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Solid Inclusion Piezothermometry I: Comparison Dilatometry

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Abstract

Solid inclusion piezothermometry is a technique for determining the pressure and temperature of inclusion of one mineral in a host mineral. Application of the technique requires knowing the pressure-temperature curves of constant difference in natural strain between two crystallographically oriented mineral rods. For this purpose, two different kinds of comparison dilatometer, designed for use in an internally-heated high pressure gas apparatus, have been developed and are described in detail. Each design offers advantages and disadvantages in fabrication, demand upon size of sample material, and versatility.

The comparison dilatometers have been used at pressures up to 7 kbar simultaneously with temperatures up to that of the low-high quartz transition. Data obtained have been cross-checked successfully between the two kinds of comparison dilatometer and between results from them and previously-determined physical properties of synthetic periclase, synthetic halite, and quartz. As a test of accuracy, results from one design of comparison dilatometer were compared with predictions for periclase *vs* halite for the pressure range up to 7 kbar and temperatures up to ~600°C. The maximum deviation between experimental results and prediction was ~6°C at any pressure. Such accuracy is more than sufficient for developing data for use in solid inclusion piezothermometry and may be of value in determining equations of state.

Introduction

In reconstructing and analyzing the geologic history of an area, a frequent need has been data on the pressure and temperature of an igneous or metamorphic process. Solid inclusion piezothermometry is a procedure for determination of a pressure and temperature of crystallization of a rock by taking advantage of elastic effects in host minerals around mineral inclusions (Rosenfeld and Chase, 1961; Rosenfeld, 1969; Adams, 1971; Cohen, Rosenfeld, and Adams, 1972) or in the inclusions themselves (Harris *et al*, 1970). As currently pursued by us, the method, stripped of details, uses the following strategy:

(1) From a thin section of a given rock, small portions containing appropriately-oriented host inclusion pairs are separated from the slide and subjected to adjustment of pressure (P) and temperature (T) using experimental apparatus; a P and T is found for each pair that just causes the disappearance of the optical effect in the host due to deviatoric stress.

(2) The investigator then refers to sets of *isomekes* (Gr. "equal" + "length"), curves previously determined using separate pieces of host and inclusion minerals. Isomekes are $P - T$ curves along each of which the distance between two reference points

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embedded in the host mineral remains equal to that between two reference points embedded in the inclusion mineral. In Part II of this series (Adams *et al*, 1975), isomekes are shown to be equivalent to curves of constant difference in natural strain between the two minerals in specific crystallographic directions. For each mineral pair, that particular isomeke is selected which incorporates values of the variables determined in the first operation. Barring complications, that isomeke will pass through the P and T at which the inclusion was incorporated in the host. The intersection of independent isomekes for two or more different pairs should be T_f and P_f, the temperature and pressure of crystallization, respectively.

In this first of two related papers, we detail the design, testing, and limitations of the comparison dilatometers that were built to yield direct determination of isomekes. The second paper of this series gives a theoretical treatment of solid inclusion piezothermometry, the calibration for quartz included in garnets (or the converse), and application of this calibration to conditions of crystallization in selected metamorphic terrains with particular reference to the Al_2SiO_5 triple point.

Comparison Dilatometry

We have developed two basically different types of comparison dilatometer. Both monitor isomekes in an internally heated argon apparatus and are designed in such a way that completion of an electrical circuit indicates presence on the isomeke. Our designs differ from that of Bridgman (1949, p. 194) and many others in that no slidewire or equivalent electrical system is used.

One-Contact Dilatometers

These dilatometers are formed by clamping together pieces of the two minerals to be compared so that the effects of suitable change in P and T will cause a small, preset gap to close. Electrically conductive coatings on the two minerals allow current to flow upon contact and this indicates presence on a particular isomeke. Electrical contact can be broken only by returning to the *same side* of the isomeke as that of the starting condition.

Description of "J" Dilatometer. This dilatometer (Fig. 1), developed by Adams (1971, p. 26–28), was used in the work reported by Adams, Cohen, and Rosenfeld (1970). Even though superseded by the more easily constructed "opposed rods" dilatometer discussed later, it is illustrated here because it is the first dilatometer used to determine isomekes and because five experiments by Adams *et al* (1975) were carried out with it. The "J" dilatometer consists of two elongate pieces machined from oriented samples of the crystals to be compared. The upper crystal, or "rod", is clamped to the lower riser of a crystal shaped into a vertically flattened "J", the "base". The device is converted into an electrical switch by evaporating a thin metal coating onto the top and free end of the rod and onto the base along one side and opposite the free end of the rod. Attachment of electrical leads from a monitor outside the argon apparatus to the coating on top of the rod and on the side of the base completes the circuit.

The rod, which has the cross-sectional shape of an isosceles right triangle, is guided by a complementarily shaped right angle groove in two supports. The rod is clamped to the base by a U-shaped spring of tungsten wire constrained by a centered longitudinal groove in the bottom of the base. So that one of these supports will serve as a fiducial marker for subsquent strains of rod and base, the clamping force of the tungsten spring is positioned near that support. Thus the relatively high frictional force prevents sliding of the rod relative to that support. Motion of the free end of the rod relative to the base thus is only a function of strain of the rod along its length between the free end and the clamped position.

