at a site called Snowcap (with temperatures measured by submersible of 6–65 °C) to vigorous black-smoker activity at Roman Ruins (measured temperatures up to 276 °C) (Binns and Scott, 1993; Binns et al., 1996). Anhydrite veins with fluid inclusions from the subsurface core samples provide evidence of fluids varying from about 150 °C (the lower temperature limit for anhydrite precipitation from seawater) to about 385 °C (Vanko et al., 2004). Primary fluid inclusion assemblages indicate that the solutions periodically boiled (Vanko et al., 2004). While the majority of inclusions contain aqueous fluid of near-seawater salinity, there are also numerous examples of both very dilute [T_m(ice) as high as 0.0 °C] and very saline (saturated with respect to halite at room temperature) fluids.

The origin, characteristics, and significance of the anhydrite-hosted fluid inclusions are discussed in detail by Vanko et al. (2004). However, in order to extract valid microthermometric data from anhydrite-hosted inclusions, it is necessary to recognize the effects of stretching and gypsum formation during the heating and freezing experiments, respectively. Measurements of ice melting temperature when gypsum is present, and measurements of homogenization in inclusions that have been overheated and stretched may lead to erroneous interpretations of primary hydrothermal conditions.

4. Results

4.1. Visible changes as a result of freezing runs and their cause

The most obvious change in anhydrite-hosted fluid inclusions occurs during and after freezing runs. Over the course of several cycles of freezing and heating, typically between around −50 and 0 °C, many inclusions develop prismatic crystals and/or granular ornamentation on their walls (Figs. 1 and 2). When specimens that have been frozen are left overnight, it is common for more crystal growth to occur and for more inclusions to display new crystal growth. The new crystals' volume is frequently too large to be explained by pure precipitation from the low-salinity fluid (e.g., Fig. 1). A better hypothesis to explain a new phase within or on the walls of an anhydrite-hosted aqueous inclu-
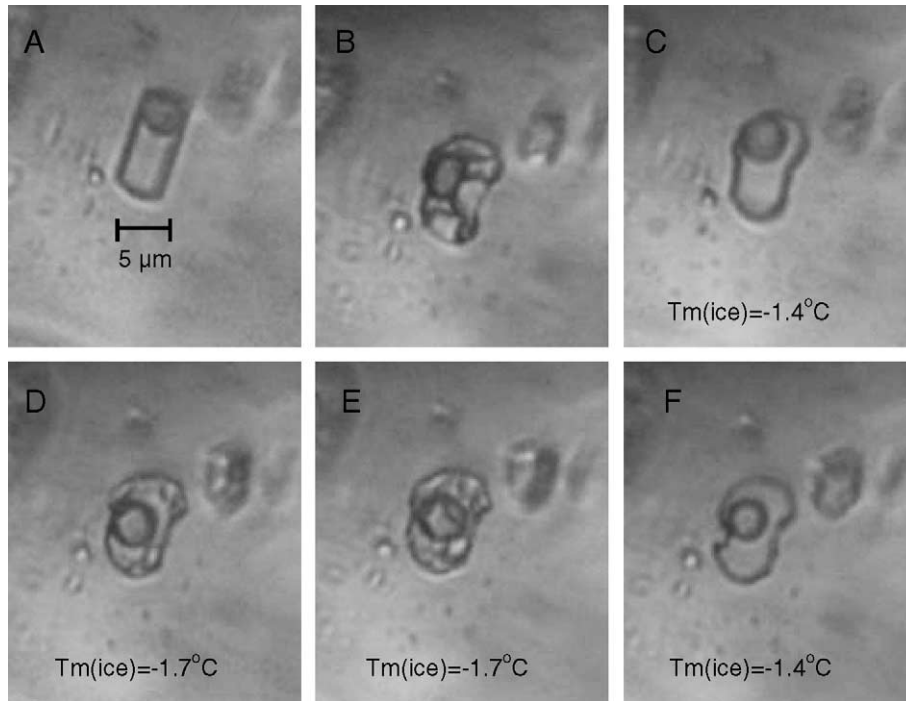
Fig. 1. Sequential room-temperature photomicrographs of inclusion # 25 from sample ODP193-1188A-16R-2 (Piece 15, 109–111 cm), illustrating the changes that took place during freezing and heating runs. (A) Inclusion # 25 prior to any microthermometry. (B) After a freezing run (during which numerous other inclusions were studied), inclusion # 25 is observed to contain newly formed gypsum crystals. (C) On heating, the gypsum crystals disappeared and T_h was measured at 284 °C. After cooling, T_m(ice) was measured at -1.4 °C. (D) After more of the freezing run, new gypsum crystals formed, and T_m(ice) was measured at -1.7 °C. (E) One day later T_m(ice) was re-measured at -1.7 °C. (F) After heating to 130 °C the gypsum disappeared, and a new measurement of T_m(ice) gave the original -1.4 °C. Note how the inclusion shape has been modified significantly relative to the initial shape.
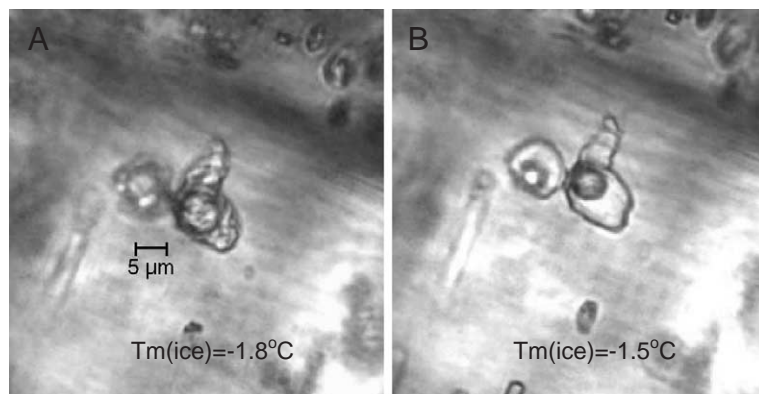

