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Authors

Hulse, C.O.

Copley, S.M.

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EQUIPMENT

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C. O. Hulse and S. M. Copley

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HIGH-TEMPERATURE COMPRESSIVE DEFORMATION EQUIPMENT

C. O. Hulse* and S. M. Copley†

Inorganic Materials Research Division of Lawrence Radiation Laboratory
and Department of Mineral Technology, University of California,
Berkeley, California

October 27, 1965

ABSTRACT

Three apparatus for deforming ceramic materials in compression at elevated temperatures have been developed. Methods used for loading, alignment, and measurement of stress and strain are described.

*C. O. Hulse is now research scientist at United Aircraft Corporation, East Hartford, Connecticut.

†S. M. Copley is now research associate at Pratt & Whitney Aircraft, New Haven, Connecticut.

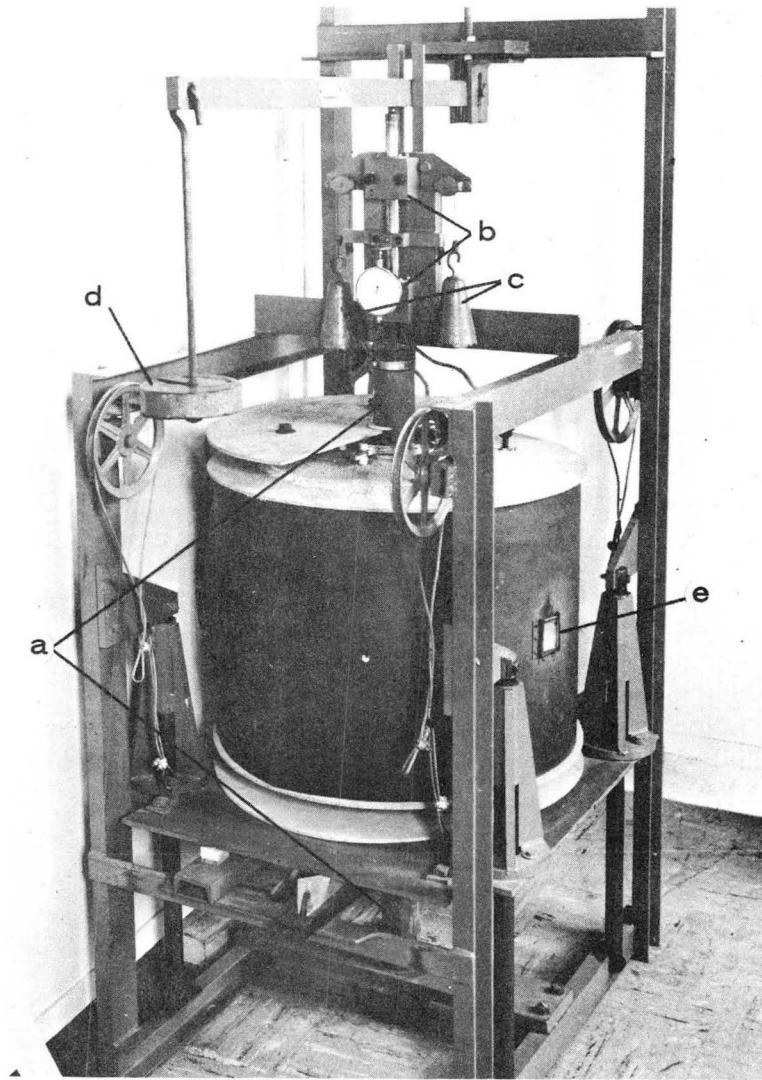
1. INTRODUCTION

As part of a continuing investigation of the mechanical behavior of ceramic materials, three apparatus for obtaining stress-strain and creep data at elevated temperatures were developed. Compressive loading was used in all three. It offers several distinct advantages over tension and bending: (a) the stress distribution in an elastically compressed rectangular parallelepiped is both uniaxial and uniform, (b) no special specimen grips are required for compressive loading, which would be a design problem for tensile loading at elevated temperatures, (c) smaller specimens can be used for compression experiments than for tension experiments, and (d) compression experiments are better suited for studying deformation mechanisms of ceramic materials than tension or bending because the tensile stresses accentuate brittleness, thus obscuring deformation. The first apparatus was used to study the creep of firebrick, and developed a maximum load of 1400 lb at temperatures up to 1550°C.¹ The second was used to study the stress-strain and creep behavior of MgO single crystals, and developed a maximum load of 6000 lb at temperatures up to 1200°C.^{2,3} The third was used to study the stress-strain behavior of MgO single crystals and polycrystals at temperatures up to 1600°C.⁴ These apparatus will be designated as I, II, and III, respectively. All three were designed to operate in air; however, III could be easily adapted to obtain data in a vacuum or in an inert atmosphere.

