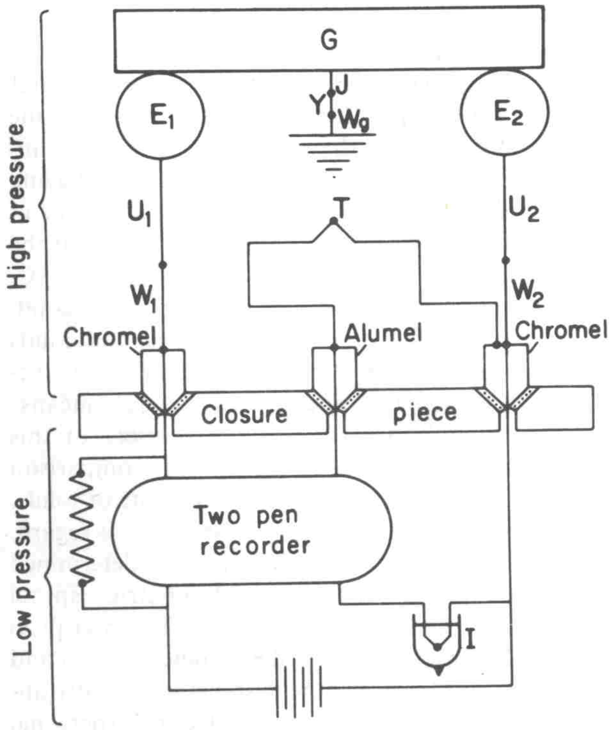

FIG. 7. Schematic diagram of electrical circuitry for the "gate"-type dilatometer. I is the ice bath for the cold thermocouple junction. Other lettered parts are same as in Figure 6.

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

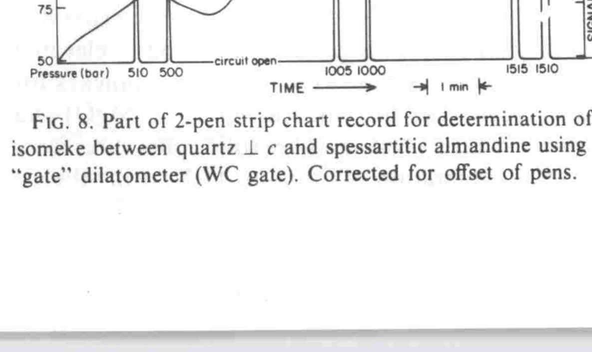

FIG. 8. Part of 2-pen strip chart record for determination of an isomeke between quartz ⊥ c and spessartitic almandine using the "gate" dilatometer (WC gate). Corrected for offset of pens.

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 $\sim$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,