One electrical lead is attached to the tungsten clamp. The clamp, in turn, makes electrical contact with the metal coating atop the rod by means of a conductive paint or by a roller bearing made of stainless steel hypodermic tubing. The other electric lead terminates in a groove on the side of the base

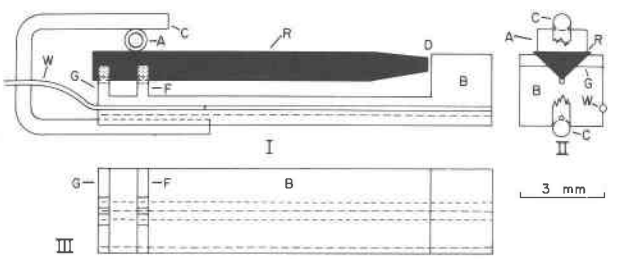

FIG. 1. Scale drawing of "J" dilatometer. I. Side view. Electrical lead to clamp (C) and insulation not shown. II. View from clamp end of dilatometer. III. View from top showing base (B) only. Letters designate the following components: A, roller bearing that is in electrical contact with metallic coating atop rod; B, base, one of minerals being investigated; C, tungsten wire that clamps rod to fiducial ridge in base; D, electrical contact, formed when metal coating at end of rod touches similar coating on upright part of base opposite; F, fiducial ridge; G, guiding support; R, rod, other mineral being investigated; W, other electrical lead, bonded with metallic contact cement to metallic coating on side of base.

and makes electrical contact with the metal coating on the base via conductive paint.

Description of "Opposed Rods" Dilatometer. To eliminate some of the difficulties associated with the "J" dilatometer, a variant, the "opposed rods" dilatometer (Fig. 2) was developed. The longitudinal 90° groove in the "opposed rods" dilatometer is through-going and is easily ground with a shaped grinding wheel. This permitted construction of narrower but simultaneously less fragile fiducial ridges for the rods by using an Airbrasive® tool and masking technique. Narrowness of fiducial ridges ($\approx$ 100 μm) contributes to precision, but this improvement is partly offset by error associated with the presence of the fiducial ridge for the second rod. The electrical circuit is similar to that of the "J" device.

This device also has the virtue of utilizing more readily available smaller pieces of mineral for both the rods and the base. Although vacuum-deposited conductive coatings are still necessary on the tops and contacting parts of rods, this design eliminates dependence on conductive paint, at best a somewhat unreliable component of electrical circuitry. A roller bearing is kept near each fiducial ridge so as to maintain high frictional resistance to sliding between rod and fiducial ridge but low frictional resistance at the free end.

Experimental Procedure. An isomeke is determined for a particular setting of the gap by noting the set of $P-T$ points for which a voltage source just causes a detectable current to pass through the device (that is, the switch is closed). Normally an isomeke is determined with this type of device by slowly changing, say, temperature until the circuit is just closed, then *immediately* reversing the sense of temperature change so as to break the circuit before force builds up at the electrical contact and causes a rod to reset by sliding at the clamped support(s). The pressure is then changed and the previous operation repeated to locate another point on the isomeke, and so on. Figure 3 is part of a strip chart record that shows a typical example of isomeke determination.

Distinction is made between data points obtained on the P-T path going away from starting conditions and those obtained on the return path. Reproducibility is necessary for a run to be accepted. This test was applied to all dilatometers described here.

For this kind of dilatometer, the quality of coating and its adhesion to mineral surfaces is critical. For example, experimental data for "make contact" and "break contact" had to be treated separately until improved coating techniques rendered the distinction unnecessary.

The dilatometer is reset to determine a new isomeke by changing the gap width γ between the free end of the rod (r) and the corresponding part of the base (b) in the "J" device, or between the free

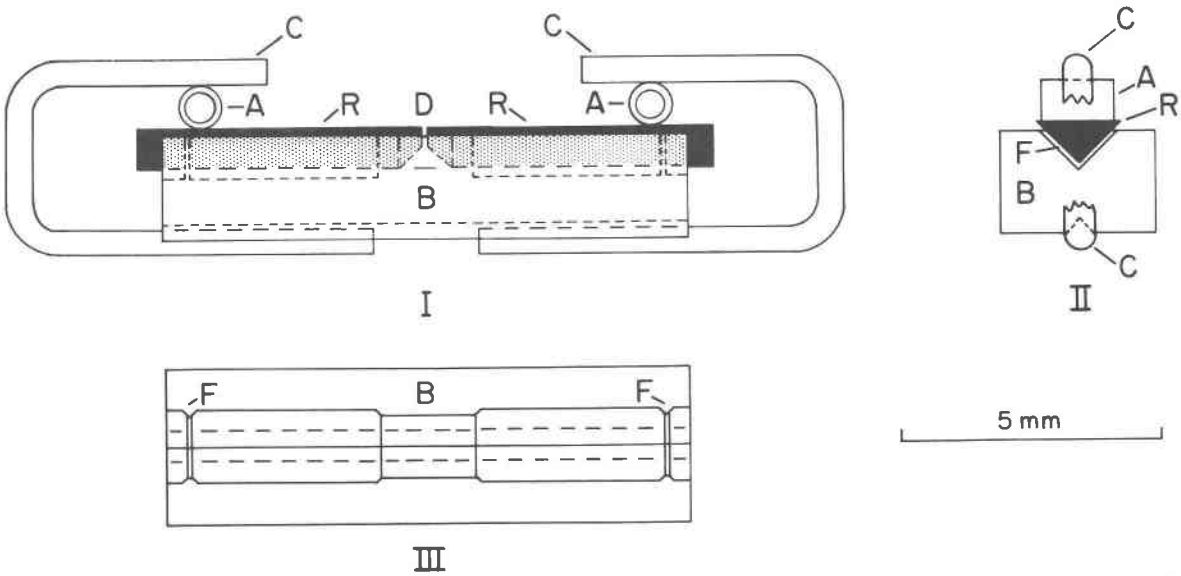

FIG. 2. Scale drawing of "opposed rods" dilatometer. I. Side view. Two insulated electrical leads, not shown, are attached to clamps (C). II. End view. III. View from top showing base (B) only. Letters designate the following components: A, roller bearings that are in electrical contact with metallic coatings atop rods; B, base, one of minerals being investigated; C, tungsten wires that clamp rods to fiducial ridges in base; D, electrical contact, formed between coatings at ends of rods; F, fiducial ridges; R, rods, other mineral being investigated. Thermocouple (not shown) is placed near dilatometer during experiment.

