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PROGRESS REPORT

ON

NASA RESEARCH GRANT ~~20~~-NGR-05-020-086

MECHANICAL BEHAVIOR OF POLYCRYSTALLINE  
NON-METALLICS AT ELEVATED TEMPERATURE

for the period

April 1, 1965 through September 30, 1965

submitted to

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## MECHANICAL BEHAVIOR OF POLYCRYSTALLINE NON-METALLICS

### AT ELEVATED TEMPERATURE

(SC-NGR-05-020-084)

Summary of Research for the Period April 1, 1965 to September 30, 1965

The creep behavior of polycrystalline aluminum oxide and sodium chloride is currently being studied. Aluminum oxide, a material of great technological interest, exhibits covalent bonding. It has few slip systems and little ductility even at high temperatures. Sodium chloride exhibits ionic bonding. It has several slip systems operating at high temperatures, resulting in considerable ductility.

The purpose of this study is to resolve existing discrepancies in the literature concerning the creep rate of aluminum oxide, to determine the relative importance of the different possible modes of creep deformation in non-metallics, and to investigate the reasons underlying the fact that creep in single crystal non-metals is apparently controlled by anion diffusion while in polycrystalline non-metals, creep is apparently controlled by cation diffusion.

Work to date has been primarily concerned with the development of facilities for creep testing of aluminum oxide and with the preparation of samples of aluminum oxide and sodium chloride.

#### Creep Testing of Aluminum Oxide

The design and construction of a creep machine for testing cylindrical samples in compression at constant stress in controlled atmospheres up to 1700°C is essentially complete (Figure 1). Special fea-

tures include a constant stress lever arm and a high sensitivity extensometer. The design of the lever arm is discussed in the Appendix. A furnace using silicon carbide heating rods has been designed and built.

The extensometer (Fig. 2) uses sapphire rods to drive a miniature linear variable differential transformer (LVDT), Schaevitz Type Number 100MS-LT, range  $\pm 0.150$  inch. The LVDT will be connected to a Schaevitz Model CAS 2500 carrier-amplifier-demodulator system, which operates at an output voltage of 2.5 vdc and a coil excitation frequency of 2500 cps. The resulting LVDT sensitivity is about 7.5 millivolts per 0.001 inch displacement or 18.7 millivolts per 1.0% strain of a 0.250 inch sample. The alignment and calibration of the load system, furnace and extensometer have not been completed.

Fifty samples of the Lucalox variety of aluminum oxide have been prepared and are ready for testing. The ends have been ground flat and parallel to within 0.0601 inch. The density has been determined by measuring the sample weight and calculating the sample volume from length and diameter measurements. The density ranges from  $3.91 \pm 0.02$  to  $3.98 \pm 0.02$  grams/cubic centimeter. Using a theoretical density of 3.996 grams/cubic centimeter, these values correspond to  $98.0 \pm 0.5$  to  $99.8 \pm 0.5$  percent of theoretical density.

Correspondence is under way with Speedway and Lexington Laboratories to obtain other samples of high purity aluminum oxide.

The intercept technique will be used for quantitatively determining grain size and shape. A study of the statistics of this technique is under way. The evaluation of the amount of intergranular separation in deformed samples will probably require using the lineal analysis

technique.

The technique of differential density measurement<sup>(1)</sup> is under consideration for following the development of intergranular voids. In this technique the density of a standard is compared with that of a sample before and after deformation. Although absolute density is not determined, changes in density smaller than 0.01% can be detected.

#### Creep Testing of Sodium Chloride

Equipment for compressive creep testing of sodium chloride in air or in an inert atmosphere is available and operative.

Single crystal samples of sodium chloride are being grown by the Center for Materials Research, Stanford University, and an extrusion press has been made so that dense polycrystalline samples can be made by extruding single crystals.