2. GENERAL DESCRIPTION

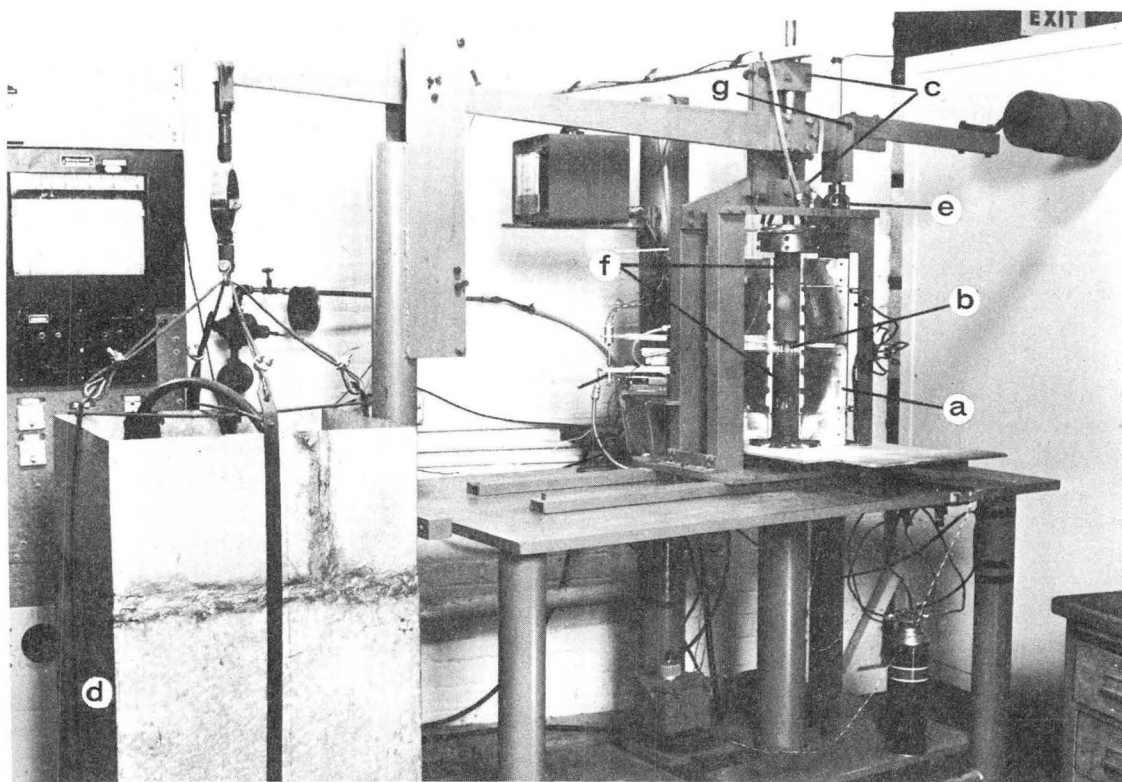
Figure 1 shows an overall view of Apparatus I. A vertical tube furnace with SiC elements was mounted on a counterweighted platform, which could be lowered to expose the specimen. The load was transmitted by two SiC rams, a, which can be seen protruding from the furnace. The upper ram was attached to a 1-in. -diam steel rod that was parallel and in line with the ram axis. This rod was positioned by two linear ball-bearing bushings, b, so that motion was possible only parallel to the ram axis. These bushings were mounted on a heavy steel plate that could be moved in three dimensions, with adjustment bolts to secure alignment. The load was applied through a ball at the end of the rod by means of a lever system with a 3:1 mechanical advantage. Strain was measured optically through a port, e, in the side of the furnace by a method to be described in Section 3.

Apparatus II is shown in Fig. 2. The specimen was heated by a small wire-wound furnace, a, (Kanthal A-1, Kanthal Corp., Stamford, Conn.), which was split so that both sides could be removed to expose the specimen, b. SiC rams were again used to transmit the load and were positioned by linear ball bearing bushings, c, as in Apparatus I. The load was applied by filling tank d with water at the end of a lever system with a 10:1 mechanical advantage. The upward force on the lever fulcrum was



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Fig. 1. Compressive deformation Apparatus I: a: SiC loading rams; b: alignment bushings for upper movable ram; c: counterweights for upper ram; d: loading weight; and e: Vycor window in Globar furnace for measurement of deformation.



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Fig. 2. Compressive deformation Apparatus II: a: half of furnace; b: specimen; c: alignment bushings; d: loading tank; e: universal joint; f: SiC rams; and g: lever fulcrum.

borne by a steel strut with a universal joint in it, e, which connected to a steel plate bolted to the floor. Strain was measured through a port in the furnace with an electromechanical device to be described in Section 3.

For tests above room temperature the furnace was controlled by an Electro-volt reactor system using a 5-mV full-scale recorder with scale suppressions. The temperature sensing for the controller was four Pt/Pt + 10% Rh thermocouples in series whose beads were in contact with the specimen. The temperature was measured by another thermocouple using a mirror potentiometer which could be read to 0.002 mV. The total temperature variation was normally within 0.5°C.