Fig. 2. (A) Inclusion # 36 from sample ODP193-1188A-16R-2 (Piece 15, 109–111 cm) photographed at room temperature after a series of freezing runs. The inclusion bears the telltale graininess that hints at the presence of newly formed gypsum. T_m(ice) was measured at -1.8 °C. (B) After heating to 145 °C the same inclusion appears clear and gypsum-free at room temperature. A new measurement of T_m(ice) gave -1.5 °C, which is considered to be the temperature yielding the "true" fluid salinity.

sion is that gypsum, the stable low-temperature hydrate (Fig. 3), nucleated and grew according to:

$$CaSO_4 \text{ (anhydrite)} + 2H_2O \text{ (aqueous)} \rightleftharpoons CaSO_4 \cdot 2H_2O \text{ (gypsum)}$$

(1)
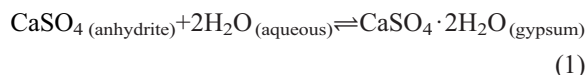
Bassanite, the hemihydrate [$2CaSO_4 \cdot H_2O$], is an intermediate phase between gypsum and anhydrite that may occur in arid soils (e.g., Azam, 2003), evaporites (e.g., Douglas and Yang, 2002), and even in the September 11, 2001 World Trade Center dust (Clark et al., 2001). However, at low to moderate temperature (e.g., 40 °C) and high water activity, bassanite is about twice as soluble as gypsum and anhydrite, so it is not a stable phase in Fig. 3 and is probably not the hydrate observed in the fluid inclusions.
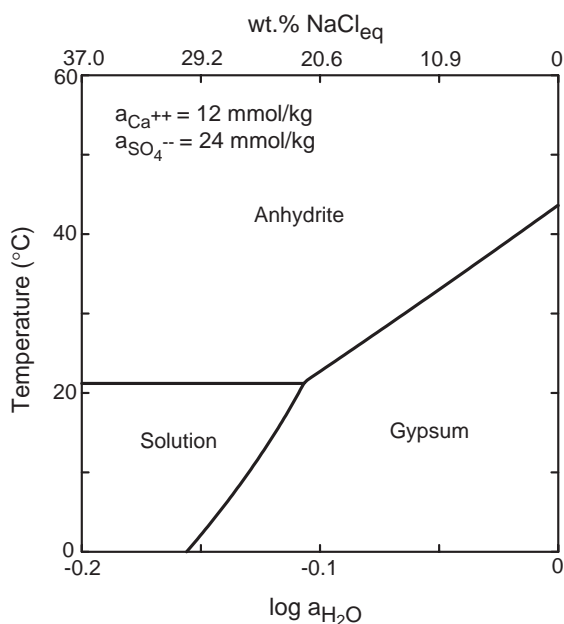

