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New Test Method to Determine Modulus of Elasticity of Rocket Grain Material

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A new testing technique was demonstrated for measuring the modulus of elasticity of low-modulus materials such as rocket propellant grains. The technique described herein offers substantial advantages over the dog bone testing method. The testing procedure is less complicated, and data reduction is simpler. More importantly, a biaxial stress field can be established in the test specimen to simulate conditions encountered during pressurization of a nonlinear and viscoelastic material (e.g., propellant used in solid rocket motors). For metals or linearly elastic materials, dog bone testing may be considered a viable technique through which material properties such as the modulus of elasticity can be extracted accurately. However, for highly filled materials or propellants that exhibit nonlinear and viscoelastic behavior, results obtained through the uniaxial stress data of dog bone tests do not provide material characteristics under biaxial strain conditions. Lack of such material properties will be detrimental to the ability in evaluating and predicting the integrity of the components.

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The technique uses a thick, circular disk constrained at its outer diameter surface and loaded uniformly over one circular face by gas pressure. The technique was demonstrated by using a circular disk cast from EZ-Cast™ 521 thermosetting plastic material. Normal displacement at the center of the disk as a function of static pressure was measured. Modulus of elasticity versus deformation calculations were made using a closed-form solution and finite-element computer codes. Modulus values of 300 and 210 psi were obtained using the two approaches, respectively, based on the measured center displacement. These values correlate reasonably well with the previously generated modulus value of 250 psi using the uniaxial "dog bone" method. As expected, the dynamic pressurization tests yield a higher modulus than that obtained by static pressure. However, a change of pressurization rate from 0.1 to 90 psi/s gives only a 10% increase in modulus.

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PREFACE

The authors are grateful to Dr. Sherrie L. Zacharius for her suggestions regarding the determination of glass transition temperature and the dynamic modulus of EZ-Cast™ material and the extensive discussions regarding the behaviors of polymeric materials. We also wish to thank Emil M. Kaegi for his assistance in acquiring the data and Dr. Munson A. Kwok for the technical discussions during the preparation of the manuscript. In addition, Tim J. Bixler and Randy L Williams' efforts in preparing the cast samples, and Mr. Robert Shenk's efforts in performing DSC and rheovibron tests for several EZ-Cast™ specimens are appreciated.

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I. INTRODUCTION

The determination of the modulus of elasticity of low-modulus materials such as rocket propellants by the conventional, uniaxial "dog bone" technique has certain limitations. The specimens need to be cast or fabricated precisely into the required dimensions. Because of the low modulus (high compliance) of the material, prestresses may exist in the specimen before testing as a result of alignment. In addition, the stiffness of the deformation measuring device, such as an extensometer, has been shown to be comparable in magnitude to that of the specimen.[1] The term "stiffness" is defined here as the ratio of the applied force to the deflection in a linear structural element, e.g., the K value in $F = Kx$ for a linear spring. Consequently, this stiffness needs to be determined experimentally and extracted from the measured overall stiffness. This procedure undoubtedly complicates the dog bone testing and data reduction.

One important characteristic of nonlinear viscoelastic materials such as rocket propellants in SRMU is that their mechanical response under biaxial stresses cannot be accurately derived or predicted from uniaxial test data. Thus, the response under biaxial stresses needs to be experimentally measured. This necessitates biaxial testing to generate the mechanical properties for these materials. Lack of these properties will lessen the ability to evaluate and predict the operational integrity of the components.

A new test technique has been demonstrated that alleviates many of the above-described problems. This method uses a thick, circular disk made of a low-modulus polymer material. The disk is constrained at its outer cylindrical surface and loaded uniformly over one circular face by gas pressure. The normal displacement at the center of the disk is measured by a displacement gage.

The diameter of the disk specimen is based upon the dimensions of the test chamber and the fixtures. The thickness of the specimen was chosen by considering the requirements necessary for measurement and analysis. Generally speaking, the diameter of the disk specimen should be sufficiently large so that the normal displacement at the center of the specimen can be accurately measured. The specimen needs to be thick enough such that no nonlinear large deformations occur and yet thin enough so that the theory of plates can be applied. It is judged that the chosen thickness satisfies these requirements.

This report presents a description of the test arrangement, specimen configuration, experimental data, and the results of a closed-form solution and a finite element analysis from which the value of the modulus of elasticity for EZ-CAST™ 521 is determined.