ends of the rods in the "opposed rods" device. This adjustment of the position of the rod(s) is normally done under a microscope at room conditions (designated by subscript o). γ is estimated from

$$\gamma \equiv l_{bo} - l_{ro} \approx [(\beta_{bo} - \beta_{ro})(P_1 - P_o) - (\alpha_{bo} - \alpha_{ro})(T_1 - T_0)]l_{bo} \quad (1)$$

where l_{bo} and l_{ro} are the lengths of mineral being compared, α is the linear coefficient of thermal expansion, β is the linear coefficient of compressibility, and P_1 and T_1 are the P and T at which one desires to intercept the new isomeke.

It is evident from the nature of devices of this type, their convenient adjustment at ambient conditions, and the necessity that $\gamma \geq 0$ (compare Equation 1 above) that they permit determination of isomekes only in that part of P-T space on one side of the isomeke passing through ambient P and T. Isomekes in the other part of P-T space are attainable by construction of a congruent device in which the oriented material of rod(s) and base are interchanged.

Accuracy. As a check on accuracy of this type of dilatometer, a "J" device was made of quartz with the base oriented $\|$ c and the rod $\perp$ c. This combination was selected because of the existence of accurate data on α and β for quartz (McSkimin *et al,* 1965). Figure 4 shows data for runs projecting to the temperature axis at 31°C and 65°C.

The points for both runs conform rather closely to straight lines having a slope of 0.0360°C/bar below approximately 3 kbar. Using equation (7-II),[3] this slope almost exactly equals the 0.03605°C/bar calculated for 25°C and 1 bar using data for quartz from McSkimin *et al* (1965). Lines bracketing the calculated slope in Figure 4 represent a standard deviation of 0.0076°C/bar, calculated from analysis of error propagation (Bevington, 1969, p. 56–65) assuming conservatively estimated standard deviations $\sigma_\beta = 10^{-9}$ bar^{-1} and $\sigma_\alpha = 10^{-7}$ °C^{-1}. An estimate of the precision with which thermal expansion or compressibility in one direction, say $\|$ c, could be determined is obtained by combining the above estimate of standard deviation in slope with the same conservative assumptions for $\sigma_{\beta\perp c}$ and $\sigma_{\alpha\perp c}$. We obtain $\sigma_{\alpha\|c} = 0.18 \times 10^{-6}$ °C^{-1} and $\sigma_{\beta\|c} = 0.0074 \times 10^{-6}$ bar^{-1}.

Critique. These devices have the great advantage of simplicity of design; and some excellent data, such as those in Figure 3, have been obtained with them.

[3] Equations so labeled are from Part II of this series (Adams *et al,* 1975).

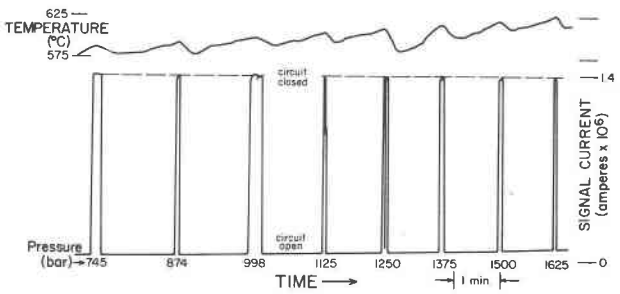

FIG. 3. Part of 2-pen strip chart record for determination of an isomeke between quartz (48° from c) and almandine using the "J" dilatometer. Corrected for offset of pens.

Their designs do not make strong demands on precision of machining. Unlike the design of Bridgman (1949, p. 194) and in common with our other dilatometers, possibility of hysteresis and other error due to "stick-slip" of sliding parts is reduced essentially to nonexistence. There are also difficulties. Construction of the base of the "J" dilatometer requires a rather large homogeneous crystal—a major objection when dealing with natural materials in which zoning and inclusions are commonly present.

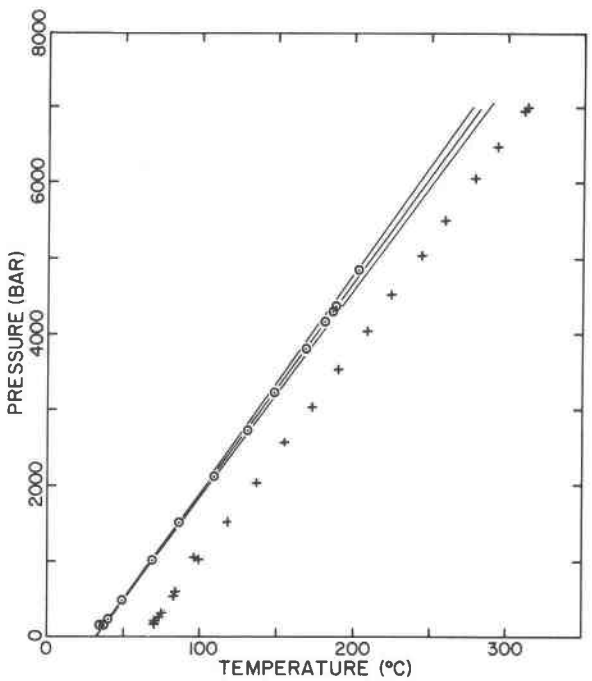

FIG. 4. Data points obtained with "J" dilatometer for determination of two isomekes between quartz $\perp$ c and quartz $\|$ c. The intermediate straight line associated with the set of experimental points denoted by circles was determined using Equation (7-II) and data at 25°C and atmospheric pressure from McSkimin *et al* (1965). Straddling lines indicate standard deviation of slope based on estimated standard deviations of properties used in Equation (7-II).