## APPENDIX

### A Constant Stress Lever Arm for Compression Creep Testing

#### 1. Description of Lever Arm

Lever arm devices for maintaining a constant stress during tensile creep testing have been designed<sup>(2, 3)</sup> and used in many laboratories, but only one such lever arm for use in compression creep testing appears in the literature<sup>(4)</sup>. It is shown schematically in Figure 3. The load applied to the sample with this lever continually increases as the sample deforms and the lever deflects. The increase in load maintains the stress approximately constant as the sample cross-sectional area increases. The precision with which the stress is maintained constant is a function of the interrelation between the sample length,  $l_0$ , the lever arm length,  $a$ , and the lever angle,  $\gamma$ . The lever arm was designed for samples 0.375 inch long and has a vertical dimension,  $D$ , of 47.30 inches. Stress is maintained constant within  $\pm 1.6\%$  up to 23% engineering strain.

While designing a similar lever for use with samples 0.250 inch long, it was realized that a compact lever arm for use with samples of any length could be made. The new lever arm (Figure 4) has a vertical dimension of 7.312 inches. It was designed for five different sample lengths, 0.125, 0.250, 0.375, 0.500 and 0.625 inches, with two levels of stress precision ( $\pm 1\%$  up to 18% strain;  $\pm 2\%$  up to 28% strain) for each sample length. These ten different conditions correspond to ten different lever angles. The desired lever angle for a given test is chosen by hanging the load from the appropriate notch in the top of the

lever arm. The steel insert in which the notches are cut is removable, allowing another insert with notches cut for still different sample lengths to be installed if desired. For complete flexibility, a notched block could be mounted on a micrometer screw at the top of the lever, allowing easy selection of any desired lever angle.

A counterweight prevents the weight of the lever itself from applying a force to the sample by causing the center of gravity to lie on the axis of rotation. A constant balancing load can be added to counterbalance the weight of the ram testing on the lever arm.

For reproducible force application, each test must start with the lever arm in the horizontal position. This position can be determined with a dial gage or LVDT activated by the lever arm.

The force applied to the sample can be determined by measuring the lever arm deflection and calculating the force. If a force transducer is used in the load system, it must be quite stiff, for any deflection in the transducer will cause the lever to deflect and apply excess force to the sample.

The advantages of constant stress lever arms are as follows. They apply a continuously corrected load to the sample as opposed to the discontinuous corrections achieved by periodically changing the load in a dead load system (Figure 5). This is an advantage because no artificial discontinuities are introduced into the test results. It is also an advantage from a personal point of view, eliminating the need for constant attendance of the creep machine during overnight tests.

Continuously corrected loads can also be applied to samples by properly programmed servohydraulic or servoelectric testing machines,

but the lever arm is a very much cheaper alternative.

## II. Derivation of Lever Arm Design

The appropriate lever angles were determined by setting the maximum deviation of the actual force from the desired force equal to 1 and 2 percent and solving for the lever angle for given sample lengths. The desired force on the sample is given by

$$F = \frac{1}{1 - e} F_0$$

where  $F_0$  equals the initial force and  $e$  equals the engineering strain in the sample. The force developed by the lever arm is given by

$$F = \frac{\sin(\gamma + \theta)}{\sin \gamma} F_0$$

where  $\gamma$  equals the lever angle, and  $\theta$  equals the lever deflection (Figure 3).

$$\theta = \frac{\Delta l}{a} = \frac{l_0}{a} e$$

where  $\Delta l$  equals the change in sample length during deformation,  $l_0$  equals the initial sample length, and  $a$  equals the length of the lever arm.

The following table lists values of the lever angle,  $\gamma$ , as a function of the sample length,  $l_0$ , divided by the lever arm length,  $a$ , for  $\pm 1\%$  and  $\pm 2\%$  deviation.

$\frac{l_0}{a}$	$\gamma$	
	$\pm 1\%$	$\pm 2\%$
.1	4°53'	4°26'
.2	9°42'	8°38'
.3	14°23'	12°50'
.4	18°54'	16°51'
.5	23°08'	20°45'



The length of the lever arm,  $a = 1.25$  inches, was chosen such that the maximum expected deflection,  $\Delta l = 0.15$  inch, causes less than 1% change in  $a$ .

