Apparatus for High Pressures and Temperatures



Fig. 5. Typical calibration curve showing difference between temperature of reference thermocouple and sample temperature as measured by thermocouple between piston faces under pressure.

CALIBRATIONS

Due to the solid steel pushers there is substantial axial heat flow and consequently a moderately large temperature gradient away from the center of the furnace where the specimen is located. This could be mitigated by using a furnace wound much more heavily at the ends, or by the use of high-strength low-conductivity pusher material. These measures are expensive, however, and have been eliminated on finding that a reproducible sample temperature can be attained without them. The difference between sample temperature and reference thermocouple temperature is moderately sensitive to the position of the furnace. One-quarter of an inch difference in position of the furnace typically will change this difference by about 20°C. In practice, the furnace is positioned, by moving the hinges up or down, until the sample temperature as shown by a thermocouple between the pistons in the sample position is near the reference thermocouple temperature. A temperature calibration is then made, yielding a result like that in figure 5. This calibration is reproducible, so long as the apparatus is not changed. A new calibration is made after every change in the pistons, holders, or pushers. Thermocouple temperatures are recorded on a Foxboro six-point recorder which facilitates this calibration. It was found necessary to calibrate the recorder.

The force developed by the ram is determined by the bourdon gauge and the area of the ram. This is calibrated by inserting a Baldwin Southwark CXX precision load cell in place of the apparatus. It has been found that the U. S. Gauge Company gauges are initially within their stated tolerance of

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0.5 percent of full scale, and that the friction on the Blackhawk rams is 1 percent. The Heise gauge is regularly used to check the U. S. gauges. It is desirable to measure the diameter of the piston faces to 1 percent or better, which we do with a cathetometer. One must be sure that there are no elements in the system which can introduce any undetermined friction or in any way bear any of the force which is thought to be applied to the specimen. In our apparatus, there are two such elements: the plate which aligns the ram (pl. 1), and the stainless steel ring. The alignment plate has been found by the above force calibration to introduce no measurable friction. The stainless steel ring clearly bears some load outside the piston face. This has been evaluated by measuring the deformation in the ring and roughly calculating the force required to produce this. The magnitude of this force varies with pressure, temperature, and sample size, but in our configuration it is always less than



Apparatus, with furnace open to show piston assembly.

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