That dilatometer is not easy to construct, since the 90° groove in the supports, even though not demanding precision, is obtained by hand-lapping with a very short stroke. The fundamental asymmetry in the "J" dilatometer might also be increased by any temperature inhomogeneity within the high pressure apparatus. Scanning electron microscope examination shows that vacuum-evaporated metal coatings crumple when scratched, tear from pressure welding, and occasionally buckle after high temperature runs due to differential strain between coating and substrate or expansion of gas beneath the coating; such damage may cause nonrandom errors that are difficult to detect or evaluate. The lesser problem of resetting of the devices can be offset by careful experimental procedure. The earlier-mentioned necessity of making two devices of this type, interchanging materials of rod(s) and base, to explore all of *P-T* space is an added difficulty.

Two-Contact ('Gate') Dilatometer (Figure 5)

This dilatometer is formed by clamping rods of the two minerals to be investigated side-by-side to a nonreactive base. Only when both rods are of equal length do the two simultaneously make contact with a spring-loaded, hinged gate. Such contact completes a series electric circuit, and the flow of current indicates presence on a particular isomeke. Electric contact can be broken by *any P-T* change away from the isomeke.

Description. In the "gate" dilatometer, any condition of equidistance $l_x = l_y$ between fiducial ridges F_1 and F_2 in the base B, and the free ends of the rods R_1 and R_2, is indicated by completion of an electrical circuit. When the circuit (Fig. 7) is completed, *i.e.*, when on an isomeke, the electrical path from the voltage source is: strip chart recorder; chromel lead through closure piston to chromel terminal; gold wire

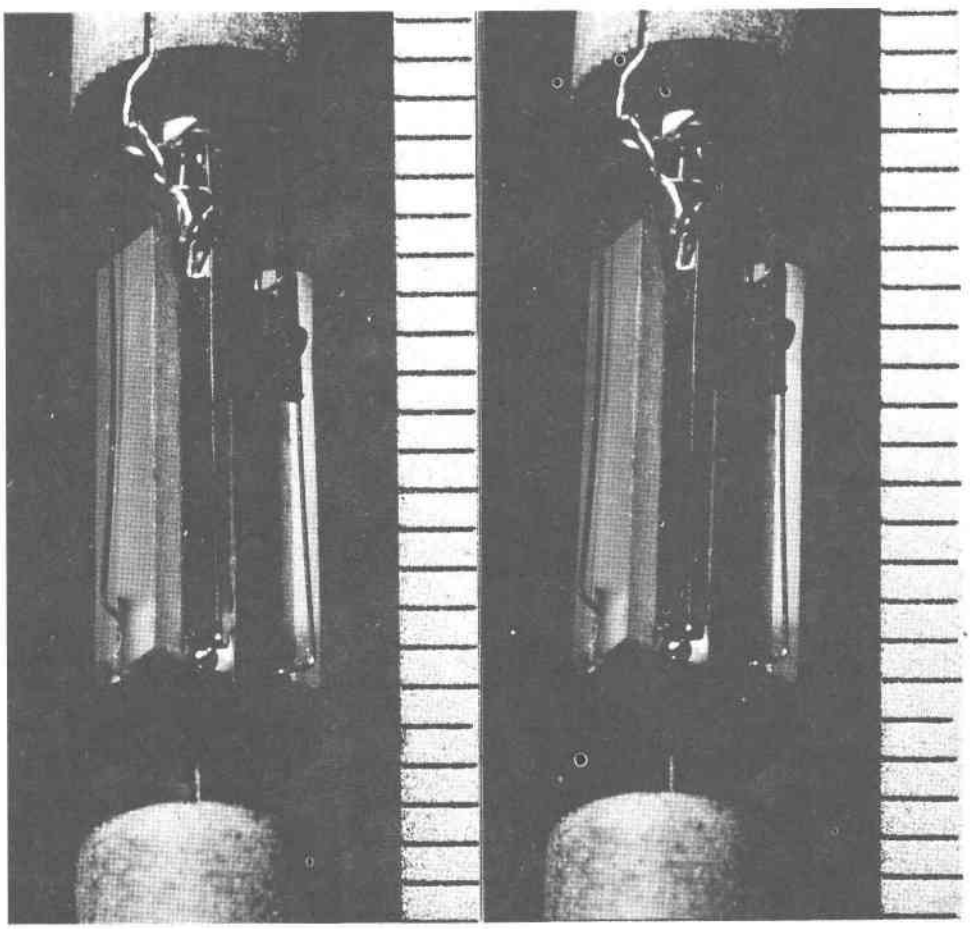

FIG. 5. Stereoscopic photograph of "gate"-type comparison dilatometer. Units on scale are millimeters. See Figure 6 for identification of parts.