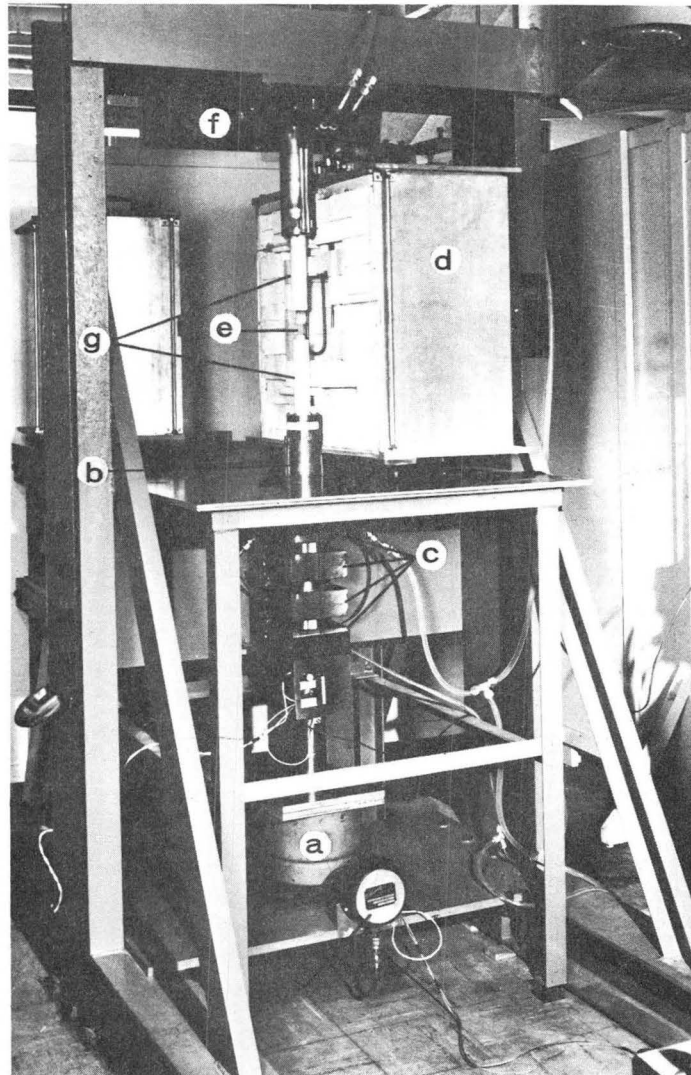
Apparatus III is shown in Fig. 3. Force is applied by an air-activated diaphragm cylinder, a, to a cylindrical column, b, which is aligned by three linear ball-bearing bushings, c. The column extends into the furnace, d, and transmits force to the specimen, e. Another load column enters the top of the furnace and is fastened to a triangular plate, f, which was positioned by opposing bolts. The load was applied to the specimen with alumina loading rams. The specimen was heated by furnace d with MoSi₂ heating elements (Super Kanthal, Kanthal Corp., Stamford, Conn.). Strain was measured through a port in the side of the furnace with an electro-mechanical device similar to that used in Apparatus II. Stress was measured with a Bourdon tube pressure transducer connected to the diaphragm cylinder. The temperature was measured and controlled with a single Pt/Pt 10% Rh thermocouple pressed against the side of the specimen. Three-mode proportional control was used as in Apparatus II.

3. DISCUSSION

Loading Rams

In Apparatus I and II the loading rams were dense, hot-pressed SiC cylinders with cylindrical cavities in one end to reduce heat losses. These rams had satisfactory strength and did not react with either the firebricks or the MgO single crystals at temperatures up to 1250°C. In Apparatus III, dense, impervious Al₂O₃ loading rams were used because compatibility tests revealed extensive reactions between MgO and SiC at about 1500°C. MgO and Al₂O₃ also react; however, it was found that this reaction could be controlled by placing Pt sheet 0.001 in. thick between the two materials. To further protect the Al₂O₃ loading rams, Al₂O₃ buttons were placed between the specimen ends and the loading ram surfaces. The Al₂O₃ had satisfactory strength and was considered superior to the SiC because of its low thermal conductivity.

In compression tests on single crystals, it is frequently found that shear on the active slip systems has a component on a plane perpendicular to the stress axis. This component produces a displacement of one specimen end relative to the other so that, to obtain meaningful results, one



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Fig. 3. Compressive deformation Apparatus III: a: diaphragm cylinder; b: loading column; c: alignment bushings; d: half of furnace; e: specimen; f: triangular alignment plate; and g: Al_2O_3 rams.

must control the friction conditions for sliding on the loading-ram surfaces. Kocks has approached this problem by coating the loading-ram surfaces with a teflon film so that the sliding friction is minimized.⁵ Our measurements have been restricted to multiple-slip orientations in which the resolved shear on a plane perpendicular to the stress axis is zero provided that a sufficient number of the equally stressed slip systems are active. A positive end constraint was applied to insure that the necessary intersecting slip occurred. A detailed discussion of this type of compression test has been given by Copley.⁶