II. EXPERIMENTS

A. PREPARATION OF SAMPLE

To explore the viability of the proposed new method, a circular disk of 2-in. thickness and 5.5-in. diameter cast from EZ-Cast™ 521 (AREMCO Products, Inc.) was used. EZ-Cast™ 521 is a thermosetting, rubber-like plastic commonly used for making molds for metal castings. The product has a milky appearance. Before casting, this product, in liquid form, was placed into a vacuum chamber to eliminate the entrapped air as much as possible. After vacuum treatment, the material was almost clear. The rubber disk was cast in and bonded to the inner surface of a hollow PVC tube. The PVC tube was 2 in. high, 5.5 in. inner diameter (ID) and 6.5 in. outer diameter (OD). The entire disk and PVC tube were mounted in a pressure test fixture. Figure 1 shows the dimensions of the sample and the PVC tube assembly.

During the casting of the disk, the PVC tube was closed with a bottom and a top lid. The top lid had two sprues to allow for the complete filling of the mold and any thermal expansion of the casting during curing. The mold was then cured in an oven at 250°F. The curing time was 2 hr for the disk specimen. Because PVC softens at 121°C (250°F), care was taken to avoid losing the shape of the tube during the curing operation.

The disk and its PVC tube were allowed to cool slowly to room temperature to reduce residual thermal stresses in the disk. During cooling, small surface fissures appeared on the surface of the EZ-Cast. To produce a smooth face on the disk, the circular faces were covered with an additional thin layer of EZ-Cast. The disk was then reheated to 121°C (250°F) for 1/2 hr to cure the added material. After the second cure, the OD of the PVC tube was machined to the final dimensions necessary to fit into the pressure cell.

The bond between the EZ-Cast 521™ disk and the PVC retaining tube was found to be excellent after curing. The disk was pressure tested with 100 psi nitrogen gas, resulting in a total load of 9500 lb without delamination at the OD.

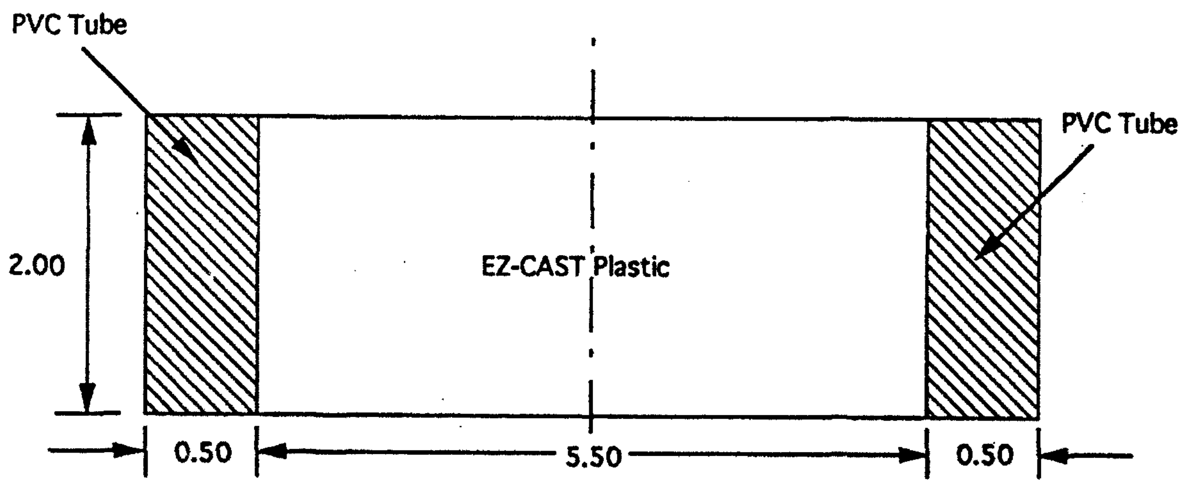

Figure 1. Geometric configurations of EZ-CAST™ specimen and PVC tube.

B. DESCRIPTION OF THE APPARATUS

Figure 2 shows the pressure cell with the specimen installed. The volume of the pressurized region above the sample was kept small (about 100 cm^3, or 6.1 in^3) to permit fast filling with the gas. A calibrated, linear, electronic extensometer was used to measure the center deflection of the disk. The pressure was measured by an electronic pressure transducer. The accuracy of the pressure measuring capability is better than 0.1 psi. Data from both sensors were registered by an automated data collection system utilizing a PC. The computer could be manually activated to take data for static tests or could be also run in an automatic mode to take 20 sample pairs per second for dynamic tests. In the dynamic mode, the computer also activated a fast electric valve to admit the pressurizing gas into the chamber. Calibration factors for both sensors were applied to the data by the computer.