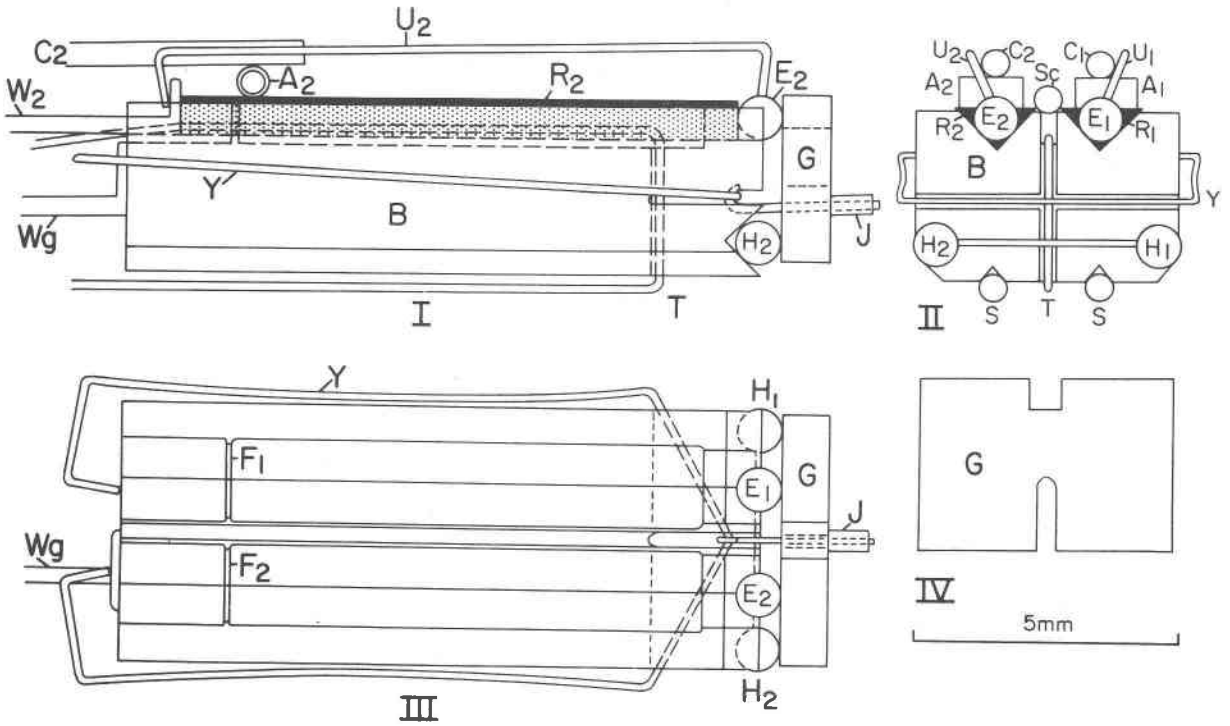

FIG. 6. Scale drawing of "gate" dilatometer. I. Side view, supports (S) removed for clarity. II. View from gate end with gate removed. III. View from top; some components removed for clarity. IV. Gate. Letters designate the following components: A_1, A_2–roller bearings; B–base; C_1, C_2–tungsten wires that clamp rods to fiducial ridge in base; E_1, E_2–electrical contact ball bearings; F_1, F_2–fiducial ridges; G–gate; H_1, H_2–hinge ball bearings; J–connection between gate and gate spring; R_1, R_2–rods of minerals being investigated; S–supports for dilatometer; T–thermocouple; U_1, U_2–spring clamps that keep electrical contact balls, E, in contact with rods, R, and also serve as part of the electric switch circuit; W_2–electrical lead from closure piece to electrical contact ball, E_2 (note: W_1 not shown); W_g–electrical lead, when connected, from gate to ground; Y–gate spring.

W_1 to clamped end of rod R_1; through W-Re alloy spring clamp U_1 to coated 0.7938 mm (0.03125 ± 0.00001 inch) stainless steel ball bearing E_1 that was welded to U_1 before coating; through polished, optically flat, tungsten carbide gate G; and then back to the voltage source along a path on the other side of the device, symmetrically equivalent except for absence of the recorder. The chromel-alumel thermocouple T (Figs. 6, 7) is placed in a longitudinal central groove in the base with the junction located symmetrically in the plane of the rods. The voltage developed by the thermocouple is charted by one pen of a two-channel recorder, and current through the electrical contact circuit is charted by the other pen. Lead W_g from the gate to ground *via* J and the gate spring Y is useful primarily for trouble-shooting malfunctions in electrical contacts.

Certain design features of this dilatometer merit attention. The position at which the gate is loaded by J lies just beneath the intersection of two hypothetical lines connecting the points of contact between E_2 and H_1, and E_1 and H_2. Except for the small deformation of rods, balls, and gate under the slight spring forces used, the method of loading the gate assures that there will be an electrical signal only when the centers of the four balls are coplanar. Under this circumstance, presence on an isomeke is assured, given sufficiently precise shaping of base, balls, and gate.

The five straight lengths of 0.508 mm diam. tungsten wire, S and C, support the dilatometer. Supports S terminate on both ends in segments of fired pyrophyllite rod that simultaneously hold the device, conduct electrical wires to the dilatometer, fill space inside the cylindrical furnace, and center an encapsulating cylinder of stainless steel with internally nested cylinder of gold. The high thermal conductivity of the last reduces thermal gradients in the vicinity of the dilatometer to negligible values. Springs C cause roller bearings A to pin the mineral rods to fiducial ridges F and also hold the dilatometer in place. The roller bearings are an important feature, since they also effectively decouple the dilatometer from flexural motions of the supporting assembly.