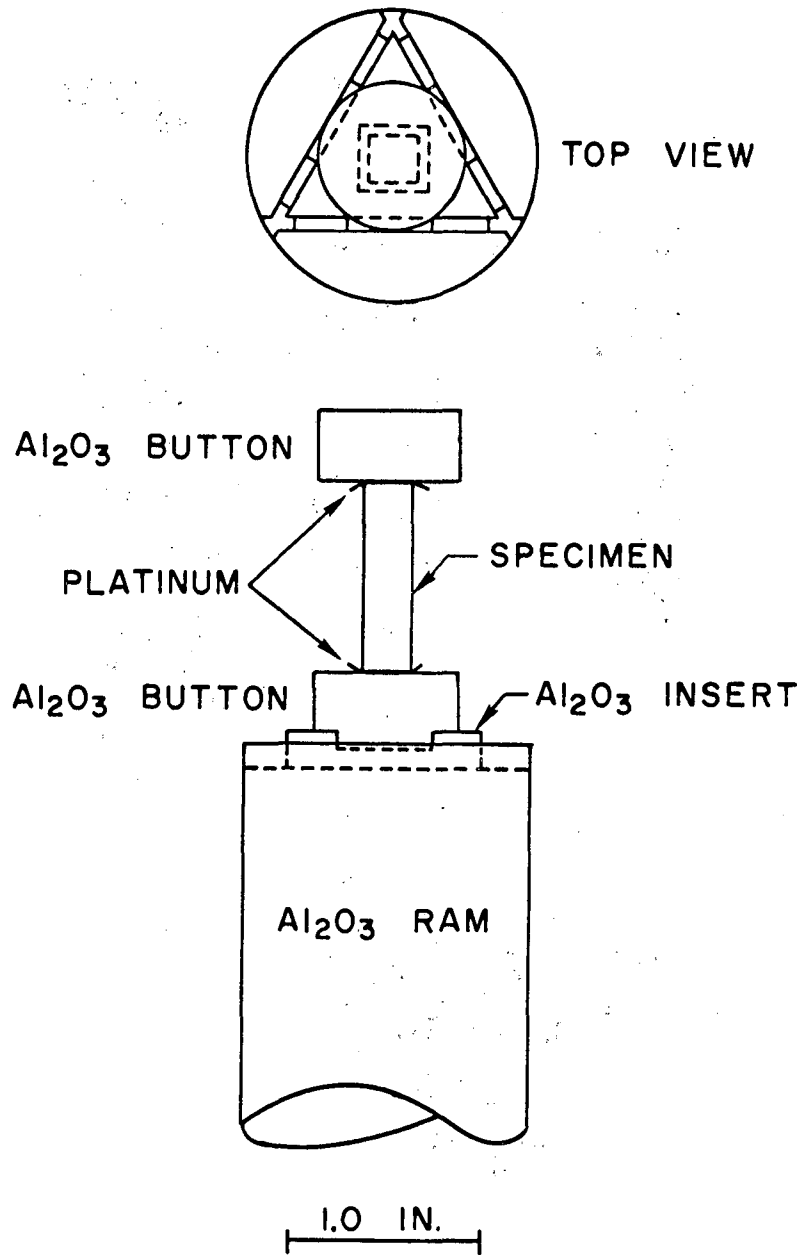
Several methods for pinning the specimen ends to the loading-ram surfaces were tried. The most successful of these involved cutting slots in the ram surfaces and then shaping ceramic pieces to slide in these slots up against the specimen at a small angle to its faces, thereby holding it in place. Figure 4 shows the arrangement in more detail for one end. A small preload was constantly applied to the sample during heating to aid in holding this system together. The specimens adhered quite strongly to the buttons because of a limited reaction of the Pt sheet with MgO and with Al_2O_3 .

The resistance of the load column to side forces is generally weakest where the ceramic loading ram is fastened to the rest of the load column. Figure 5 shows the three types of connectors that were used. Figure 5a shows a water-cooled steel cup connector that was used in Apparatus I and II. This type proved inadequate, however, and was replaced by the water-cooled connector shown in Fig. 5b. In this connector, the loading ram is gripped by two layers of eight transite chocks. Each chock is backed by a stainless steel plate and is individually bolted against the loading ram after it is centered in the cup. Figure 5c shows the connector used in Apparatus III. Solid Al_2O_3 cylinders 8.5 in. long, 1.5 in. in diameter, and with 3.0-in. -diam flanges were used as rams. The flanges were slotted with a diamond saw so that a stainless steel ring with internal water cooling could be placed on the furnace side of the flange and bolted through a water-cooled base plate to the rest of the loading column.

Another problem in compression loading is that of making the loading ram surfaces and specimen ends parallel. It was found that the ram surfaces could be accurately paralleled by observing an extinction slit for light between ram surfaces in two perpendicular directions. In this procedure the rams are run together until they almost touch. By looking through the slight opening between the two ram bearing surfaces toward an evenly lit, bright background, one can accurately determine the degree and sense of nonparallelism of the two surfaces by watching how the light between the two ram surfaces disappears as the rams are slowly brought together. Compression specimens were shaped with parallel ends by using a jig described previously.