Fig. 3. The relative stability of anhydrite and gypsum as a function of temperature and water activity; the activities of Ca^{+2} and SO_4^{-2} are fixed at the values shown. Decreasing temperature favors the hydration of anhydrite to gypsum. However, at very low values of water activity (because of very high salinity, for example) both anhydrite and gypsum may be undersaturated. The diagram was constructed using Geochemist Workbench 4.0 (Bethke, 1996) and the thermo.dat database. Pressure is 1 bar. Note that the effect of pressure is to increase in the anhydrite–gypsum transition temperature by 1 °C per 80 bars (Blount and Dickson, 1973).

These same mineral stability considerations predict that gypsum, newly formed at low temperature, should decompose to anhydrite and water at high temperature (Fig. 3). Tivey et al. (1998) reported that gypsum in their inclusions typically dissolved between 108 and 120 °C. In the present study, the new gypsum crystals disappeared between about 120 and 130 °C. Some of the crystals shifted in position as they shrank and disappeared, indicating that to some extent the gypsum crystals are within the fluid inclusions and not just embedded in the walls. The equilibrium temperature of gypsum decomposition varies as a function of the activities of water, calcium and sulfate (e.g., Fig. 3). However, the experimental decomposition temperatures noted by Tivey et al. (1998) and in this study appear to be quite a bit higher than equilibrium predictions, suggesting a kinetic control on decomposition, at least at the fairly rapid heating rates of 10–20 °C per minute that were used.

The observed growth and decomposition of gypsum provoked several questions. If gypsum grows by hydration of the host mineral, then how severely might that affect the fluid salinity and, consequently, the measured ice melting temperature? If freezing runs are performed prior to heating runs, does the growth and decomposition of gypsum have any effect on the inclusion's homogenization temperature? Finally, how much overheating of the fluid inclusions beyond their homogenization can be tolerated before the inclusions stretch (e.g., Bodnar and Bethke, 1984)?

4.2. Test #1 — does T_m(ice) decrease with gypsum growth?

If gypsum replaces anhydrite, then the residual fluid in the inclusion must become more saline. One way to test this is to repeat measurements of the ice melting temperature (the freezing point depression) before and after the crystals have appeared. For example, the inclusion in Fig. 1 has T_m(ice) = −1.4 °C when no gypsum is present, but T_m(ice) = −1.7 °C with the gypsum crystals. The one in Fig. 2 had T_m(ice) = −1.8 °C when first determined with gypsum crystals, but T_m(ice) = −1.5 °C after the gypsum was cleared by heating. Several other examples are documented in Table 1. The largest shift in T_m(ice) was 1.5 °C, corresponding to a salinity increase from 1.4 to 3.9 wt.% NaCl equivalent. These results demonstrate

Table 1
Examples of observed changes in ice melting temperature and inclusion salinity as a result of the growth of gypsum during freezing runs