The base was machined from annealed, stoichio-

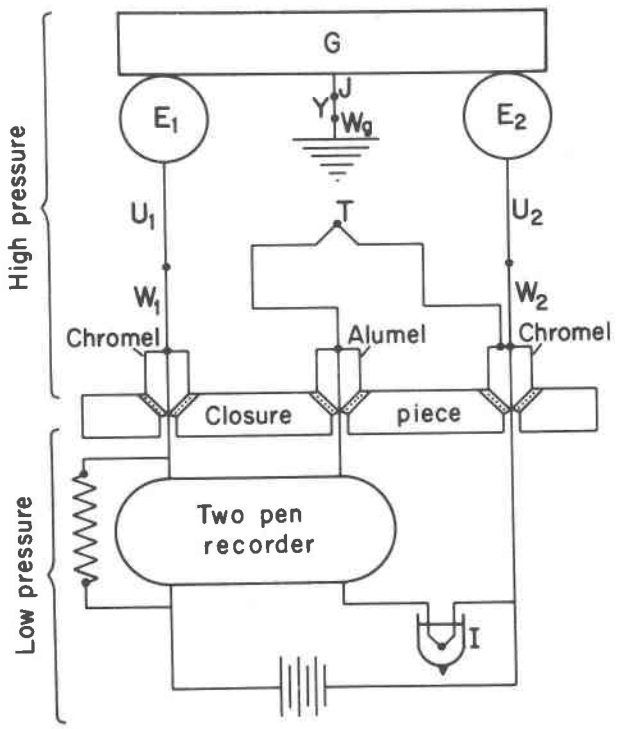

FIG. 7. Schematic diagram of electrical circuitry for the "gate"-type dilatometer. I is the ice bath for the cold thermocouple junction. Other lettered parts are same as in Figure 6.

metric spinel ($MgAl_2O_4$), manufactured using the Czochralski process by Union Carbide. It proved very satisfactory. Spinel was selected because of its high crystal symmetry, homogeneity, absence of any easily cleaved direction, high electrical resistivity, hardness, strength, low coefficient of thermal expansion, high density of packing of oxygens (to preclude possible problems from diffusion of argon at high pressure), and highly annealed character corresponding to maximum deviatoric stress $\approx 10^2$ bars (maximum birefringence $\approx 10^{-5}$). Wires of tungsten and various tungsten-rhenium alloys maintained their elasticity to the highest temperatures employed (~800°C in a dry argon atmosphere) and therefore proved satisfactory in construction of the supports and springs. Supports S and spring clamps C are made of pure tungsten wire, whereas slightly more ductile wire of $W_{95}Re_5$ or $W_{74}Re_{26}$ is used for parts that must be bent or welded—U, J, Y, and wire welded to H_1 and H_2. The stainless steel (440C) balls are manufactured by Winsted Precision Ball Company for ball point pens. To enhance durability under experimental conditions, the electrical contact balls E are coated on the contact side at 600°C in a vacuum evaporator, first with chromium, and next with $Pt_{80}Pd_{20}$ alloy. In spite of the coating, electrical contacts on the balls become pitted at higher temperatures; generally, a new set of electrical contact balls is used for each experimental run. In order to minimize deterioration of electrical contacts, the DC signal current is kept low, ~1.4 μA. Deterioration of electrical contacts presently limits the temperature range of accurate experimentation to approximately 650°C, although useful experiments have been performed to 800°C.

The contact side of the gate is optically flat, and the gate itself must be conducting, stiff, hard, not subject to warping, capable of taking and keeping a polish, and of low corrosivity. Gates of antimony-doped single crystal silicon yielded good results, but they rapidly decrease in their already low electrical conductivity during the course of experiments. We therefore employ very fine-grained tungsten carbide (Carmet 310) which, although it behaves well during a single run, must be polished and checked against an optical flat before starting each new run, since a fine powder forms on its polished surface upon removal to air.

Experimental Procedure. With the gate dilatometer the necessary and *sufficient* condition for presence on an isomeke is completion of the electrical circuit through the dilatometer. It is not necessary to be alert at the controls of the argon apparatus, as presence of the free-swinging gate normally precludes resetting. Figure 8 shows 10 minutes of recording from a run using this dilatometer. The finite, but small, width of the signal probably indicates a slight departure from rigidity within the dilatometer. This is perhaps due primarily to elastic flattening of balls rather than torsion of the gate, because variation in thickness of the gate does not noticeably affect the signal width. The signal width corresponds to a strain

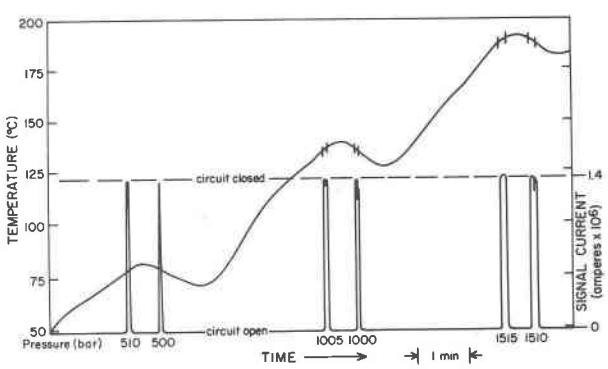

FIG. 8. Part of 2-pen strip chart record for determination of an isomeke between quartz $\perp c$ and spessartitic almandine using the "gate" dilatometer (WC gate). Corrected for offset of pens.

difference of the rods between 25×10^{-6} and 50×10^{-6}. In assessing precision, it is probably more significant to pay attention to the signal edge, where the "noise" indicates sensitivity to strain differences of $\sim 10^{-6}$, *i.e.*, length difference of ~ 100 Å. The "true" isomeke is presumably the *P-T* curve for which the two balls are equally flattened. This would probably be a curve intermediate between, and parallel to, the *P-T* curves for each of the signal edges. Since these curves are essentially parallel, there is little trouble in inferring the position of the "true" isomeke.