C. STATIC TESTS

A series of static load tests of the disk was performed. The results of four separate tests are shown in Fig. 3. All data fall along a linear deflection vs pressure line. The resolution of the deflection measurements was $\pm$ 0.005 in. The average value of the pressure-to-deflection differential ratio was (57.2 $\pm$ 1.3) psi/in.

D. DYNAMIC TESTS

Dynamic pressure tests were conducted using a realistic pressurization rate such as that measured in the SRMU PQM-1 firing test and to determine the modulus change as a function of pressurization rate. The purpose of the tests was to show that the test fixture is capable of simulating high pressure rates.

In these tests, the disk was subjected to rapid pressure changes ranging from near static (0.1 psi/s) to 90 psi/s. Figure 4 shows the results of a number of these tests. As expected, the material appeared slightly stiffer when subjected to rapid pressure increases. However, the deflection vs pressure relationship remained linear and was a weak function of the pressurization rate.

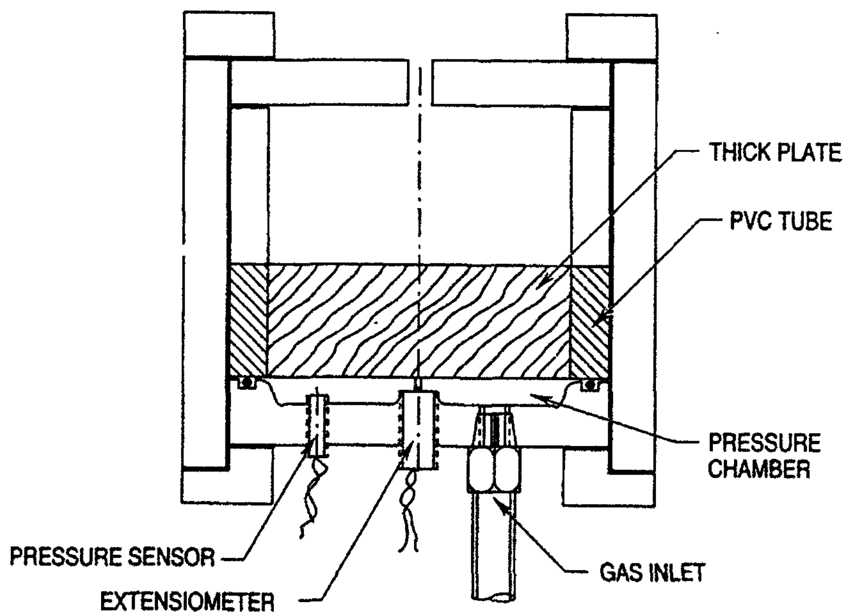

Figure 2. Schematic of pressure cell for disk testing.