Sample and inclusion number	Highest T_m(ice)	Lowest T_m(ice)	ΔT	Original salinity	Highest salinity	ΔSalinity	ΔSalinity (rel. %)
ODP193-1188A-15R-1, piece 4, 14–20 cm							
Incl. 13	− 0.8	− 2.3	1.5	1.40	3.87	2.47	177
Incl. 14	− 2.2	− 2.3	0.1	3.71	3.87	0.16	4
Incl. 15	− 1.9	− 2.1	0.2	3.23	3.55	0.32	10
Incl. 16	− 1.3	− 1.9	0.6	2.24	3.23	0.99	44
ODP193-1188A-16R-2, piece 15, 109–111 cm							
Incl. 11	− 1.7	− 2.2	0.5	2.90	3.71	0.81	28
Incl. 25	− 1.4	− 1.7	0.3	2.41	2.90	0.49	21
Incl. 29	− 0.2	− 0.3	0.1	0.35	0.53	0.18	50
Incl. 36	− 1.5	− 1.8	0.3	2.57	3.06	0.49	19
ODP193-1189B-14R-1, piece 17, 123–129 cm							
Incl. 1	− 1.8	− 2.1	0.3	3.06	3.55	0.48	16
Incl. 2	− 2.2	− 2.6	0.4	3.71	4.34	0.63	17
Incl. 3	− 1.3	− 1.6	0.3	2.24	2.74	0.50	22
Incl. 4	− 1.5	− 1.8	0.3	2.57	3.06	0.49	19
Incl. 5	− 1.3	− 2.0	0.7	2.24	3.39	1.15	51

Note: *T* in °C; salinities in wt.% NaCl equivalent, determined using the equation of Bodnar (1993).

that the growth of the new crystalline material coincides with a decrease in the ice melting temperature corresponding to an increase in the fluid salinity, essentially confirming that the newly formed crystalline material is the hydrate gypsum.

If gypsum growth fixes water molecules and leaves a residual more-saline fluid in the inclusions, then gypsum decomposition on heating may release the same amount of water to the inclusion, diluting the fluid to its original salinity. To test this, ice melting
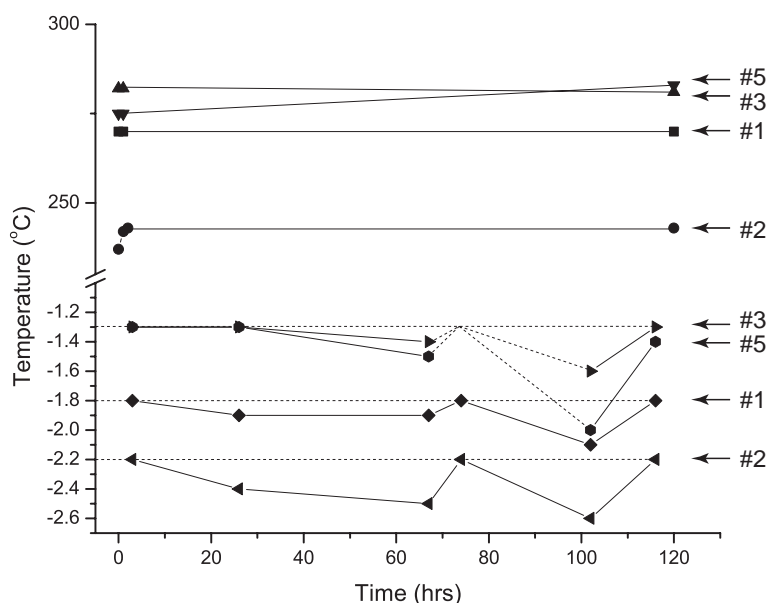

Fig. 4. Values of T_m(ice) and T_h determined and then redetermined for four inclusions over the course of about 120 h, sample ODP193-1189B-14R-1 (Piece 17, 123–129 cm). Two cycles of gypsum growth as a result of freezing and gypsum decomposition as a result of heating were carried out. The inclusions were heated to destroy the gypsum at about 72 and 115 h. The original T_m(ice) values are reliably determined even after multiple cycles of gypsum growth. Similarly, three out of four inclusions gave the same T_h after multiple cycles of gypsum growth.

temperatures were determined before and after gypsum growth, and then again after heating and decomposition of the gypsum. To illustrate this, four inclusions were studied in detail through several cycles of gypsum growth and decomposition over the course of about 120 h (Fig. 4). Only one inclusion failed to give exactly the original T_m(ice) value, and that one value was only 0.1 °C low, within the uncertainty of the microthermometric technique. Consequently, heating to the point of gypsum decomposition releases water quantitatively back into the fluid inclusion. This was also observed by Tivey et al. (1998), who reported that T_m(ice) values were recoverable to ± 0.1 °C.