In each determination of an isomeke, the earliest *P-T* points are redetermined at the end of the run as a check against resetting or deterioration of electrical contacts. An important check before starting a run was to ascertain that the balls E were seated in the 90° grooves. Setting of the gap γ between one ball and the gate is based on Equation (1). The magnitude of γ determines δ_{x-y}, the natural strain difference, in conformity with (5-II):

$$\delta_{x-y} = \ln\left(1 + \frac{\gamma}{l_{xo}}\right) \approx \frac{\gamma}{l_{xo}}. \qquad (2)$$

In contrast with the "J" and "opposed rods" devices, γ may be either positive or negative, depending only upon arbitrary assignment of the labels "x" and "y" to the rods. Thus *all* of *P-T* space is accessible by mere adjustment of the rods. Setting gap widths of 0 μm to 20μm, using a machinist's microscope at ambient conditions, is not too difficult, since the permitted fractional error in setting is rather large. We were able to set gaps with a precision of 2 μm or better.

Accuracy. To give accurate results, the gate dilatometer makes greater demand on precision machining than do the "J" and "opposed rods" dilatometers, both of which adhere more closely to the principles of kinematic design (Wilson, 1952, p. 104). As well as having a number of parts, a basic reason for this demand is the dependence upon a longitudinal vertical plane of symmetry that maintains itself during experimentation. During development of the "gate" device, data from both kinds of devices were cross-checked, with results becoming concordant for the latest models.

For a test of accuracy of the "gate" device, we used independent standards whose relevant properties had been studied over a range of simultaneously elevated *P* and *T*. Figure 9 shows data for four isomekes for single crystal rods of synthetic periclase (MgO) and synthetic halite (NaCl). The solid lines are from numerical integration of Equation (7-II) using the α's and β's from second degree trend surfaces fitted to the values of these quantities for MgO (Spetzler, 1969, p. 151–171) and NaCl (Spetzler, Sammis, and O'Connell 1972, p. 1735–1737). Below 1 kbar the data points forming the curve having a 45°C intercept at 1 bar lie essentially on a straight line having a slope equal to that calculated from α's and β's at 25°C and 1 bar, using Equation (7-II). At 7 kbar the maximum departure in temperature is only ~6°C. The agreement between the two approaches is good. It thus appears that, given sufficient care, both kinds of comparison dilatometer can give isomekes that accord with those determined by independent means.

Although it is not the primary purpose of this paper to explore the suitability of comparison dilatometers for work on equations of state of solids, it is obvious that they may prove useful in this regard. To check further on accuracy, we determined isomekes for synthetic stoichiometric spinel (subscript *s*) against "standards" NaCl (subscript *h*) and MgO (subscript *p*) in order to determine α_s and β_s at 25°C and 1 bar. Synthetic spinel is currently undergoing investigation by R. J. O'Connell (personal communication), and preliminary information is available. Solution of two simultaneous equations of the form of (7-II) for *m*, the derivative of an isomeke, yields:

$$\alpha_s = \frac{(\beta_h - m_{h-s}\alpha_h) - (\beta_p - m_{p-s}\alpha_p)}{m_{p-s} - m_{h-s}} \qquad (3a)$$

and

$$\beta_s = \frac{m_{p-s}(\beta_h - m_{h-s}\alpha_h) - m_{h-s}(\beta_p - m_{p-s}\alpha_p)}{m_{p-s} - m_{h-s}} \qquad (3b)$$

From Equation (3) and expressions for error propagation based on estimated standard deviations, we get

$$\alpha_s = (5.86 \pm 0.17) \times 10^{-6}\ °C^{-1}$$

$$\beta_s = (0.1708 \pm 0.0022) \times 10^{-6}\ bar^{-1}.$$

O'Connell (personal communication), from ultrasonic measurements, obtains

$$\beta_s = (0.1689 \pm 0.0005) \times 10^{-6}\ bar^{-1}.$$

If we use O'Connell's value of β_s as given and solve equations like (7-II) and (3) for α_s and $\sigma_{\alpha s}$, first using m_{h-s} and then m_{p-s}, we obtain, respectively,