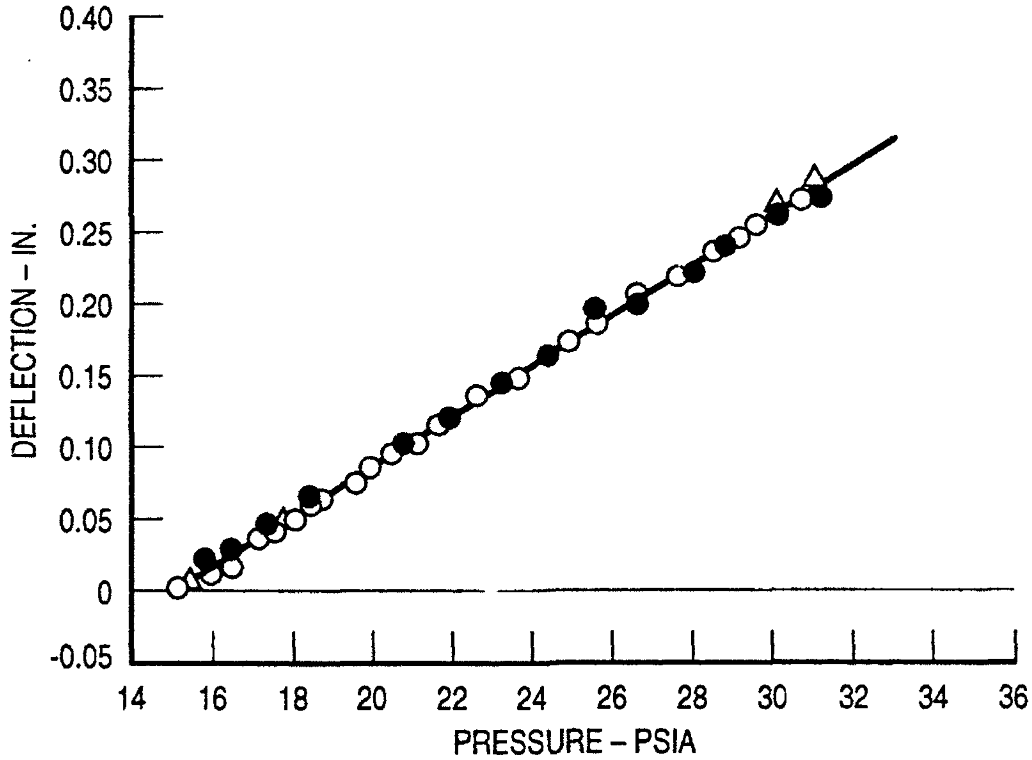

Figure 3. Deflection at center of disk vs static pressure.

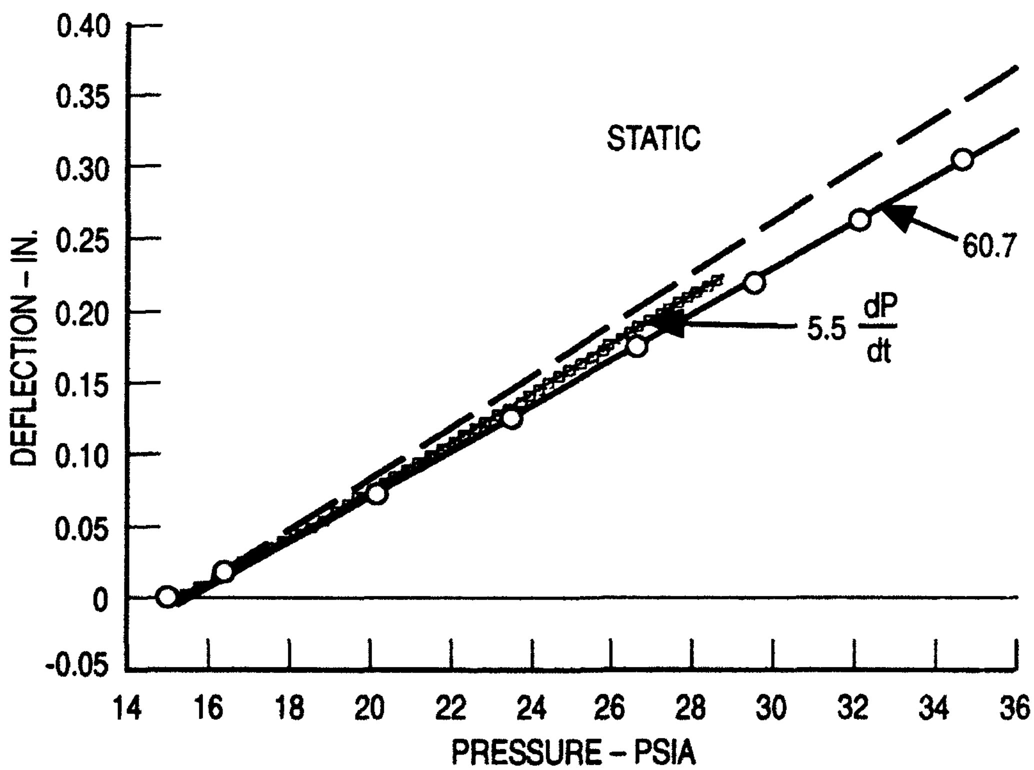

Figure 4. Deflection at center of disk vs dynamic pressure.

III. ANALYTICAL CALCULATIONS

A. APPROXIMATE CLOSED-FORM SOLUTION

The normal displacement at the center of a thick circular disk that is rigidly held at its outer cylindrical surface and loaded uniformly over one face is determined approximately by[2]

$$U_o = \frac{3P}{16}\left[\frac{a^4(1-\nu^2)}{Eh^3}\right]\left[1+\frac{4h^2}{a^2(1-\nu)}\right], \tag{1}$$

where

U_0 (in)	=	the normal displacement at center,
h (in)	=	thickness of the disk,
a (in)	=	disk radius,
E (psi)	=	Modulus of elasticity of the disk material,
ν	=	Poisson's ratio of the disk material. It is assumed to be 0.5 here, and
P (psi)	=	applied uniform pressure.