The vertical dimension of the lever arm,  $D = 7.3125$  inches, was chosen to give a 1:1 lever ratio for  $\gamma = 9^{\circ}42'$ , corresponding to a sample length of 0.250 inch and a force deviation of  $\pm 1\%$ .

The determination of the above values of the lever angle involved mathematical approximations which are currently being rechecked. When this is completed and when the lever arm is actually put in use and evaluated, a complete report on the design and the performance of the constant stress compression lever arm will be written.

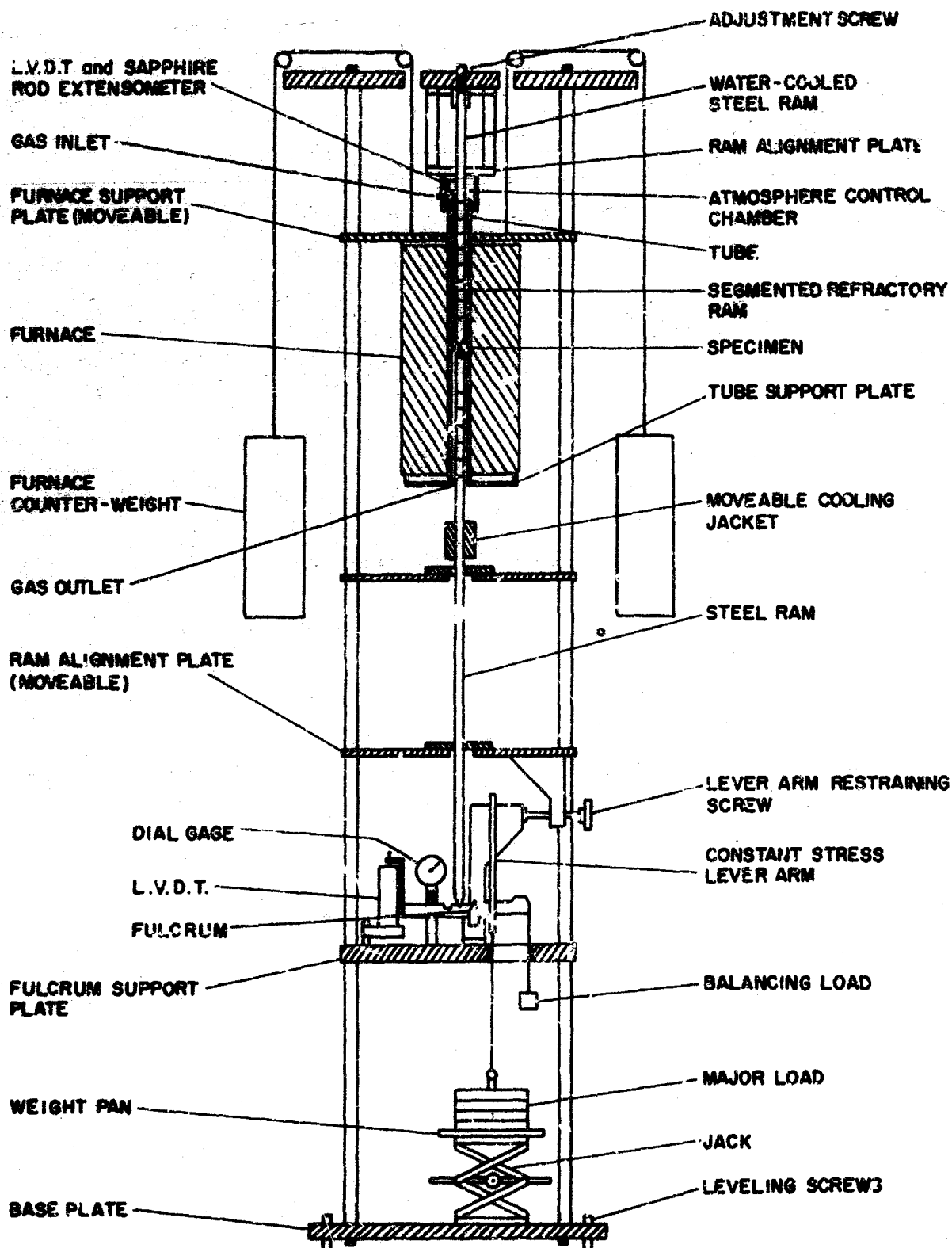


Fig. 1. Creep apparatus for compression testing at constant stress in controlled atmospheres at high temperature.

