A New Hydrostatic Compression Tester for Anisotropic Filled Polymers

ALBERT H. LEPIE and MARK B. MORAN, *Chemistry Division, Research Department, Naval Weapons Center, China Lake, California 93555*

Synopsis

This paper describes a testing device designed for measuring the isotropic or anisotropic hydrostatic compression properties of filled polymers like solid propellants, including the initial void content and the bulk modulus. The propellant test samples used are either cubical, cylindrical, or rectangular specimens. Three pressure-sealed linear variable differential transformers (LVDTs) are used to monitor the dimensional changes of a cube specimen during hydrostatic pressurization simultaneously in the XYZ directions. The moduli E_{11}, E_{22}, and E_{33} and the compression strain ratios ν_{21}, ν_{32}, and ν_{31} can be determined as a function of pressure. The three LVDTs can also be used to measure the length changes of three rectangular or cylindrical test specimens under hydrostatic compression only in the vertical Z direction. The bulk modulus and void content can be computed when isotropic behavior is assumed. The entire test procedure is controlled by an Apple II microcomputer via an AI13 12-bit analog input system. Some typical test results obtained with undamaged and damaged propellant samples are described.

INTRODUCTION

The volume compression properties of viscoelastic materials under an applied hydrostatic pressure (P) are described by the bulk relaxation modulus $K(t)$.[1] The volumetric strain is usually defined by the relative volume change $\Delta V/V_0$. If the hydrostatic pressure changes as a function of time and the volume response follows immediately, then the bulk modulus is given by

$$K(t) = -P(t)/(\Delta V/V_0) \tag{1}$$

The bulk viscoelasticity of unfilled polymers is of interest only at temperatures below $T_g + 20°C$. Volume changes at higher temperatures are too fast for a convenient observation. Highly filled polymers, like solid propellants, display a slower viscoelastic response at room temperature than unfilled polymers do.

The bulk relaxation modulus of propellants at constant pressure then becomes

$$K(t) = -P/(\Delta V/V_0)(t) \tag{2}$$

The bulk creep compliance is defined by

$$B(t) = 1/K(t) \tag{3}$$

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Several devices for measuring the hydrostatic bulk compression properties of polymers have been described in the literature.[2-6] Some of the devices, suitable for higher pressure, utilize linear variable differential transformers (LVDTs) or strain gauges for measuring volumetric changes under hydrostatic pressure.

The hydrostatic compression device described in this paper uses a similar technique. The test specimen is immersed under an inert fluid and is pressurized by a nitrogen gas phase above the fluid. The dimensional changes of the sample are monitored by LVDTs. Compression experiments with propellants under nitrogen gas, instead of a fluid resulted in hydrostatic compression curves that indicate a possible diffusion of gas into voids at higher pressure.

APPARATUS AND TEST SPECIMENS

A drawing of the hydrostatic compression tester is shown in Figure 1. It consists of a 1-gal pressure vessel (K) with a rating of 3000 psi, manufactured by the Parr Co.[7] The lid of the vessel was modified by the same company and holds a pressure gas inlet (A), a rupture disk (B), a thermal well (L), and 18 electrical feed-throughs (C). Three pressure sealed linear variable differential transformers (LVDTs) (E) are attached to three aluminum sam-