Inserting the values of the specimen dimensions into Eq. (1), the relation of E, P, and U_0 is expressed as

$$E = 5.2592\ P/U_0\ . \tag{2}$$

By applying the (57.2 ± 1.3)psi/in. slope for P/U_0 to Eq. (2), a modulus of elasticity value of 301 ± 5 psi is obtained. This value is 20% higher than the 250 psi determined by "dog-bone" tests of the same material.[1]

Values for the modulus determined by Eq. (2) from the dynamic test results of Fig. 4 are depicted in Fig. 5. In Fig. 5, the E values for a number of dynamic tests are plotted against dp/dt, the rate of pressure increase. It is seen that the measured increase in E is about 10% for a change in dp/dt of 3 orders of magnitude.

B. FINITE-ELEMENT DISPLACEMENT ANALYSIS

A linear, elastic, finite-element analysis was performed for the EZ-Cast™ 521 rubber disk to predict the displacement response to the application of a uniform pressure at one surface of the disk. The results of this analysis and the experimental displacement data serve as a viable tool in determining the Young's modulus, E, of the rubber material. Two computer codes were used for the analysis. The first is an Aerospace-developed elastic-plastic code, "SAAS III," for axisymmetric or plane-strain (stress) solids.[3] The second is the "ABAQUS" code developed by Hibbitt,

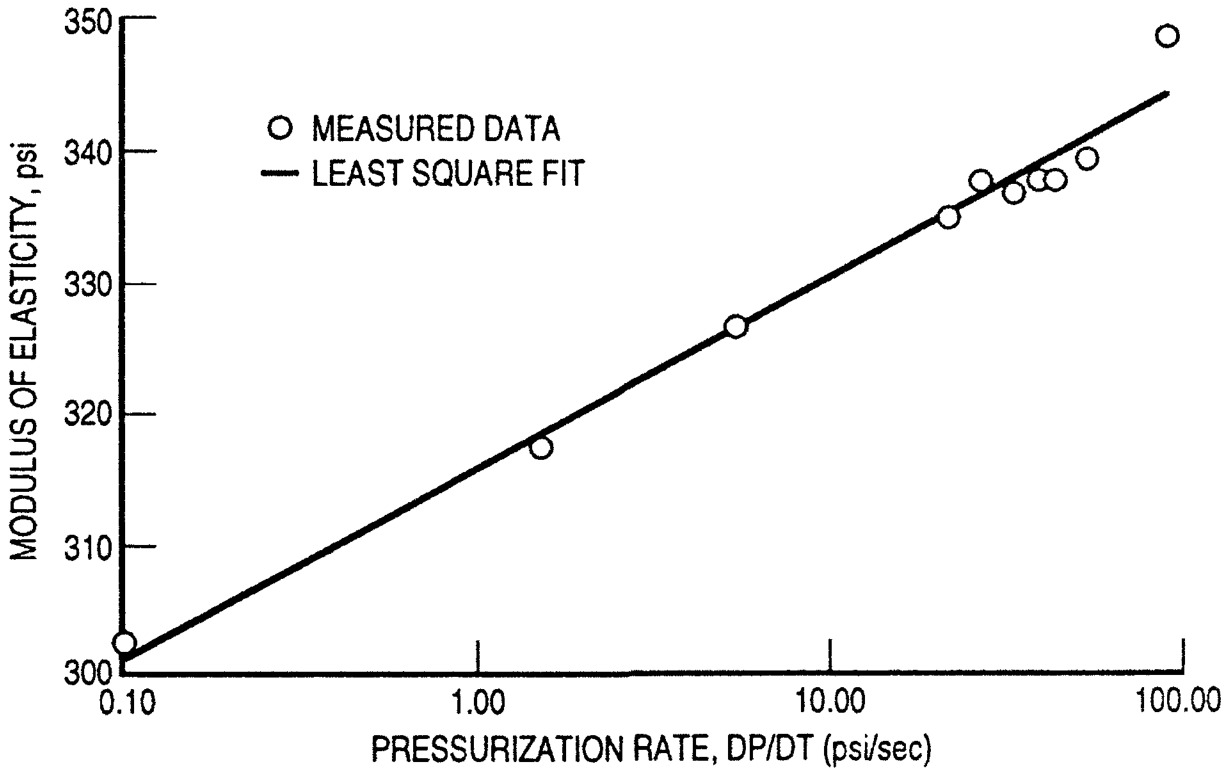

Figure 5. Dynamic modulus of EZ-Cast™ 521 vs pressurization rate.

