Letters

A new method for microstrain testing in compression

Experiments to study the early stages of plastic deformation in crystalline solids require measurement of strain to a sensitivity of 10^{-6} or better [1], and although transducers are readily available for this purpose, accuracy can be difficult to achieve. This can be due to either the properties of the testing machine itself or the geometrical arrangement of the transducers used and the way they are coupled to the specimen or machine.

Among the features uniaxial testing machines should have for microstrain work is the ability to accurately indicate the stress and strain in the specimen at all times. This, however, is impossible to achieve for stress when the overall stiffness constant of the machine is low or variable [2]. Furthermore, if the stiffness constant varies with load, a constant speed of crosshead motion will not result in a constant strain-rate of the specimen [3]. Other difficulties appear to have been encountered with compression testing in particular [4-9]. For example, the lack of mechanical stability under small loads can require the use of a permanent stress bias [4, 7, 9] and specimen-end effects and compression-anvil indentation can lead to errors in the strain [5-7, 9]. We have also found susceptibility to vibration and spurious strain signals which result from crosshead reversal to be serious problems for compression testing on lead-screw machines. We have, therefore, designed a compression jig which overcomes these difficulties; it can be used in both the microstrain and macrostrain regions.

The conventional uniaxial testing arrangement can be represented schematically by a system of springs connected in series, and is basically soft [2, 10]. If K_L is the effective spring constant of the load cell, and K_M the overall constant of the remainder of the machine, a consideration of the effect on the system of a force exerted by the specimen easily shows that the effective spring constant K of the system as seen by the specimen is given by

$$K^{-1} = K_L^{-1} + K_M^{-1}. \qquad (1)$$

Thus, as stated above, if K_L and K_M are not sufficiently large, transient load effects in the specimen cannot be detected. The jig we have designed for compression tests is similar in principle to that used by Burbach [10] to study yielding in tension, and consists of high-modulus rods connected in parallel with the specimen and load cell, as shown schematically in Fig. 1. Plates A and B remain parallel at all times, and K_R is the total spring constant of the rods. The overall spring constant for springs connected in parallel is the arithmetic sum of the individual constants, and is, therefore, much larger than that for the same springs connected in series [10]. The effective spring constant K as seen by the specimen for the system depicted in Fig. 1 can be shown to be given by:

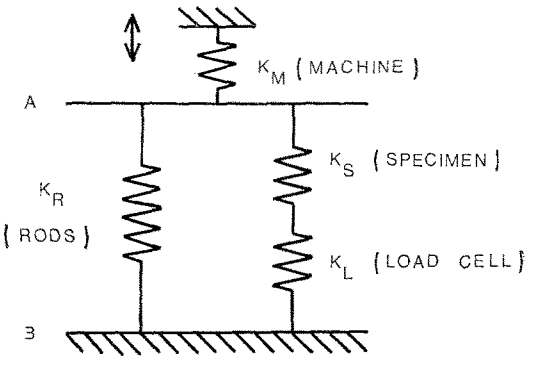

Figure 1 Schematic representation of the testing jig.

$$K^{-1} = K_L^{-1} + (K_M + K_R)^{-1}, \qquad (2)$$

which approximates to

$$K^{-1} = K_L^{-1} + K_R^{-1} \qquad (3)$$

if $K_R \gg K_M$. Thus by placing high-modulus components in parallel with the specimen and load cell in uniaxial tests, the effect of the machine characteristics on the detection of specimen transients is avoided. The load cell must, of course, be hard if full benefit is to be gained.

Although the value of such a system for studying macroyielding phenomena such as yield drops has long been recognized [11], the value of its use for microstrain tests has not been exploited. The only requirement is the use of a