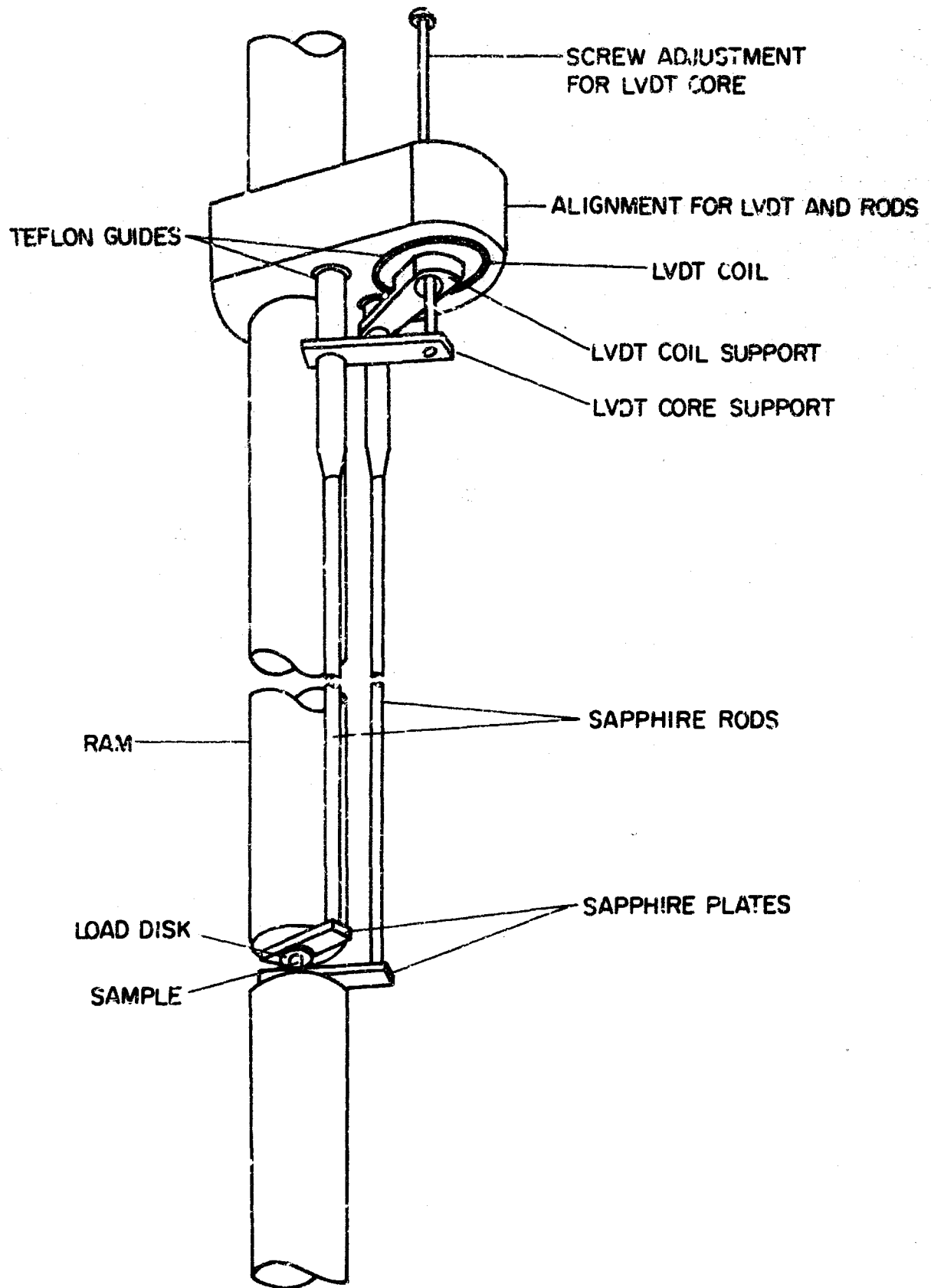


Fig. 2. Extensometer for use at high temperature.