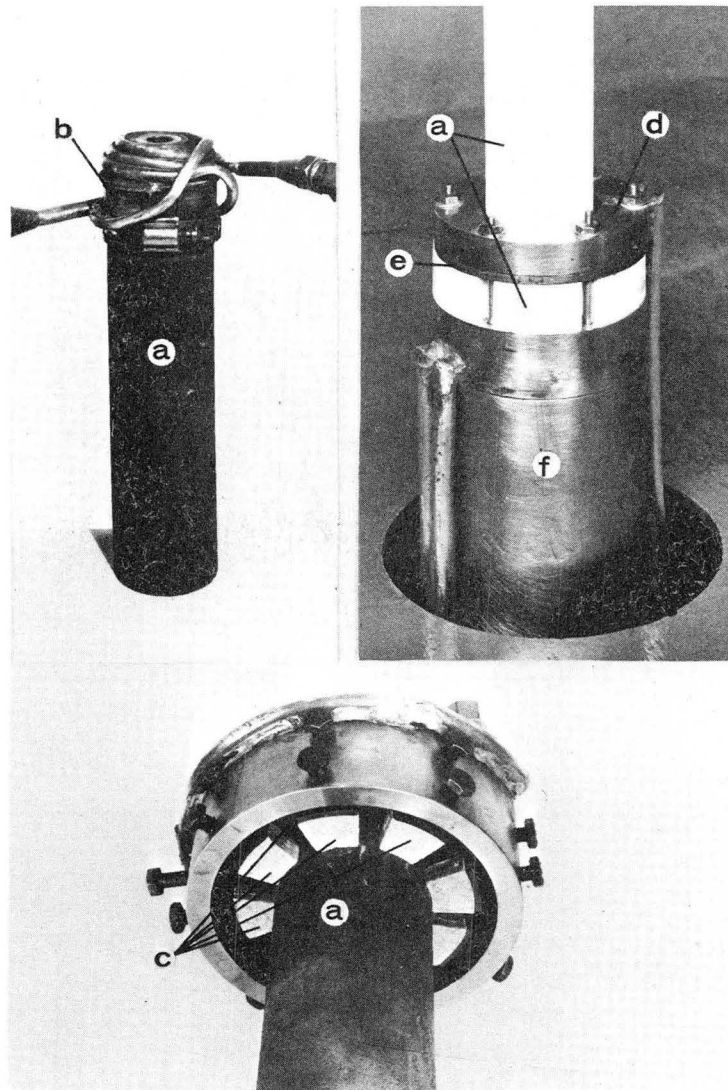
Loading Rate

In Apparatus II and III the load was applied at a constant rate. At small strains, at which a constant force rate can be regarded as a constant stress rate, the slope of a stress-strain curve of a specimen loaded in this



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Fig. 4. Method of preventing lateral end translation of single-crystal specimens.



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Fig. 5. Compression ram attachments to the metal alignment system:
a: compression rams; b: water-cooled slotted steel cup; c: Transite
pieces; d: internally water-cooled ring; e: copper gasket; and f: main
steel loading column.

manner is therefore inversely proportional to strain rate. The constant-stress-rate method of loading has been discussed in detail by Copley and Pask.⁴

In Apparatus II a constant loading rate was obtained by filling the tank at a constant rate. The flow rate was controlled by adjusting a valve. It was necessary, however, to use an overflow tank to insure a constant head of water. The hydraulic loading mechanism used in Apparatus III is diagrammed in Fig. 6 and shown in Fig. 7. (It does not appear in Fig. 3 because it had not been installed at the time that picture was taken.) The control valve, a, is capable of supplying 0 to 100 psi air pressure to the pressure tank, b, which is almost filled with oil. During a stress-strain test, pressure is transmitted by an oil line from the pressure tank to a diaphragm cylinder, c. The diaphragm cylinder has an effective end area of 50 in.², and thus 100 psi can produce a force on the loading column of 5000 lb.