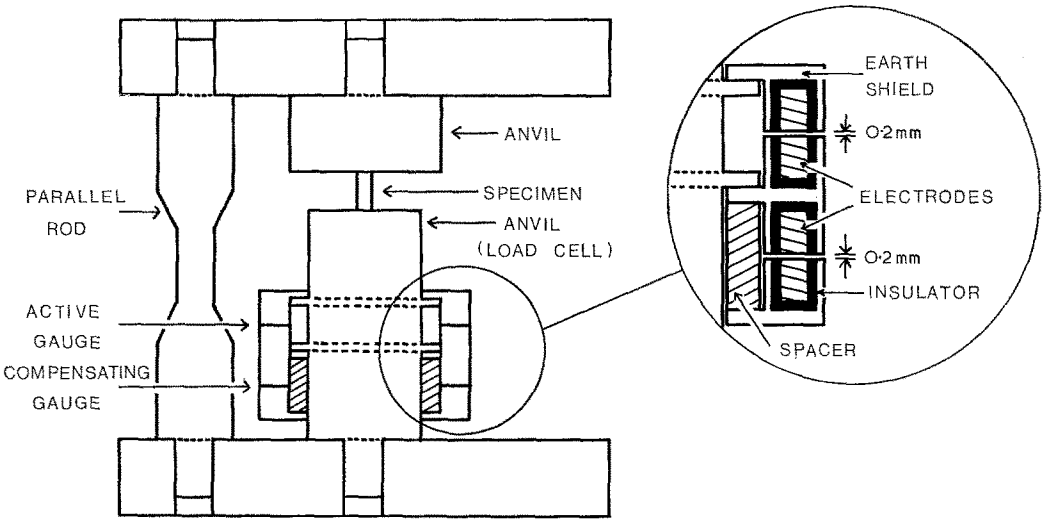

Figure 2 Vertical section through the testing jig; only one parallel rod is included for clarity.

testing machine capable of straining the parallel rods to achieve the desired specimen strain. The rods must only deform elastically, of course, and so, from a knowledge of the machine load capacity and the maximum specimen strain required, the dimensions of the rods can be calculated.

The jig is shown diagrammatically, but drawn to scale, in Fig. 2. Only one of the three rods, which are arranged symmetrically around the jig axis, is shown for clarity. The metal components of the jig are made from hardened EN26 steel, and the rod dimensions are such that a 100 kN load on the jig produces a 1% strain in the specimen. The specimens used are 1 cm long and up to 4 mm in diameter, and the upper and lower compression anvils have dimensions such that they absorb less than 10% of the length change in the rods, even when the specimen has a modulus as high as that of EN26. To measure the load on the specimen, the lower anvil is used as a load cell by measuring its strain over a 1 cm gauge length. As this has to be done with a resolution at least ten times that used for the specimen strain, we have used an annular parallel-plate capacitance transducer coupled to an Automatic Systems Laboratory 1055 displacement meter. This measures the "active" transducer gap relative to that of a standard transducer with fixed gap, which is also mounted on the lower anvil and provides a degree of temperature compensation. A section through the load cell is also shown in Fig. 2. The insulator in the transducers is Araldite compound AY103, which has a thermal-expansion coefficient compatible with that of EN26, and this permits use of the jig over a range of temperatures. As with any microstrain system, the cell must be kept at a fairly constant temperature during a test. For the load-cycling tests we have been concerned with, a simple version of the double-walled cryostat system described by Reed [12] has given sufficient temperature control for successful use of the jig down to 77 K. The cell may be calibrated by placing weights on the lower anvil, and it gives a linear output up to at least 1000 kg load.

To minimize errors due to misalignment of the anvils and specimen ends, the compression faces of the anvils were machined parallel to within 2.5×10^{-6} m and the specimen ends are ground using the procedure of Meakin [4]. As specimen ends effects and anvil indentation are known to be a problem with microstrain compression tests [5-7, 9], we measure specimen strain with resistance strain gauges of the temperature-compensating type. Two diametrically opposed gauges are used for each specimen. With strain and load plotted directly on an x-y recorder, the system gives a resolution of 1 kg of load and 2×10^{-6} strain per cm of chart with little noise. The accuracy is such that the elastic modulus measured on the chart always falls within 5% of the dynamic modulus calculated from elastic constant data. A particular advantage of the jig is that it can be preloaded to render

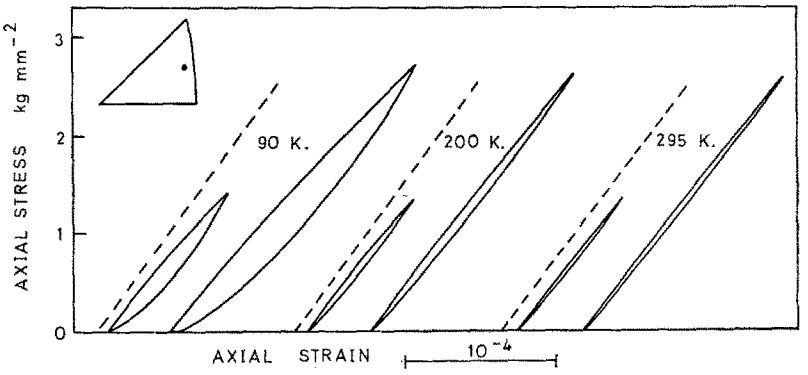

Figure 3 Typical hysteresis loops, as traced from the *x-y* recorder chart, obtained for two different amplitudes at 90, 200 and 295 K. The tantalum specimen orientation is shown in the inset unit triangle.

the specimen measurements virtually independent of extraneous vibrations, friction in couplings and alignment errors, which are problems with conventional arrangements at low loads. We have used a specimen length of 1 cm and an anvil spacing of 1.005 cm, thereby allowing a preload of 50 kN on the jig before the specimen is loaded. An additional feature is that since the parallel rods only deform elastically and there are no moving parts within the jig, a constant strain rate of the specimen is readily obtained.