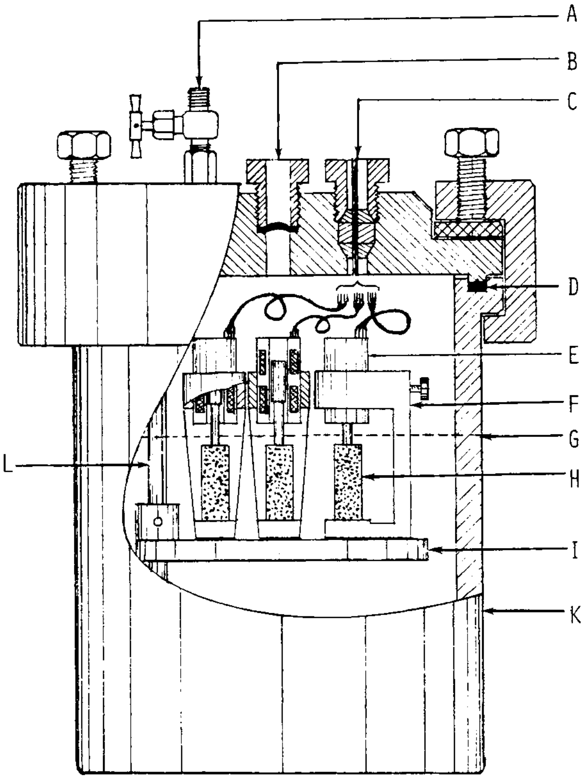

Fig. 1. Device for measuring hydrostatic compression properties of anisotropic materials (set-up for three isotropic specimens).

ple holders (F) and are used for monitoring the dimensional changes of either three single samples (H) in the *Z* direction or a cube specimen simultaneously in the *XYZ* directions. The LVDTs and signal conditioners are manufactured by Schaewitz.[8]

The specimen holders with the test samples attached are supported by a table (I) that can be vertically adjusted along the tubing of the thermal well (L). The specimens are immersed under Fluorinert FC-43[9] with a liquid level as indicated by the dashed line (G). Fluorinert does not cause any swelling of the propellant within 4 days. Silicone grease was used to attach the cylindrical specimens to the sample holders and LVDT cores.

The assembly of a cube specimen with three LVDTs attached in the XYZ directions is illustrated in Figure 2. The core of the LVDTs (A) and the bottom of the aluminum sample holders (B) are glued to the cube specimen (C) with 5-Minute Epoxy. Special care is taken to use only a thin layer of epoxy to minimize the amount of air bubbles between the metal surface and the test sample.

The entire test procedure is controlled by a microcomputer via an AI13 analog input system,[10] which makes the test results independent of the operator.

COMPUTER PROGRAM

The AI13 12-bit, 16 channels A/D Analog Input System was used for DC voltage readings from the three LVDTs' signal conditioners into the memory of a 48K RAM APPLE II+ microcomputer. The AI13 A/D card has seven bipolar sensitivity ranges from 0 to ±5 volts.

The computer program for an automation of the compression test was written in Applesoft Basic and calls for an assembly language subroutine, which takes subsequential voltage readings from each channel addressed at a rate of 17 readings/s. The subroutine is a part of the software, supplied by Interactive Structures, Inc. The computer takes 20 voltage readings per data point at the existing pressure and stores the arithmetic mean value. The program then displays the next higher pressure setting on the screen and the gas pressure must be adjusted by an operator via a regulator valve. After a delay time of 70 s, the program takes the next set of voltage readings, and so on. When the pressure reaches 900 psi, a depressurization sequence in 100 psi steps to ambient pressure is performed, while the voltage reading sequence continues.

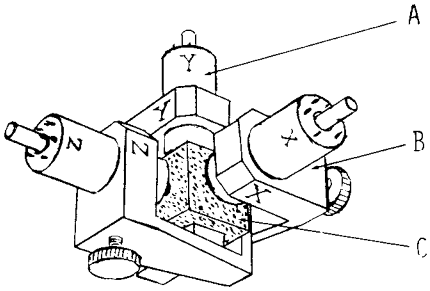

Fig. 2. Cube assembly with three LVDTs.

The millivolt data are then stored on disk files and can be retrieved for evaluation. The bulk modulus is computed from the slope of the volume vs. pressure curve between 400 and 900 psi. The initial void content is determined from the intersect of the bulk modulus curve with ambient pressure. The program also has routines for calibration of the LVDTs, printouts of tables, and plotting of the results via an HP 7225B XY plotter.

CALIBRATION OF THE LVDTS

The three pressure sealed LVDTs are controlled by three signal conditioners[8] that apply a high frequency voltage to the LVDTs and demodulate their output with an accuracy of ±15 VDC/±0.1 in. displacement. Calibration of the LVDTs is carried out by means of a micrometer device with 0.0001 in. accuracy. The core of the LVDT is advanced in steps of 0.0005 in. by the micrometer. The computer averages the output voltages of 20 readings per core setting and calculates the linear regression of the displacement vs. millivolt curve. The slope of the straight line in in./mV is the calibration constant C_1. The sensitivity of the Schaewitz LVDTs is approximately 145 mV/0.001 in. core displacement and linear.