4.3. Test #2 — does T_h change as a result of gypsum growth?

Gypsum formation during repeated freezing runs and its decomposition on heating in some cases results in dramatic shape modification of the fluid inclusions (Fig. 5; see also Fig. 1). Nevertheless, the modified inclusion should have the same volume as the original inclusion, and therefore its homogenization temperature should be unchanged. Homogenization temperatures of the four inclusions in Fig. 4 tend to confirm this hypothesis. Inclusion 2 homogenized at 237 °C. On further heating, inclusions 1, 5 and 3 homogenized sequentially at 270, 275, and 282 °C. Having been overheated considerably, inclusion 2 then exhibited a new T_h of 243 °C (i.e., the inclusion stretched—see below).

About 120 h later, after several cycles of freezing and heating (Fig. 4), the homogenization temperatures were determined again. Two were exactly the same, one was 1 °C lower, and a fourth was 8 °C higher. There was no obvious difference in the shape, size, or other aspects of the latter inclusion, so the cause of the increase in T_h is impossible to determine. Some stretching may have been caused by the repeated production of ice—in other freezing runs a small percentage of these anhydrite-hosted inclusions decrepitate or obviously leak simply as a result of the freezing. The conclusion is that the majority of inclusions subjected to freezing runs, even if gypsum has grown and decomposed several times, still yield reliable homogenization temperatures.

4.4. Test #3 — does T_h change simply as a result of overheating?

Observations were made to determine whether inclusions were stretched when heated above their homogenization temperatures. For example, five inclusions were photographed and then heated to their homogenization temperatures, which varied from 136 to 280 °C (Fig. 6). After cooling to room temperature new photographs show that one inclusion decrepitated, the one inclusion that homogenized at 280 °C appears unchanged, and the remaining three inclusions all appear to have stretched. Judging from the amount of vapor bubble expansion, the degree to which each inclusion was overheated correlates with the amount of stretching (Fig. 6). Inclusion 2 in Fig. 4
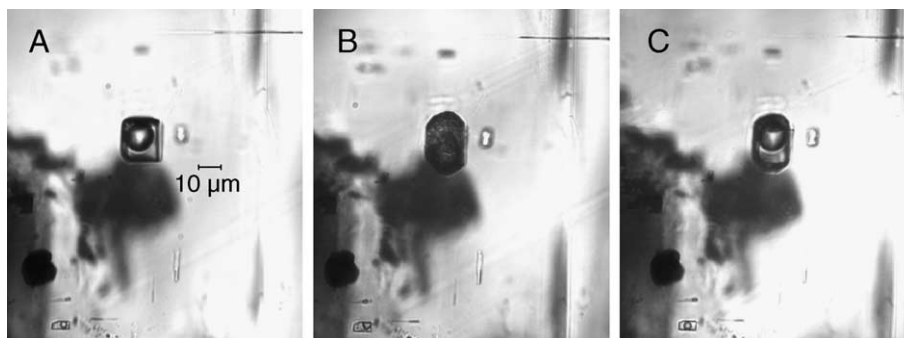