Tracings taken from the recorder chart of typical stress-strain loops produced by load-unload tests at a frequency of 0.1 Hz on a tantalum single crystal at several temperatures are shown in Fig. 3. The elastic line calculated from the single crystal elastic constants is shown as a broken line. The loops are clearly lenticular in shape, and resemble those seen in tension by many workers. It may therefore be that the parallelogram shapes observed by Meakin [4] arose from machine or misalignment effects, as concluded by Tomalin [7]. The sensitivity achieved by use of the jig enables direct comparisons to be made between the results of uniaxial microstrain tests and conventional low-frequency internal friction experiments. It can be seen from the limited data of Fig. 3 that the damping (fraction of energy dissipated per cycle) for a given stress amplitude varies strongly with temperature, in broad agreement with the torsion pendulum results of Chambers [13]. We also find that the damping decreases with increasing stress amplitude and is almost independent of frequency in the range 0.02 to 0.5 Hz, and further analysis of the results is in progress [14]. As stated earlier, the jig

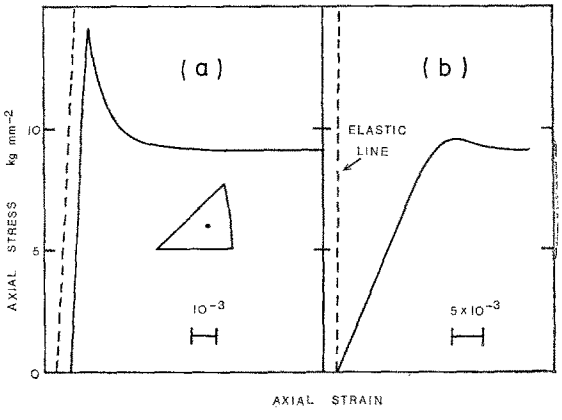

Figure 4 Stress-strain curves, for identical specimens of tantalum, obtained using (a) the testing jig and (b) a lead-screw machine in a conventional manner. The specimen orientation is shown in the inset unit triangle.

described here can also be used for studying macroscopic yielding. The advantage of doing so is clearly shown in Fig. 4, where the stress-strain curve for a tantalum single crystal at room temperature obtained using the jig is compared with the curve for an identical specimen obtained using an Instron machine in the conventional manner. It should be noted that the virtual absence of a yield drop in the latter case does not depend on the manner in which the strain was measured.

In conclusion, unless rapid load changes occur within the specimen, there is no need to use a hard jig for microstrain measurements *per se*, but the advantages to be gained from its use are considerable, and do not appear to have been noted previously for the particularly difficult area of compression testing. The jig described

has been used with a 300 kN machine in order to obtain macroscopic yielding, but there is no reason why similar designs should not be used with machines of more moderate capacity.

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Quartz matrix isolation of radioactive wastes

One of the factors to be considered in the increased utilization of nuclear fission for energy production is the safe transportation and storage of the radioactive wastes derived from chemical reprocessing of spent nuclear fuels. Several components of the wastes will be highly radioactive and thus constitute a threat to man for 10^4 to 10^6 years. The presently conceived solution is to melt the wastes into glass forming compositions, solidify this melt in storage containers and either store these containers in shielded vaults or bury them in geologically stable strata such as bedded salt formations [1].

The solidified glass should be dense, hard, able to withstand constant temperatures of up to 600° C from radioactive decay and highly insoluble. Present processes use low solubility phosphate or borosilicate glass compositions. However, most phosphate based glasses devitrify above 550° C and borosilicate compositions often undergo liquid-liquid phase separation during melting. Both phenomena result in significantly reduced leaching resistance.

It is well known that ceramic articles fashioned up to 10^4 years ago have survived burial with little apparent degradation. Certain ceramics would also be ideal matrices for isolating radioactive wastes. However, the temperatures necessary to form mixtures of these wastes and most ceramics into dense solids are hundreds of degrees above the highest values now employed in waste solidification processes (1000 to 1200° C). It is suggested that with the ceramic processing technique, hot-pressing, a dense solid may be formed out of the radioactive waste and a low leachability crystalline or glassy matrix at or below 1000° C. In order to demonstrate this idea common sand (quartz) was chosen as one matrix material. Detailed results using ten other less effective matrix materials will be described elsewhere [2].

The simulated radioactive waste chosen for this study was PW-4m, a typical light water reactor fuel reprocessing waste whose major components are Na_2MoO_4 and nitrate solutions of Sr, Ba, Cs, rare earths and Zr as fission products and Fe, Cr, Ni from corrosion of fuel and reprocessing tanks. The liquid PW-4m was dried at 110° C and calcined at 500° C for 6 h and then ground to pass a −200 mesh sieve. The matrix material was quartz (99.9% SiO_2,