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THERMAL EFFECTS OF SHEAR IN OPPOSED-ANVIL HIGH-PRESSURE DEVICES

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ABSTRACT

The effect of shearing on the average sample temperature in opposed-anvil high pressure devices is measured as a function of time, shear rate, pressure, temperature and sample material. While temperature rises as large as 50°C have been observed, for shearing conditions frequently employed, the average temperature rise should be insignificant.

Methods for introducing additional shear in the use of opposed-anvil high pressure devices have been described by Bridgman (1937) and by Datchile and Roy (1960). Their use has been shown to promote the kinetics of various phase transformations under pressure and to permit phase diagrams to be extended to lower temperatures than are possible without such shearing action. While it seems intuitively likely that heat would be generated by the shearing process, for at least some reactions the application of shear has been found to increase the rate of transformation from high to low temperature phases as well as the reverse.

Before considering the possible effects of shear on the thermodynamics and kinetics of phase transformations, it seemed appropriate to investigate directly its effect on the average sample temperature. This was prompted by observations of Kimmel (1968) who noted a strong smell of monomer on shearing polymethyl methacrylate at 12 kbar, room temperature. This observation indicated that depolymerization was occurring and suggested a substantial temperature rise in the sample under shear, possibly on a small scale.

The method of measuring this temperature rise consists of modifying the standard opposed-anvil geometry by cutting a slot, 0.05 cm deep, diametrically across the face of one anvil. In this slot a thermocouple could be embedded, usually with epoxy resin, in contact with the sample but insulated from the anvil both thermally and electrically. The output of this thermocouple was recorded on a strip chart recorder, as was in most cases the output of a second thermocouple placed in contact with the base of the anvil. Runs were made with various shear rates, temperatures and pressures on aluminum oxide powder, and at various pressures at room temperature on powders of quartz, soda-lime-silica glass, and

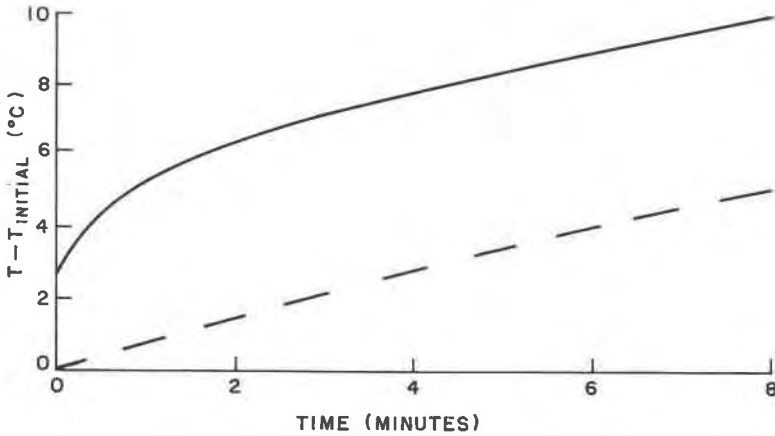


FIG. 1. Variation of sample temperature (solid) and anvil base temperature (dashed) with shearing time. Sample Al_2O_3 at 5 Kbar and 50 cycles/min shear at room temperature.

polyethylene. For all runs at room temperature, the sample thickness was 0.05 cm; for runs at elevated temperatures, the sample thickness was 0.10 cm. In all cases, the diameter of the anvil face was 0.95 cm. The tip of the sample thermocouple was embedded in the sample near its center. The shear arm assembly rotated the bottom anvil with respect to the top anvil through an angle of approximately 4.3 degrees in each half-cycle. The shear rate was varied by varying the rpm of the driving motor. In

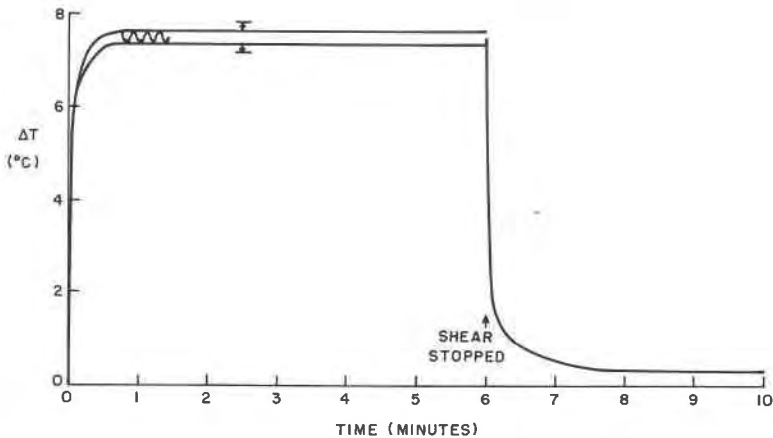


FIG. 2. Temperature difference between sample and anvil base versus shearing time. Band shows amplitude of fluctuation with frequency of the shear rate. Error bar shows long term fluctuation of band center. Sample Al_2O_3 at 15 Kbar and 50 cycles/min shear at room temperature.

runs at temperatures above ambient, an external clam-shell furnace was used. In all cases, the pressures to be reported are nominal pressures obtained from a gauge in the hydraulic system.