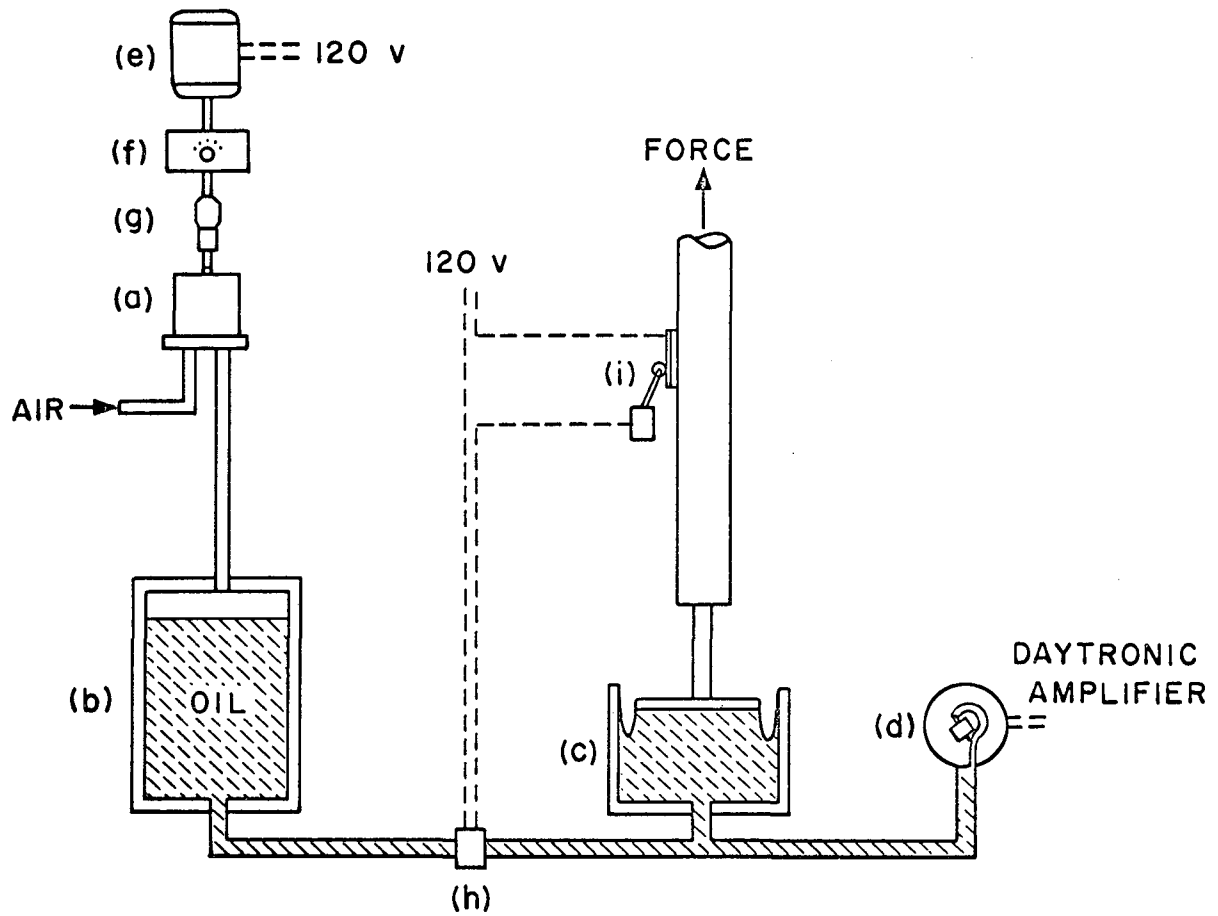
During a stress-strain test, the stress on the specimen is obtained indirectly by measuring the pressure in the diaphragm cylinder with a Bourdon-tube pressure transducer, d. The stress system is initially calibrated by (a) making a load cell with resistance strain gages, (b) calibrating the output of this load cell with a dead-weight machine, and (c) using this cell to produce a plot of force on the load column vs output of the pressure transducer. A linear relationship is obtained with no hysteresis. Thus by suitably adjusting amplification it is possible to read force directly on a time base or x-y recorder.

Various constant force rates are effected by coupling a constant-speed motor, e, to an adjustable-speed transmission, f. The transmission drives a micrometer, g, which depresses the actuator on the pressure valve at a constant rate. The response of the pressure valve is linear and thus a constant loading rate is obtained. The response of the system is quite rapid, depending on the setting of the air-bleed valve on the delivery side of the pressure-regulating valve.

A relay valve, h, is placed on the line connecting the pressure cylinder to the diaphragm cylinder (Fig. 6). This valve is operated by a switch on the loading column, i, so that if the specimen breaks during loading, displacement of the loading column causes the relay valve to close, thus abruptly stopping the loading column because of the incompressibility of the oil.