Fig. 5. (A) Inclusion # 3 from sample ODP193-1188F-26Z-1 (Piece 4, 62–69 cm) photographed at room temperature prior to microthermometry. (B) The same inclusion after freezing runs, and photographed several days later at room temperature. Gypsum growth on the inclusion walls and shape modification are apparent. (C) The same inclusion photographed at room temperature after it was heated to greater than 130 °C, which caused the gypsum to disappear. Note that the inclusion shape was strongly modified.
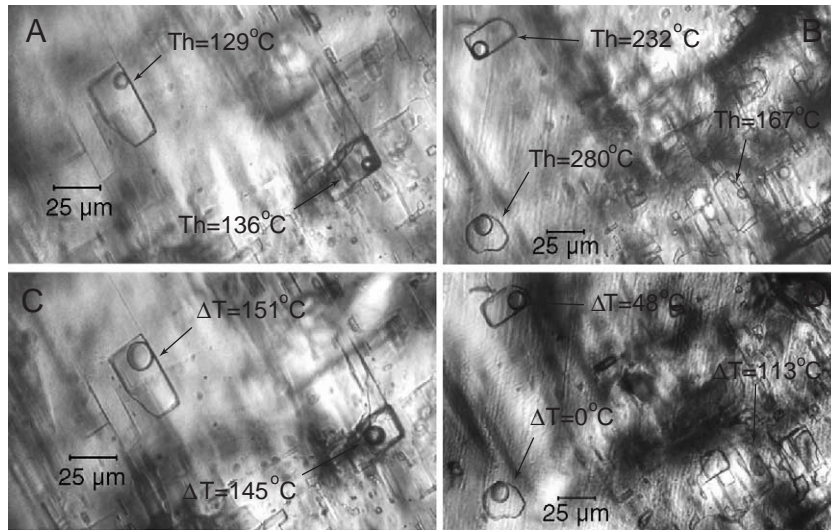
Fig. 6. Sequential photomicrographs taken at room temperature illustrating the stretching of inclusions that were heated as little as a few tens of degrees above their homogenization temperature. Sample ODP193-1188A-7R-1 (Piece 12, 66–68 cm), inclusion # 1 through 5. (A) and (B) show five inclusions prior to heating. The inclusions were then heated, and they are annotated with T_h values, determined in sequence as T increased. (C) and (D) show the inclusions after they cooled to room temperature. The highest T reached was 280 °C; the inclusions are annotated with the degree to which they were overheated. In each inclusion except the one that was not overheated ($\Delta T = 0$ °C), the vapor bubble has expanded, indicating that the inclusion has stretched. One inclusion ($\Delta T = 113$ °C) decrepitated.

is another example. Having been overheated by 45 °C, the original T_h value of 237 °C increased to 243 °C.

These results indicate that, indeed, just a few tens of degrees of overheating can result in irreversible deformation of the inclusions. This demonstrates that anhydrite hosts are relatively weak and deformable, and that heating measurements must be made in such a way as to record homogenizations progressively with increasing temperature.

5. Discussion

5.1. Gypsum growth

Microthermometric studies can yield valuable data, which, if properly interpreted, provide critical constraints on the chemical and physical properties of the fluids present during mineral growth or some later event (Roedder, 1984). Most inclusion studies justifiably ignore the effects of post-entrapment fluid–host interactions because of the insignificant solubility of the host mineral (commonly quartz). In some cases, though, reactions between fluids and the host mineral cannot be ignored. For example, aqueous-carbonic

inclusions in wollastonite can back-react at lower temperature to produce calcite and quartz (Heinrich and Gottshalk, 1995). The phenomenon described in this paper is a similar type of back-reaction which occurs as hydrothermal fluid, trapped in an anhydrite host, reacts at low temperature to produce gypsum, the stable low-temperature hydrate.

Tivey et al. (1998) also noted gypsum growth, frequently about 12 h or so after freezing runs in anhydrite-hosted inclusions. They considered two possible explanations. One was that gypsum is a reaction product of anhydrite with fluid. But Tivey et al. (1998) discounted this explanation because they expected that if this were happening, they would have obtained higher salinities from the smallest inclusions (a surface area to fluid volume effect), and they did not. The second possibility Tivey et al. (1998) considered was that the inclusions at room temperature may have been supersaturated with respect to gypsum owing to dissolution of anhydrite upon cooling from the trapping conditions near 350 °C. The small volume of gypsum daughter crystals observed by Tivey et al. (1998) may be consistent with growth out of solution only. They suggested that the freezing runs might have promoted the nucleation of gypsum,

which subsequently grew in slowly over several hours.