The thermal behavior of the sample upon initiation of shearing is indicated on the recording in Figure 1. As shown there, the temperature measured by the sample thermocouple rises sharply at the start of shearing, then more slowly at a relatively constant rate. At longer times than those shown in the figure (30 minutes or more), the temperature levels

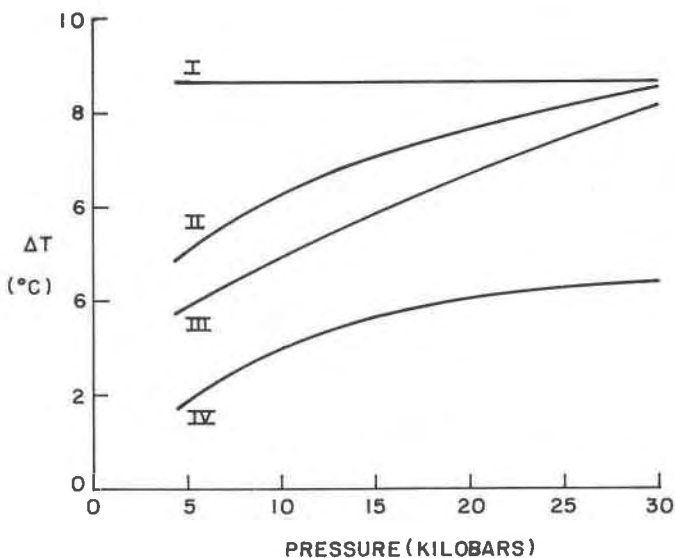


FIG. 3. Temperature difference between sample and anvil base versus pressure. 50 cycles/min shear at room temperature. I. Soda-lime-silica glass; II. Aluminum oxide; III. Quartz; IV. Polyethylene.

off. Upon cessation of shearing, there is a rather sharp decrease in the sample temperature toward the initial value (see Figure 2).

As indicated in Figure 1, the thermocouple located at the base of the anvil shows no initial sharp rise in temperature upon initiation of shearing, but indicates a rather steady rise comparable in magnitude to the steady-rise portion of the sample temperature. The temperature difference between the sample and the anvil base, shown in Figure 2, is then approximately equal to the magnitude of the initial rapid-rise portion of the sample temperature. This, in turn, is typically about one-third to two-thirds the total temperature rise of the sample (depending on the relative temperature of the anvil assembly and the surroundings).

Under most experimental conditions, the temperature rise of interest is

this difference, ΔT , between the sample and the anvil base, since the furnace temperature is most frequently controlled from a thermocouple located in the anvil base. The variation of this ΔT with pressure for a given shear rate is shown in Figure 3, and its variation with shear rate for a given pressure is indicated in Figure 4. As shown there, the difference ΔT is approximately linearly dependent on shear rate for a given pressure, rises with pressure for a given shear rate, and shows no systematic variation with initial temperature. The temperature rise varies with the nature of the sample material, being largest for the hard materials and smallest for the soft polyethylene.

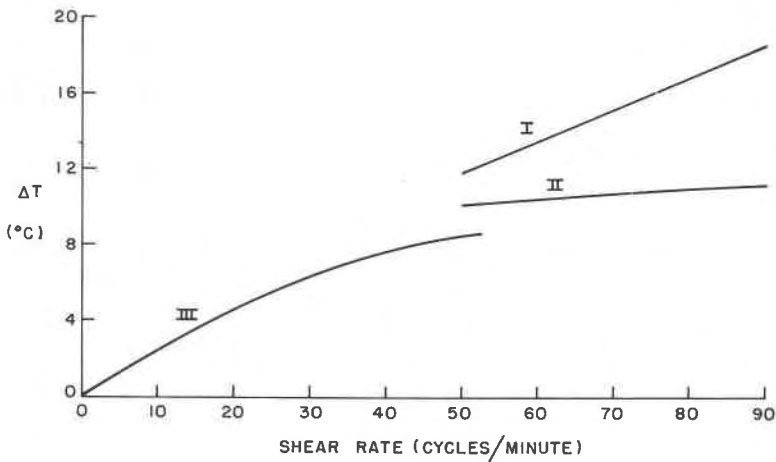


FIG. 4. Temperature difference between sample and anvil base versus shear rate for Al_2O_3 . I. 30 Kbar, 110°C ; II. 30 Kbar, 380°C ; III. 5 Kbar, room temperature.

While ΔT 's of $15\text{--}30^\circ\text{C}$ (sample temperature rises of $45\text{--}50^\circ\text{C}$) have been measured here, for shear rates frequently used—of the order of 1 cycle per minute for the present geometry (8° per min)—the difference, ΔT , should be less than a degree; and under such circumstances the average sample temperature may well be taken as the temperature at the anvil base. At higher shear rates, however, the temperature difference could be significant.

Beyond this, there remain questions as to the possible local temperature rise on shearing in the regions of contact between grains in powder-compact samples. It is clear that such temperature changes can well exceed the average values measured here; and, indeed, the smell of monomer noted by Kimmel suggests a significantly higher temperature than those estimated for the average temperature rise ($20\text{--}30^\circ\text{C}$).

ACKNOWLEDGMENTS

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MEMORIAL OF ARNO SCHÜLLER: ERRATUM

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On page 642, for November 11, 1908-March 27, 1968, read November 11, 1908-February 27, 1963.

REFERENCE

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