An additional constant C2 in the computer program considers the pressure dependency of the LVDT performance. The constant C2 is determined by running a pressurization test with 1.2 in. long glass rods in place of the propellant samples. The glass rods are pressurized at the same pressure sequence as applied to the real samples, and the voltage response is monitored by the computer. Glass has a bulk modulus of approximately 3×10^6 psi. A graphical interpolation method is used to find the constant C_2 that results in the bulk modulus of glass. The hydrostatic volume compression curve obtained with a glass rod is shown in Figure 3. The bulk modulus is close to 3×10^6 psi.

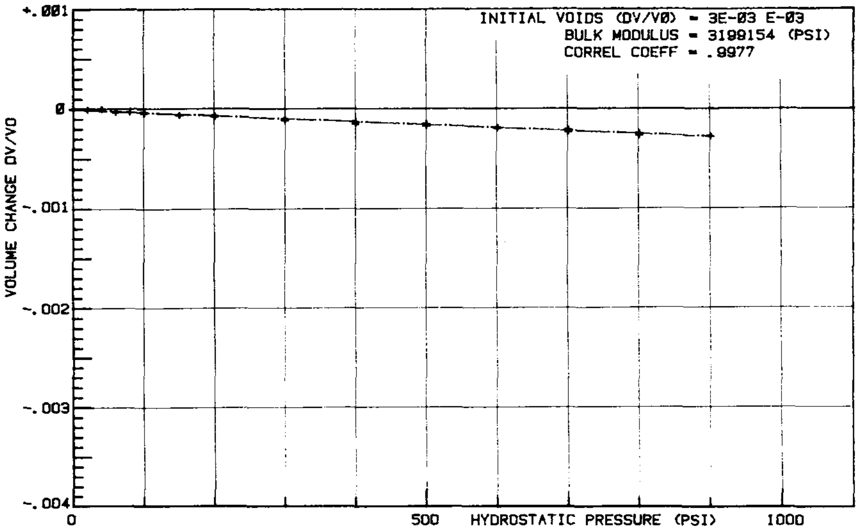

Fig. 3. Hydrostatic compression properties of glass rods.

EXPERIMENTAL

A series of hydrostatic compression tests were carried out with an undamaged and damaged HTPB composite propellant, designated MK-56B. The propellant contained 85% solids and 14% hydroxyl-terminated polybutadiene as a binder.

Two kinds of propellant specimens were used: cube specimens with approximately 1.1 in. side lengths for measuring the dimensional changes under pressure simultaneously in the *XYZ* directions, and cylindrical samples (1.2 in. long with 0.4 in. diameter) for measuring the length changes only in the *Z* direction. The cube specimens were prepared from 0.5-gal cartons by guillotine cutting. The cylindrical samples were obtained from end-bonded rectangular tensile specimens that were machined into a cylindrical uniaxial middle section.

The pressurization was carried out in 70-s time intervals, first in small steps of 20 psi and then in steps of 100 psi up to a pressure of 900 psi and back to ambient pressure. Needle valves were used for a convenient pressure adjustment. The highest volume changes were registered at low pressures at the beginning of the test when the voids in the propellant collapse.

In a previous experiment, the densities of propellant samples were measured before and after the pressurization test. The void contents computed from the density differences were in close agreement with the hydrostatic compression results obtained with LVDTs.

HYDROSTATIC COMPRESSION PROPERTIES OF A PROPELLANT CUBE

Propellant cubes with 1.1 in. side lengths were prepared by guillotine cutting. The three LVDTs were attached to the cube specimens with 5-Min Epoxy, as shown in Figure 2. The cube with the LVDTs attached was then placed into the pressure vessel and pressurized in steps at constant time intervals of 70 s.

Two repeated pressurization tests were carried out with the same test specimen. The length changes obtained in the *XYZ* directions of the undamaged cube during the first and second hydrostatic compression tests are plotted vs. pressure in Figure 4. The three solid curves indicate unusual hydrostatic compression properties of the undamaged propellant. The slope of the curves in one direction of the cube is steeper than in the other two directions. The test was repeated with two other undamaged propellant cubes that were prepared from the same propellant carton. The test results were confirmed with the second test.