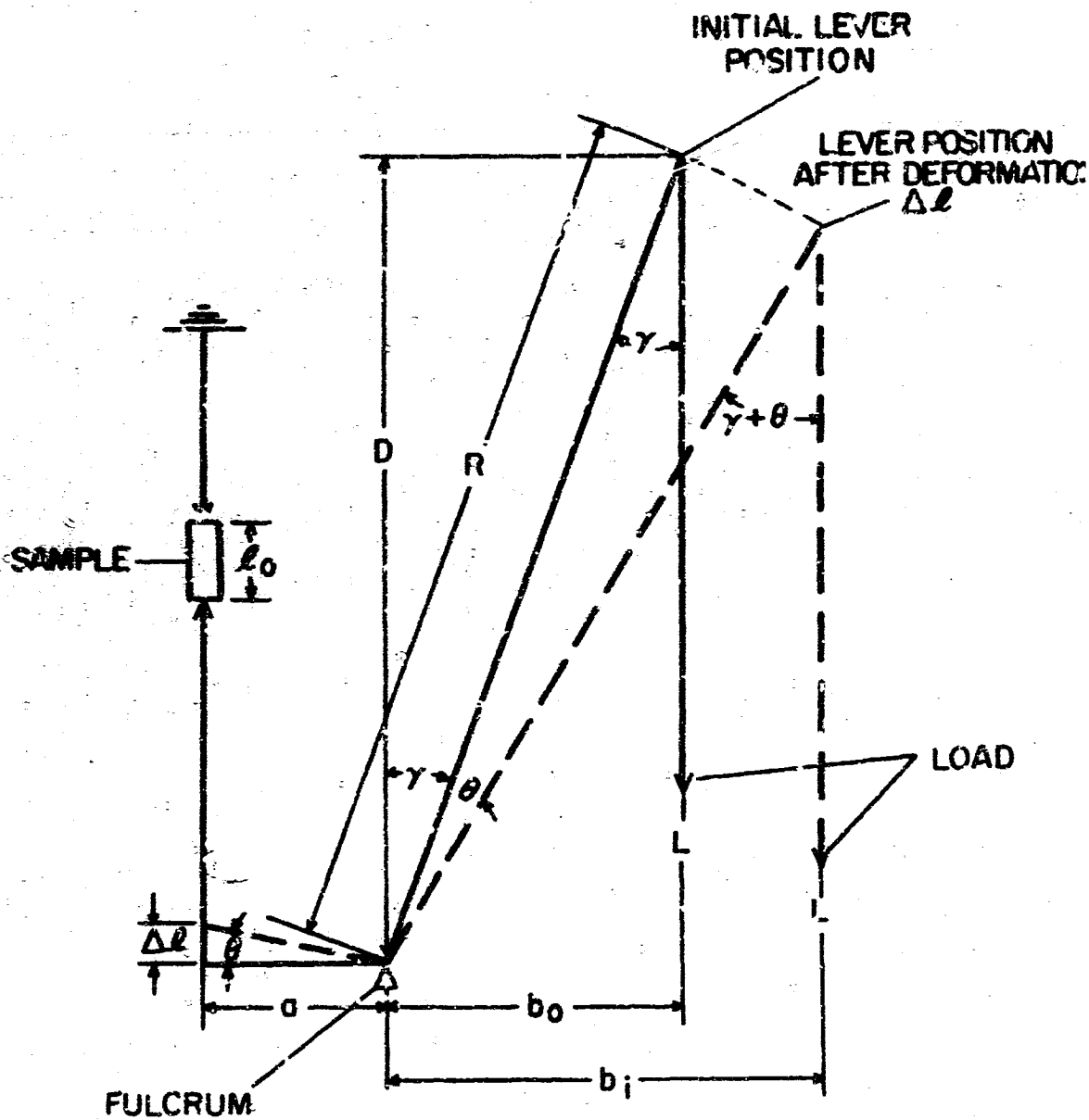


Fig. 3. Schematic drawing of constant stress lever arm showing the lever arm in the initial position and in a position resulting from deformation of the sample.