Karlson, and Sorensen.[4] The SAAS III uses 4-node elements with a constant strain formulation, while the selected CAX8RH elements in ABAQUS use an 8-node, biquadratic displacement field with reduced integration. The latter is formulated particularly for materials with incompressible or nearly incompressible material behavior.

In the SAAS III analysis, two different meshes were used. One has 25 equally spaced nodes in the radial direction between the center and the OD of the disk and 20 equally spaced nodes in the across-thickness direction. This corresponds to 456 elements. The other mesh has 40 equally spaced nodes in the radial direction and 20 nodes in the across-thickness direction. This gives 741 elements.

For the ABAQUS model, 25 nodes in the radial direction and 21 nodes across the thickness were used. Because of the 8-node element configuration, only 120 elements were generated by the codes, as compared to 456 elements in the first SAAS III mesh configuration.

In both SAAS III and ABAQUS calculations, a 250 psi Young's modulus was input. This 250 psi value was obtained from the results summarized in Ref. 1. Regarding the input value of Poisson's ratio, ν, both 0.499 and 0.4999 were used. The boundary condition was such that the degrees of freedom at the OD surface of the disk were suppressed. It was expected that the value of the Poisson's ratio when approaching 0.500 from the lower end would have a significant impact on the results. However, it was later found from the results that the Poisson's ratio was a weak function of the displacement and stress predictions for this loading and geometric configuration. This is primarily due to the lack of constraint at both flat surfaces of the disk. except at the OD surface.

The predicted and measured normal displacement at the center of the disk under a 50-psi, uniform pressure at one circular surface of the disk and E = 250 psi is shown in Table 1.

It is noted that the predicted displacements from the two SAAS III meshes are very close, indicating the results are converging to the limit. The ABAQUS code predicts a slightly larger normal displacement (1.1%) than the SAAS III predictions. These results show that the ABAQUS code gives comparable predictions to those produced by SAAS.

Based on an experimental pressure/displacement slope of 57.2 (Fig. 3), Young's moduli of 209 and 211 psi, respectively, are calculated using the SAAS III and ABAQUS analytical displacement values shown in Table 1. These are approximately 16% lower than the value of 250 psi in Table 1.

Table 1. Comparison of Predicted Normal Displacement at Center of Disk Under 50 psi Applied Pressure and E = 250 psi with Experimental Results

Model Type	Normal Displacement (in.)
SAAS III 25 x 20 mesh	0.729
SAAS III 40 x 20 mesh	0.732
ABAQUS 25 x 21 mesh	0.738
Experimental Results	0.870

IV. DISCUSSION OF RESULTS

The new testing technique is an improvement over the uniaxial "dog bone" method for low-modulus materials. It is free from alignment prestress. The disk is under a biaxial stress field, which simulates the stress in the live propellant better than a uniaxial specimen can. The test may be simpler to build and easier to execute than other related efforts such as the Lightweight Analog Model (LAM) series conducted by Hercules.[5]

The testing technique was successfully demonstrated by the use of EZ-Cast™ 521, a thermosetting plastic material with predominantly elastic properties. Normal displacements at the center of the disk were measured by either statically or dynamically applied pressure on one circular face of the disk specimen. The linear deflection pressure curve suggests that the modulus of elasticity is constant over the strain range tested.

Based on the center displacement measurements, we calculated from the formula for a thick circular plate and from two finite-element codes (ABAQUS and SAAS III) moduli of elasticity of 300 psi and 210 psi, respectively . The results show +20% and -16% differences from the uniaxial test results. The differences are probably caused by two sources: the over-prediction by the closed-form solution because Eq. (1) is for small deformation and underprediction by the finite element codes. More importantly, the variation in moduli between the dog-bone and disk specimens is primarily due to the difference in curing process.