Results from the present study suggest that hydration of anhydrite must be invoked to explain the gypsum. Successive cycles of freezing and heating may result in variable depressions of the ice melting temperature (and variable increases in salinity) within the same inclusion (e.g., Fig. 4), suggesting that the amount of salinity increase is a function of time and thermal history, not inclusion size. The inclusions may be supersaturated with gypsum as a consequence of anhydrite dissolution, but the bulk of the salinity changes observed in this study are best explained by variable degrees of hydration. The volume of gypsum in many cases appears to be much greater than could be explained by precipitation from a supersaturated solution. But hydration of the inclusion walls might easily produce large volumes of gypsum, particularly considering the fact that the hydration reaction involves a 60% volume increase—anhydrite has a molar volume of 46.0 cm^3/mole, while that of gypsum is 74.7 cm^3/mole (Robie and Hemmingway, 1995). Fig. 7 illustrates the relationship between the amount of hydrate formed and the residual fluid salinity. The calculation assumes a spherical inclusion and a homo-
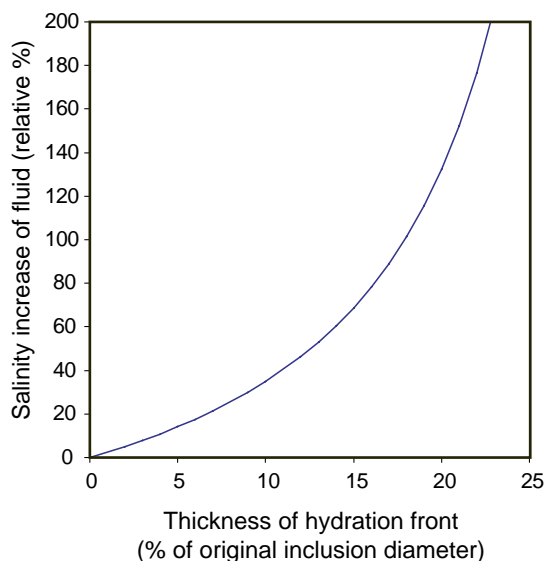
geneous hydration shell. A salinity increase of 177% relative, which is the largest increase documented in Table 1, would be achieved if the hydration front's thickness equaled 22% of the diameter of the original inclusion. The inclusions in Figs. 1 and 2 exhibited salinity increases of 21 and 19 relative %, respectively. This degree of salinity increase would correspond to a hydration shell thickness equal to 6% or 7% of the inclusion diameter. The actual appearance of gypsum in these inclusions is clearly not a homogeneous shell (Figs. 1 and 2). Instead, a relatively small number of gypsum nuclei apparently formed. It is extremely difficult, though, to determine whether the volume of gypsum that did form is equivalent to the calculated volume.

The reaction to gypsum occurs during freezing experiments — none of the specimens from PACMANUS appear to have nucleated gypsum prior to freezing runs, even though gypsum is the stable phase at room temperature. These samples were extracted from boreholes where they were presumably still quite warm (after drilling with cold seawater, the temperature at a depth of 360 m in one borehole eight days later had rebounded to 313 °C), and their residence time at room temperature has been less than three years. It is not known how prolonged a time period at room temperature might be needed to allow gypsum to nucleate, but supercooling to about -50 °C in the freezing stage frequently produces gypsum.

As noted above, gypsum formation in anhydrite-hosted inclusions has been described before. Tivey et al. (1998) showed that precipitation of gypsum from a supersaturated solution results only in minor salinity changes. The present study, though, demonstrates that the ice melting temperature and, correspondingly, the fluid salinity may shift dramatically as a result of anhydrite hydration (Table 1). The consequence for fluid inclusion interpretations is that these lower T_m(ice) values and the corresponding high salinities must be rejected as poor representations of the original fluid. When gypsum formation occurs during freezing experiments, it needs to be dehydrated periodically through heating to about 120 to 130 °C.

Fig. 7. The relative salinity increase [100*(new salinity − initial salinity)/initial salinity] calculated within a spherical fluid inclusion owing to the development of a homogenous hydration front of gypsum.

5.2. Stretching

Stretching as a result of heating beyond the homogenization temperature is common, particularly in soft

minerals (Bodnar and Bethke, 1984). With a Moh's hardness of 3 to 3.5, inclusions in anhydrite are expected to stretch much like those in calcite ($H = 3$) or barite ($H = 2.5$ to 3.5) (Bodnar, 2003). Examples from the present study show that overheating just a few tens of degrees is sufficient to cause irreversible deformation of the host. Consequently, heating data must be collected sequentially. It is recommended that for every measurement, the inclusion being observed must not have been overheated by more than about 10 °C. Researchers reporting the results of fluid inclusion studies in anhydrite hosts should verify that precautions against both overheating and stretching were taken. **[CS]**

Acknowledgements

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