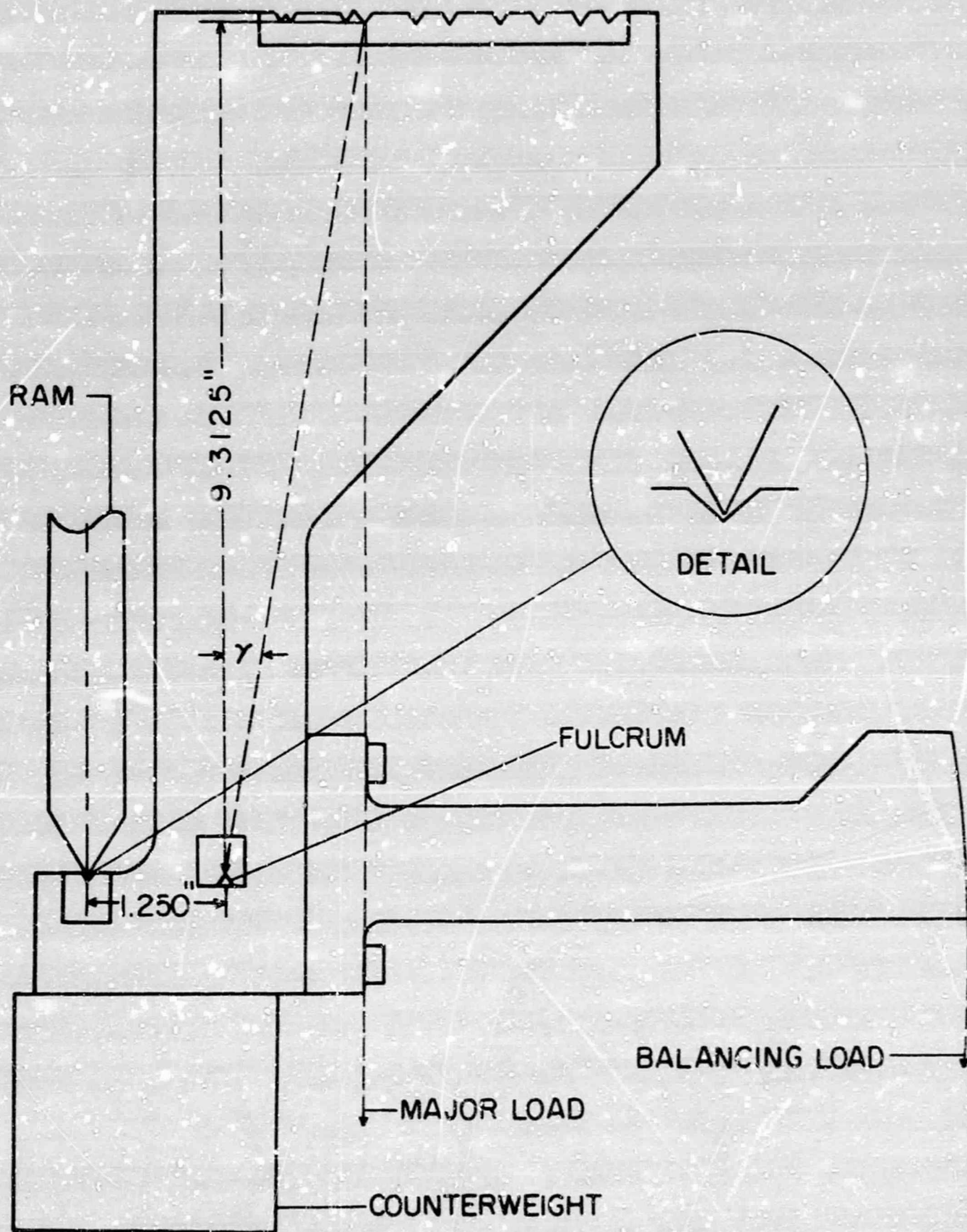


Fig. 4. Constant stress lever arm for compression creep testing.