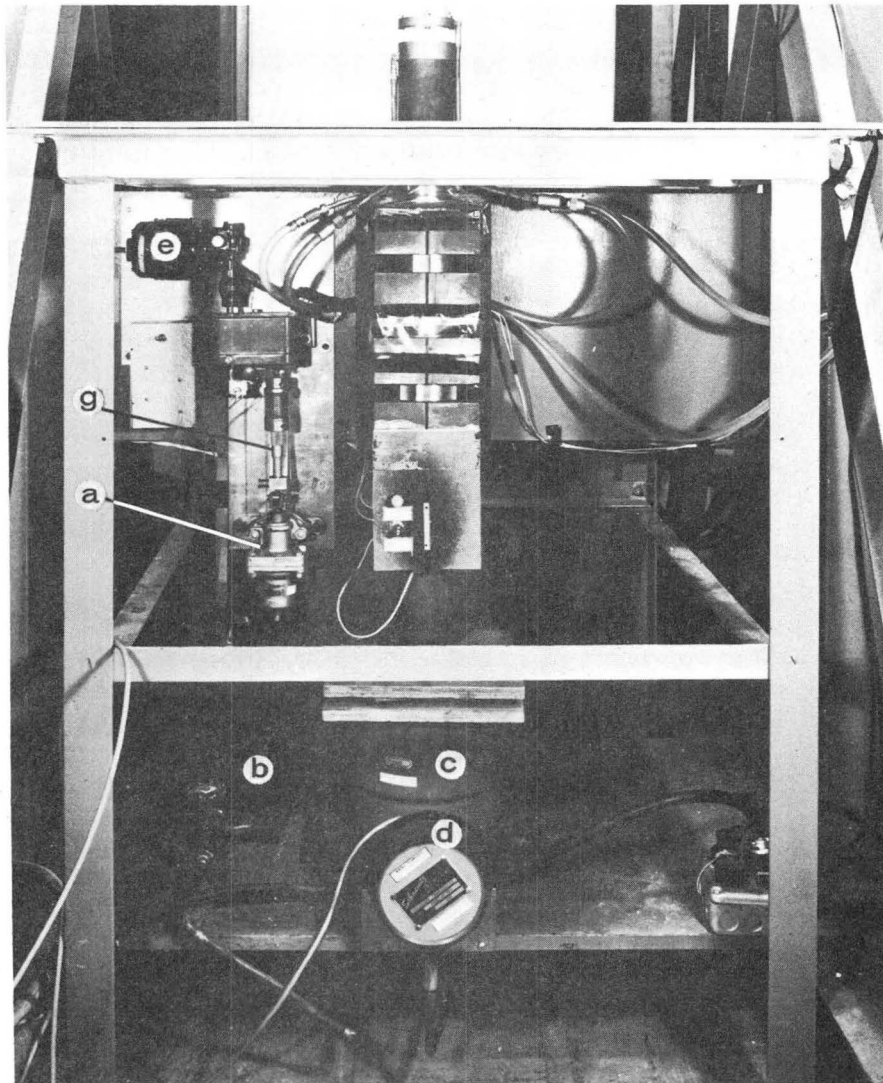
Strain Measurement

The strain was measured with Apparatus I by taking photographs of the sample through the window, e, shown in Fig. 1. A 35-mm Exakta camera was used with extension tubes and a 135-mm telephoto lens. One edge of the sample was removed except for gage-marking spikes of material about 1 in. apart near the center of the 1.5-in.-tall samples. The distances between these spikes on microfilm negatives exposed at different times during the deformation were compared with a corresponding distance on negatives made before the test, and the strain (a unitless quantity) was



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Fig. 6. Diagram of hydraulic loading system for Apparatus III: a: air pressure control valve; b: pressure tank; c: Westinghouse diaphragm cylinder; d: Bourdon pressure transducer; e: constant-speed motor; f: adjustable-speed transmission; g: micrometer; h: safety relay valve; and i: column-limit safety switch.



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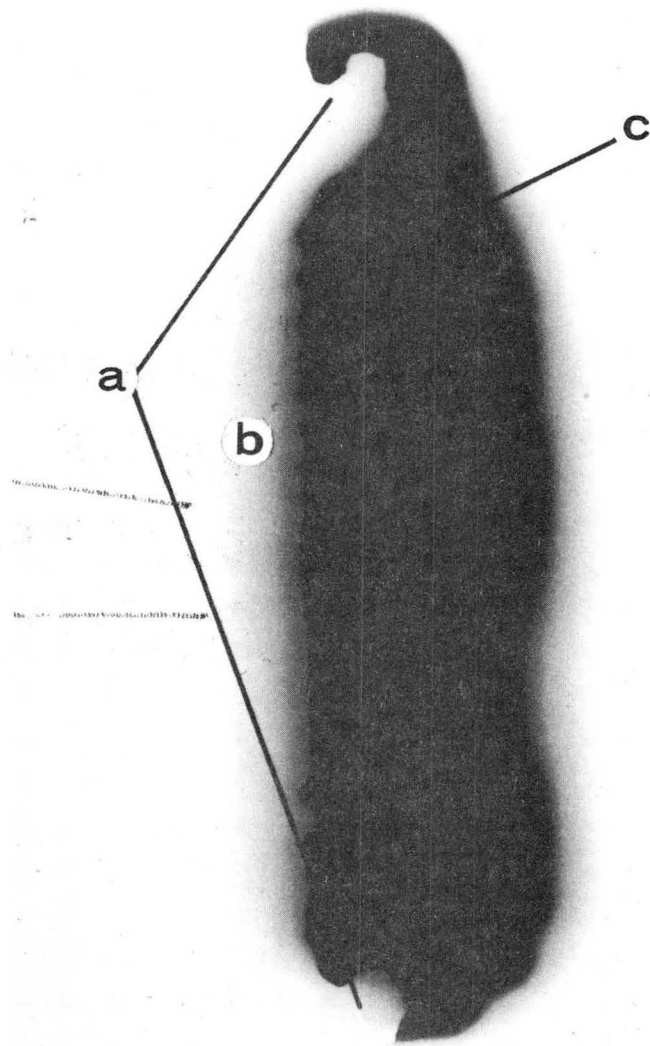
Fig. 7. Photograph of hydraulic loading system for Apparatus III.

computed from these ratios. The distances on the negatives were read at $100\times$ magnifications with an optical micrometer which could read distances to ± 0.0001 in. Because all the negatives were on the same strip of film, errors due to film shrinkage were negligible. The strain sensitivity of this method was about 1×10^{-3} in./in. Figure 8 shows a positive print from a typical negative showing the gage spikes at a. This method of strain measurement is advantageous in that it is easy to install, does not require any physical contact with the specimen, and provides a permanent record; the principal disadvantage is that it is not continuously recording.