$$\alpha_s = (5.80 \pm 0.11) \times 10^{-6}\ °C^{-1}$$

and

$$\alpha_s = (5.62 \pm 0.10) \times 10^{-6}\ °C^{-1}.$$

There is no satisfactory value in the literature for α_s,

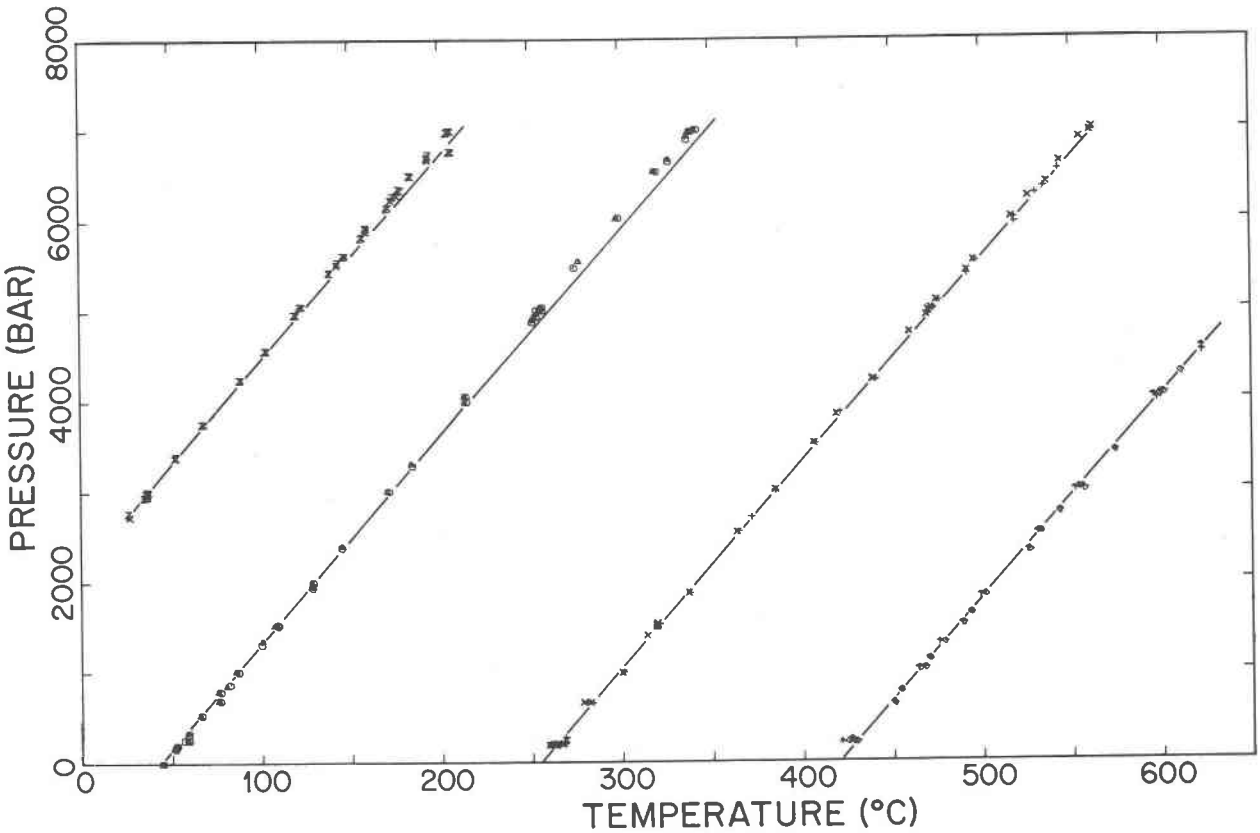

FIG. 9. Data obtained with "gate" dilatometer (WC gate) for four isomekes between synthetic periclase and synthetic halite. Solid lines are isomekes calculated from results of ultrasonic measurements by Spetzler (1969) and Spetzler *et al* (1972).

and it will be interesting to obtain an independent value for it. The success in determining β_s and the independent replication of α_s using two different isomekes suggests that the "gate" dilatometer provides accuracy that is, perhaps, sufficient for study of equations of state of solids. This is a task whose demands for experimental accuracy considerably exceed those of solid inclusion piezothermometry.

Critique. The most important reasons for adoption of the "gate" over the "J" and "opposed rods" devices are: (1) The difficult machining is concentrated into one base that is used for all experiments. Only the rods of minerals to be compared need separate machining, a task easily accomplished with conventional equipment. (2) It is unnecessary to coat the mineral rods to form an electrical circuit. (3) All of *P-T* space is accessible to isomeke determinations with only one experimental assembly, merely by appropriate adjustment of the gap between one or the other ball contacts and gate. (4) The mineral rods used are much less demanding of large single crystals. (5) The gate device, with sensitivity to strain differences $\approx 10^{-6}$, appears to be more precise by about an order of magnitude. (6) The symmetrical design and better thermocouple placement probably improve accuracy. (7) Because of the hinged gate, there is no resetting by sliding of rods on the fiducial ridges if care is taken in adusting forces of the spring clamps relative to that of the gate spring. (8) The guiding channels for electrical leads in the base and the supports S and clamps C greatly reduce the likelihood of short circuits due to movement of components. (9) The gap that determines location of an isomeke can, with practice, be more precisely set because of doubling of the *apparent* gap due to reflection by the polished gate. (10) Once assembly is completed, the gate device permits convenient and rapid switching of rods.

Some disadvantages of the "gate" device are: (1) The many more components than those of the other devices require considerable time, care, effort, and complicated procedure for their manufacture. This is especially true of the spinel base. (2) *Initial* assembly of the "gate" device takes considerably longer than

assembly of the other devices and requires patience, care, and steadiness of hand. (3) There is a possibility that the balls E will rotate during an experiment and therefore travel slightly across the ends of the rods as a result of differential strain of the wires U and the rods. This is an uncontrolled variable and conceivably could cause a slight experimental inaccuracy. (4) The small longitudinal compressive stresses on the rods, resulting from forces of springs U and Y, are approximately equivalent on both rods and probably have negligible effect.

One minor source of inaccuracy is common to both kinds of dilatometer—the finite width (~1% of rod length) of the fiducial ridge. It is conceivable that, within the width of the ridge, the "pinning" position of a rod may vary reversibly or irreversibly during the course of an experiment.

If the more demanding needs of determination of equations of state are to be satisfied, it should be possible to modify the "gate" dilatometer to eliminate some of the deficiencies cited. Work is in progress on some of these improvements.

High Pressure Apparatus

Experiments were performed in the internally heated argon high pressure apparatus described by Goldsmith and Heard (1961). Pressure was determined using a 7 kbar Heise Bourdon tube gauge, and temperature was determined with chromel-alumel thermocouples. Electrical and thermocouple leads pass through an axial hole in the closure piston (Goldsmith and Heard, Fig. 1), to two chromel and one alumel thermocouple leads, introduced in the same manner as described by Goldsmith and Heard (1961). Signal power was supplied from a 1-½ volt battery through a resistor across the input of a potentiometric millivolt recorder. Closing the circuit allowed a ~1.4 μA current to flow, the presence or absence of which was recorded on the strip chart simultaneously with the thermocouple millivoltage (Figs. 3, 8). Pressure is noted with a precision of better than ± 5 bar on the strip chart from visual readings of the Heise gauge.

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