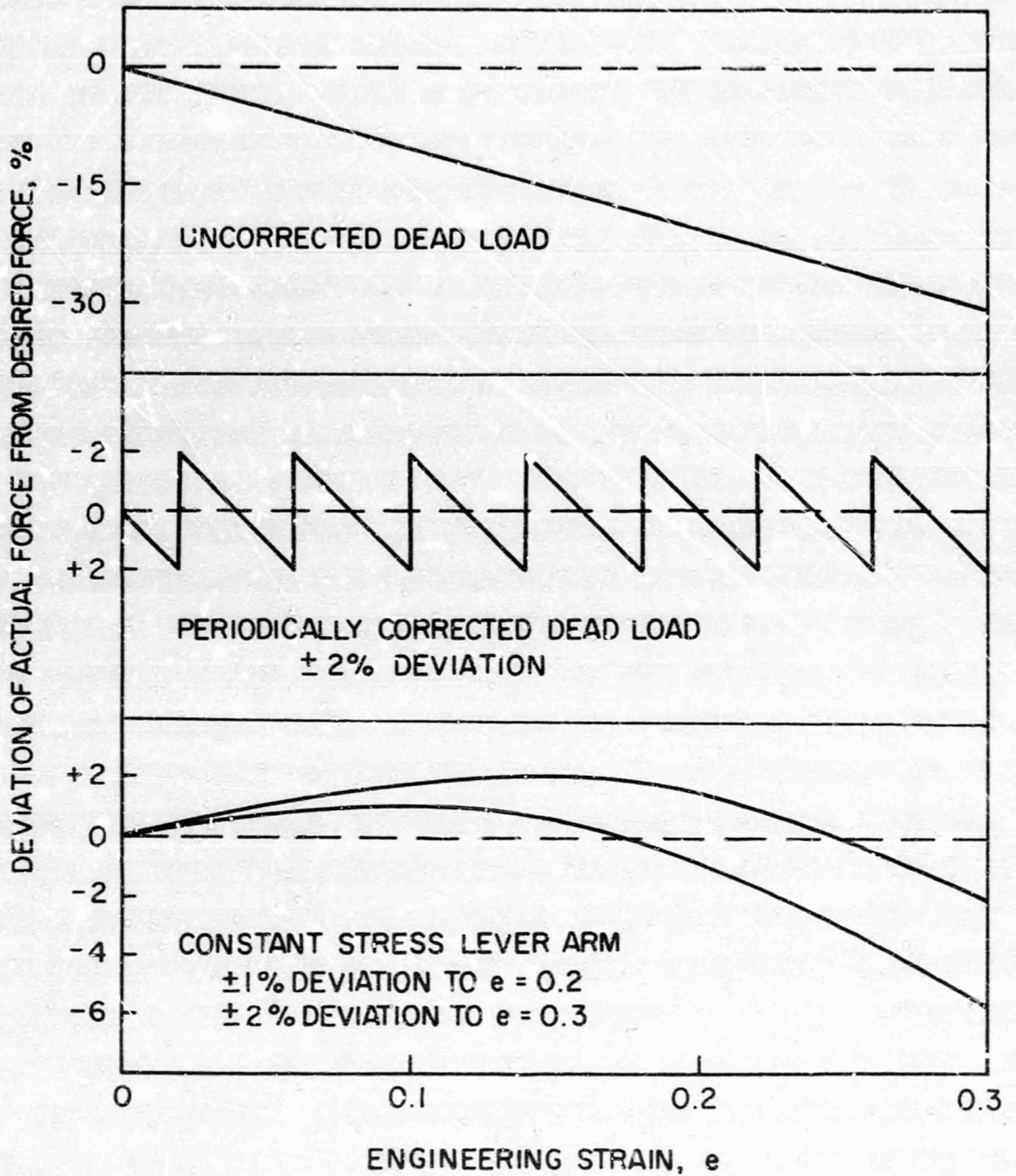


Fig. 5. Deviation of the force applied to a creep sample from the force which would maintain a constant true stress on the sample for three different methods of force application.

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