

Fig. 2. Cross section of dilatometer.

vessel. The specimen S is enclosed in bellows A which is filled with water, chosen because of its low compressibility (sodium dichromate is added to inhibit corrosion). The piston P applies compressive load to the specimen and the reaction is provided by anvil R to which one end of bellows A is fixed. Movement of the free end of bellows A displaces fluid through interconnecting holes in R to an identical bellows B attached to the other side of R. The displacement of the free end of bellows B is therefore identical to that of A unless there is volume change in the specimen. The free ends of bellows A and B are connected, via a yoke X and pressure compensator, to the core and body, respectively, of a linear variable differential transformer (LVDT, supplied 'unpotted' by Schaevitz Corp., U.S.A., so as to be suitable for operation in the high-pressure fluid). The signal from the LVDT is then proportional to the volume change in the specimen. The signal is taken from the pressure vessel through two insulating seals in the top closure and power is supplied to the LVDT through another two seals.

Since the water in the dilatometer is itself compressible, pressure fluctuations would also tend to be registered due to the corresponding relative movement of the free ends of bellows A and B. To avoid this, a pressure-compensating device is incorporated, consisting of a bellows C and another yoke Y (Fig. 1). By carefully adjusting the volume of water in bellows C relative to that in A and B, the pressure compensator will give a displacement that is exactly equal and opposite to the relative displacement of the free ends of the bellows A and B due to the volume change in the water in them, thus ensuring that there is no signal from the LVDT. This permits direct measurement of volume changes in the specimen during raising and lowering of the pressure as well as ensuring freedom from spurious signals due to small pressure fluctuations during the deformation.

The specimens for compression tests are 10 mm in diameter and 20 mm long. They are sealed inside an annealed copper jacket of 0.25 mm wall thickness, closed at each end by a steel end-piece; force-fitted steel rings effect the seal between the copper and the steel end-pieces. Extension tests can also be done by replacing the compression test assembly by an interpenetrating double-yoke arrangement into which a smaller specimen (7 mm diameter, 13 mm length) can be fitted, sealed in a copper jacket soldered at its ends to the yokes.

The pressure medium in the pressure vessel was kerosene except at 8 kb when petroleum ether was used on account of its lower viscosity. Volume-change experiments at pressures above 8 kb were not attempted because the water in the dilatometer would freeze at about 9 kb. The strain rate was about $4 \times 10^{-4} \, \mathrm{sec^{-1}}$ in all tests.

Calibration tests using carbide or copper specimens enabled a small correction to be determined for the effect of slight mis-match of the supposedly identical bellows and adjustment made of the correct amount of water in the pressure compensating bellows. The volume changes in the specimen are obtained by calibrating the LVDT signal as a displacement by connecting the LVDT directly to the piston P and then multiplying by the effective cross-sectional area of the bellows. In compression tests, the overall accuracy is believed to be $\pm~0.01$ in per cent relative volume change, while in extension tests, because of the specimens being smaller, it is about $\pm~0.02$ per cent, but, as noted later the scatter between specimens tended to exceed these figures. Also, during increase or decrease of confining pressure, somewhat greater errors, of the order of $\pm~0.02$ and $\pm~0.04$ per cent, respectively, could occur over most of the range, with even greater uncertainty at very low pressures, probably due to the presence of some air bubbles and to the collapse of the imperfectly fitting copper jackets.

After the experiments, the bulk density of the specimens was determined with a sensitivity of about 1 part in 1000 by weighing in air and under water after a thin coating of paraffin wax had been applied. This provided a check on the measured final volume change. The initial densities of the specimens was determined in the same way (Table 1).

The experiments were performed in triplicate except where otherwise stated. The method of reducing the stress-strain data has been described earlier [11]. In brief, strains are expressed as percentage change of length, referred to the initial length measured at atmospheric pressure; apparatus distortion is allowed for. Stresses are calculated from the load, corrected for friction (an external load cell was used), and are based on the cross-sectional area at the given strain, assuming that the strain has been uniform and neglecting all volume changes. The stress quoted is the 'differential stress', that is, axial stress minus the confining pressure. The errors in stress-strain measurements [11] are small generally compared with the scatter in results between specimens except in the early parts of the curves (up to about 1 per cent strain) where the errors may be considerable.