The strain-measuring device used with Apparatus II is shown in Fig. 9. It was similar in design to that used with Apparatus III. The gage lengths (normally about 0.5 in.) were set by small divots made with No. 90 twist drills in the middle section on one side face of the approximately 1-in. -tall specimens. The two horizontal sapphire rods shown at a in Fig. 9 pressed into these divots and formed one side of a double lever system with the differential transformer transducer on the other end of the levers as shown at b and c. The transformer core was mounted on a silica rod which extended upward from an Invar micrometer attached to the lower lever. This micrometer was normally used to calibrate the system at temperature just before the test was begun. The output of the linear differential transformer was amplified by a Daytronic 300B displacement indicator and then fed directly to a recorder. Both sapphire rods were independently spring-loaded against the sample. The lower rod had an internal spring-loaded shaft within itself and was vibrated by a buzzer for smooth travel. The upper rod was loaded by a spring which moved the whole platform, d, on steel balls toward the specimen. When the furnace is in place, the sapphire rods pass through a slot in the side of the furnace. Heat shields (not shown in Fig. 9) were also attached to the sapphire rods to prevent heating of the strain device. The usable sensitivity of this measurement system was about 1×10^{-4} in./in.

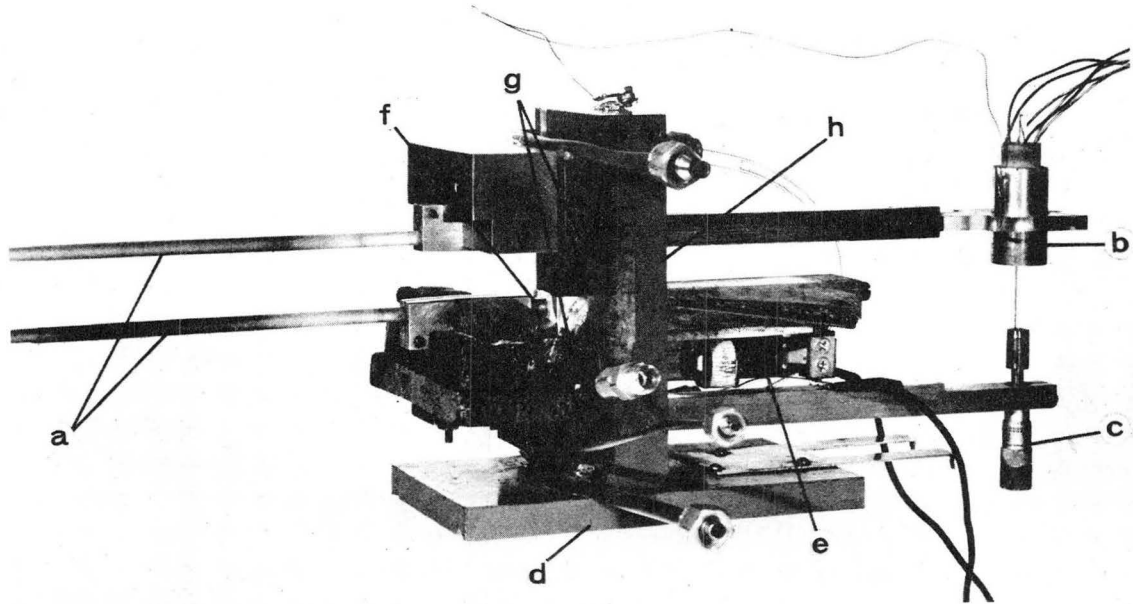
For testing down to liquid nitrogen temperatures, the sapphire rods were replaced with steel rods bent to contain a "hump" parallel to their axes. The specimen sat on a steel disc cemented to the lower ram and after the steel rods were placed in the specimen divots, a short, hollow cylindrical steel section was screwed onto the steel disc from below to form a cup around the specimen. The sides of this cup fitted into the humps in the steel rods and the cup containing the specimen was then filled with the desired coolant.

Both the strain-measuring systems described here measure deformation directly on the sample and in sample volumes away from the influence of end constraints imposed by the loading rams. For single crystals, the particular choices of gage length for specific crystal heights and widths are primarily determined by the requirement that none of the possibly active slip planes within the gage volumes should intersect directly with the loading-ram surfaces. The choices and effects of the end constraints have been discussed elsewhere by one of the authors⁶ and by others.⁷



ZN-5231

Fig. 8. Positive print of a negative used for measuring firebrick strains: a: gage spikes; b: firebrick specimens; c: dark background of hole in furnace chamber.



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Fig. 9. Strain-measuring device for Apparatus II: a: sapphire rods; b: linear differential transformer; c: Invar micrometer supporting transformer core; d: spring-loaded platform; e: buzzer; f: spring-loaded shaft in lower lever; g: pivot bearings; and h: Invar pivot support frame.

4. SUMMARY AND CONCLUSIONS

Three pieces of compressive deformation equipment have been described. The major problems encountered with this type of testing are securing and maintaining good ram alignment, preventing reactions between the loading rams and the samples, controlling the translation of specimen ends (if it tends to occur), and measuring the deformation, preferably away from the sample end constraints. The best alignment system appeared to be that described for Apparatus III (Figs. 3 and 5c). The best loading-ram material for general use appears to be commercially available dense alumina used in conjunction with replaceable end pads if necessary. A method was devised for preventing the translation of specimen ends. An accurate electromechanical device continuously records sample deformation up to 1600°C (Fig. 9). Another photographic method of measuring deformation has also been used. This latter method is relatively inexpensive and may be particularly useful at extremely high temperatures.

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