In order to investigate the possible modulus variation among different cure operations for the EZ-Cast™ 521 compound, five 1/2-in.-diam. rods were cast at 121°C (250°F) with cure time ranging from 1/2 to 3 hr. Both the 1.5(A) and 1.5(B) specimens had the same 1.5 hr cure time. Differential scanning calorimeter (DSC) and dynamic mechanical modulus tests using a rheovibron were conducted for pieces cut from the five rods, the dog-bone specimen, and the disk specimens. The former technique measures the heat capacity (specific heat multiplied by the mass) of the specimen as a function of temperature. The measured results provide the information to determine the glass transition temperature, T_g, of the material. The latter determines the dynamic modulus of the material as a function of both temperature and frequency. When the testing frequency decreases, the measured dynamic modulus approaches the static value.

Figure 6 depicts the DSC results for the five rods. The curves indicate that the glass transition temperatures for the five rods are very close (which is in the neighborhood of -40°C). Figure 7 depicts similar results for the dog-bone and the disk specimens. These DSC results indicate that the cures among all prepared specimens are comparable.

The dynamic modulus values from rheovibron tests for the five rods at 20°C (68°F) and 1.1 Hz are listed in Table 2.

Table 2. Dynamic Modulus of EZ-Cast™ Material[a]

Cure Time, hr	Dynamic Modulus, psi
3	328
1.5(A)	338
1.5(B)	410
0.5 + 1	309
0.5	242

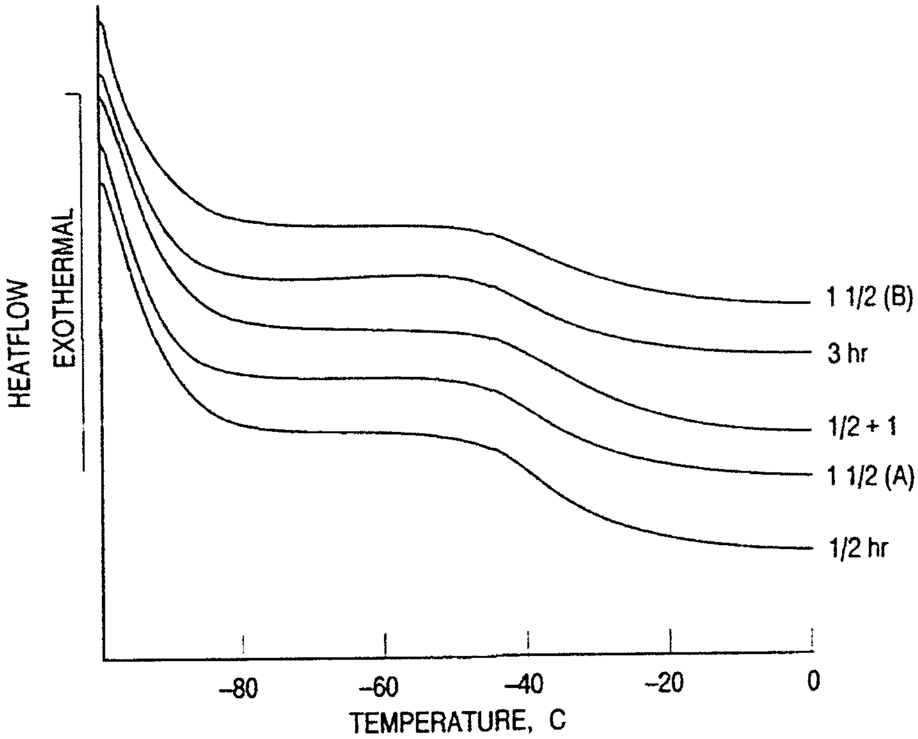

Figure 6. Differential scanning calorimeter results for five EZ-Cast™ rods with different cure time at 250°F. (Curves are displaced separately for clarity.)