The results of the second pressurization test with the same specimen are plotted as dashed curves in Figure 4. It appears that the voids in the propellant have collapsed during the first compression experiment. The time between the two tests was 2 h.

We wanted to determine if a propellant cube becomes more anisotropic after damage in the *Z* direction. An undamaged propellant cube was subjected to a repeated compression experiment on an Instron tester at 1 cm/min crosshead speed in order to produce damage. The repeated compression stress–strain curves are shown in Figure 5. The first compression curve

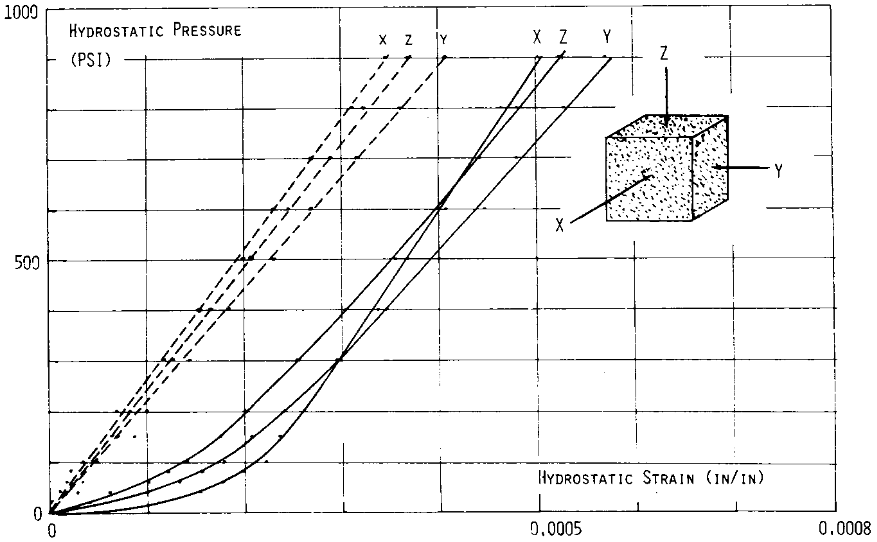

Fig. 4. Hydrostatic compression properties of an undamaged propellant cube at repeated pressurization; (—) first compression; (- - -) second compression.

indicates the properties of the undamaged propellant. The path of the second curve reflects the damage done to the propellant cube during the first compression experiment.

The damaged cube sample was then allowed to recover for 3 days in a stressless state at room temperature. The hydrostatic compression properties were then measured. The results are presented as solid curves in Figure 6. It appears that the damaged propellant cube displays moderate anisotropic properties in the *XYZ* directions. After 2 h, a second hydrostatic pressurization test was carried out with the same cube specimen, and the results are shown as dashed curves. Again, the voids have collapsed, and the cube became nearly isotropic.

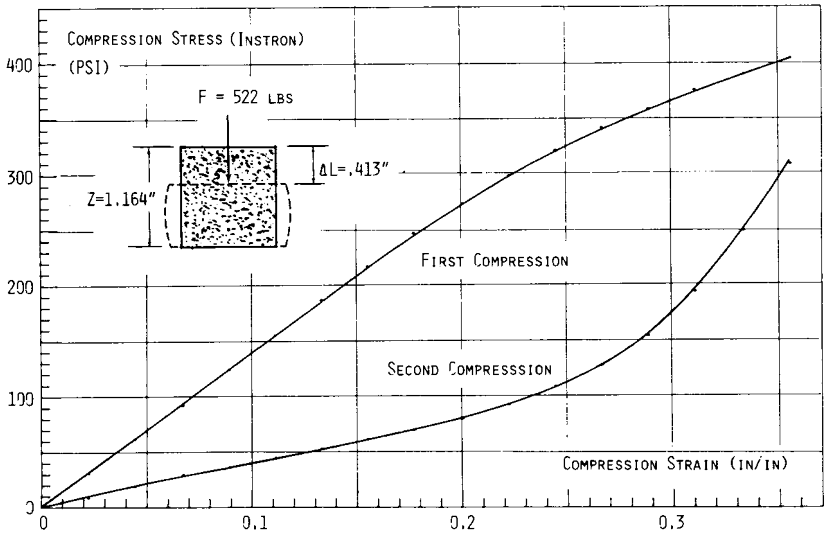

Fig. 5. Preparation of a damaged propellant cube by repeated compression loading.

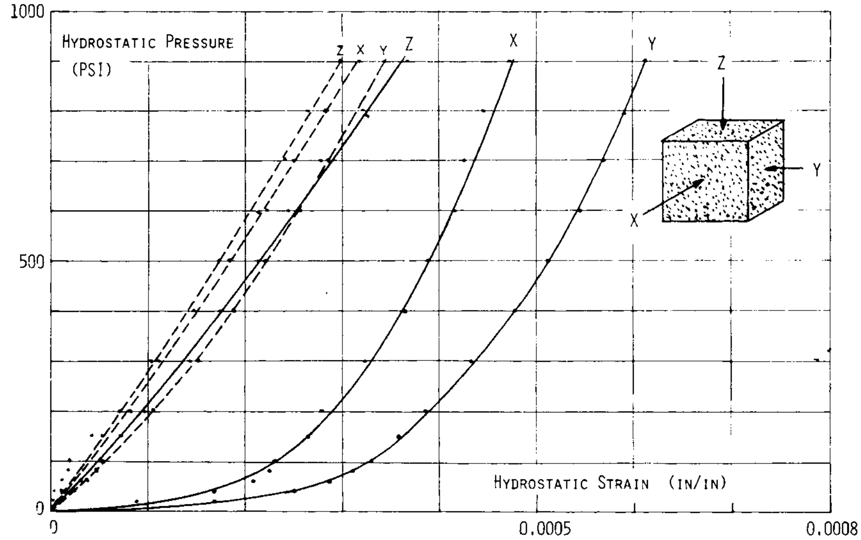

Fig. 6. Hydrostatic compression properties of a propellant cube at repeated pressurization. Cube was damaged in the Z direction: (—) first compression; (- - -) second compression.

The volume changes of the damaged cube during the first and second hydrostatic compression were computed from the length changes in the XYZ directions, assuming anisotropic behavior.

$$\Delta V/V_0 = \Delta X/X_0 + \Delta Y/Y_0 + \Delta Z/Z_0 \tag{4}$$

Figure 7 shows the relative volume changes of the damaged propellant cube during the first and second hydrostatic compression experiment. The bulk moduli were computed after eq. (2) from the slope of the straight lines of the volume vs. pressure response. The initial void content of the damaged

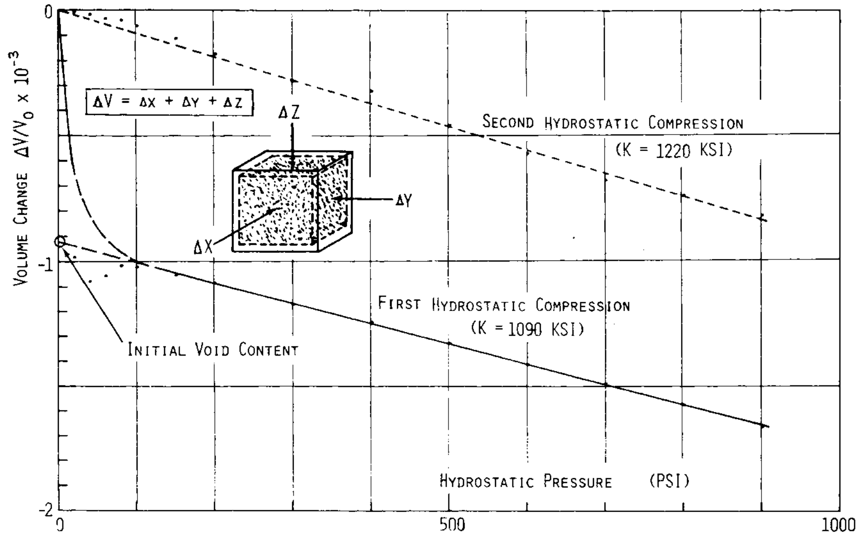

Fig. 7. Volume changes during repeated compression of a damaged propellant cube. Determination of the bulk modulus (K).

propellant was determined from the intersect of the bulk modulus curve with ambient pressure. The damaged propellant cube had a void content of 0.092% of its volume.