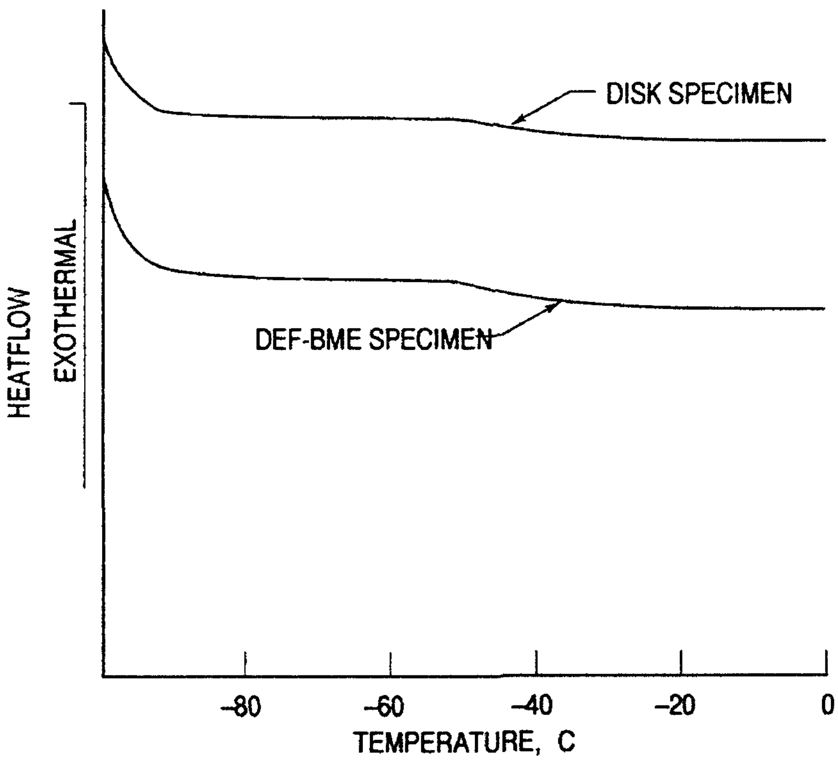

Figure 7. Differential scanning calorimeter results for dog-bone and disk.

When considering the limited number of samples and the experimental scatter, the results from the dynamic mechanical tests indicate that the samples have a comparable degree of cure. For more quantitative modulus values, however, more sample testing is required.

The data in Table 2 demonstrate that different modulus values for EZ-Cast™ are expected to exist for different specimens, such as what was observed in the dog-bone and disk specimens. A -16 to 20% variation in modulus is not uncommon for polymeric materials. Therefore, it is concluded that our proposed disk testing technique will provide accurate modulus values for any low-modulus material.

V. CONCLUSIONS

A new testing technique was demonstrated for measuring the modulus of elasticity of low-modulus materials such as rocket propellant grains. The technique offers substantial advantages over the dog bone testing method. The testing procedure is less complicated, and data reduction is simpler. More importantly, a biaxial stress field can be established in the test specimen to simulate conditions encountered during pressurization of a nonlinear and viscoelastic material (e.g., propellant used in solid rocket motors).

The technique was demonstrated by using a circular disk cast from EZ-Cast™ 521 thermosetting plastic material. Normal displacement at the center of the disk as a function of static pressure was measured. Modulus of elasticity versus deformation calculations were made using a closed-form solution and finite-element computer codes. Modulus values of 300 and 210 psi were obtained using the two approaches, respectively, based on the measured center displacement. These values show a -16 to 20% difference to the 250 psi value determined by the dog-bone method.

Differential scanning calorimeter and dynamic mechanical modulus tests were conducted for EZ-Cast™ 521 cast specimens of different cure conditions (1/2 to 3 hr). Results show that the glass transition temperatures for all specimens tested are very close; whereas, the dynamic modulus values vary as much as 70%. This indicates that the reported difference in modulus measurements is primarily caused by the property variation between the EZ-Cast™ 521 dog-bone and disk specimens.

The effects of the dynamic pressurization rate on the Young's modulus is about a 10% increase when the pressurization rate increases from 0.1 to 90 psi/s. Other advantages of this test method include the capability to determine the viscoelasticity of materials such as inert and live propellants under biaxial stress field. It can also be used to evaluate the bond strength between the material and its case. We might expect greater differences with dynamic pressurization with a truly viscoelastic material.

One must bear in mind that the propellant in a solid rocket motor is cast as a long annulus before firing. Thus, the optimum geometry for simulating the stress field in a live propellant is an annulus. However, the test procedure for exerting a desired biaxial stress field on an annulus is far more complicated to achieve and monitor. The complication is due to the difficulty in applying unequal pressure on the forward face of the annulus relative to its bore. Separating the face and the bore by means of a diaphragm introduces a host of other complications that are not trivial to overcome. On the other hand, by applying a biaxial stress field on a disk-shaped specimen, no diaphragm is necessary. Hence, all the problems associated with using a diaphragm are entirely avoided. The question remains, however, whether or not the strain rate in the disk-shaped specimen closely resembles that in an annulus-shaped specimen. Although the question cannot be answered in this work, the information on material behavior generated from a thick-disk specimen with its outer edge clamped is a major improvement over the dog bone-shaped specimen for highly nonlinear viscoelastic materials in providing data for anchoring and verification of propellant structural codes.

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