HYDROSTATIC COMPRESSION OF RECTANGULAR PROPELLANT SAMPLES

Similar repeated hydrostatic compression tests were carried out on undamaged and damaged rectangular propellant specimens, 1.25 in. long and 0.4 in. in diameter. The damaged specimen was obtained from the broken end pieces of a uniaxial sample that had been stretched to failure at a rate of 5 cm/min on an Instron tester. The hydrostatic compression properties of these samples were measured only in the *Z* direction. In this way, three specimens can be tested simultaneously.

Figure 8 shows the effect of tensile damage upon the hydrostatic compression properties in the *Z* direction of the propellant. The damaged propellant displays a higher hydrostatic compressibility than the undamaged propellant because of the formation of voids due to dewetting.

After 2 h, a second compression test was made with the same three samples in place. The results are plotted as dashed curves and indicate that the voids in the samples have collapsed. A comparison of the solid and dashed curves indicate a hardening effect that was caused by the first hydrostatic compression.

CONCLUSIONS

The hydrostatic compression tester described in this paper is a useful tool to determine the anisotropic properties of a solid propellant cube specimen in the *XYZ* directions. This information cannot be obtained from dilatometer measurements under hydrostatic compression.

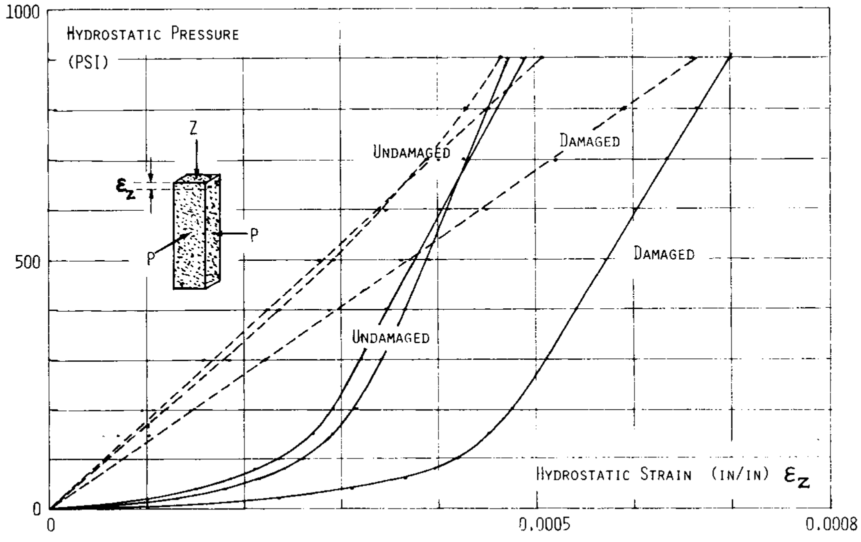

Fig. 8. Hydrostatic compression of undamaged and damaged rectangular specimens in the *Z* direction: (—) first compression; (- - -) second compression.

Propellant cube specimens that were prepared from a carton by guillotine cutting are anisotropic. We found that the hydrostatic compressability is lower in one direction than in the other two directions. (Fig. 4). This behavior was confirmed by two repeated tests with other propellant cubes from the same carton. It was not determined if this particular direction coincides with the direction of casting of the uncured propellant into the carton.

If a virgin propellant cube is subjected to damage by a compression in the Z direction on an Instron tester, it becomes highly anisotropic and less compressable in this particular Z direction.

As can be expected, the voids in a propellant collapse under hydrostatic compression. The voids do not reopen during 3 days of recovery after the pressure was released. A repeated hydrostatic compression of the same sample results in nearly linear compression properties, as shown by the dashed curves in Figures 4 and 6–8.

Additional experiments should be carried out to determine how the casting process contributes to the anisotropic compression properties of a propellant carton or propellant grain.

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