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Acousto-Ultrasonic Technique Applied to Filled-Polymer Damage Assessment

B. Tang*
Wyle Laboratories, Edwards Air Force Base, California 93523-5000
C. T. Liu†
Phillips Laboratory, Edwards Air Force Base, California 93524-7160
and
E. G. Henneke II‡
Virginia Polytechnic Institute and State University, Blacksburg, Virginia 24061

An acousto-ultrasonic technique was used to assess cumulative damage in filled polymers. Advantages of the technique include 1) high sensitivity, permitting the detection of early damage processes, 2) fast response, permitting rapid and automated inspection, and 3) need for access to only one surface of the test article. Two kinds of tests—fracture and loading–unloading tests—were performed on filled-polymer specimens. In these tests, the energy content and central frequency of acousto-ultrasonic signals were obtained. The acousto-ultrasonic technique can be used to determine the damage state in filled polymers.

Nomenclature

f = frequency, Hz
M_n = nth moment of the acousto-ultrasonic signal
$S(f)$ = power spectral density

Introduction

FOR filled polymers such as solid propellants, deformation and fracture processes depend upon the applied load or deformation history. The internal damage that accumulates as a result of loading can be described in terms of microstructural damage processes such as dewetting, vacuole formation and growth, binder rupture, etc. When a filled polymer is stretched, first the adhesive bond between the binder and the filler particles fails, and then vacuoles form at the binder-filler interface, surrounding and engulfing the filler particles. Further straining causes the vacuoles to grow and the binder to stretch. Vacuole growth and coalescence will eventually form one or more macroscopic cracks, leading to fracture failure.

In solid rocket motors, from manufacture through flight, the solid propellant grain is subjected to environmental and operational forces that tend to degrade and destroy the grain's structural integrity. Internal damage resulting from the action of such forces may seriously affect the propellant's ballistic and mechanical properties and can cause catastrophic failure of the solid rocket motor. Hence, in formulating a propellant grain, the internal ballistic characteristics and structural integrity need to be optimized. The propellant material is highly heterogeneous. The propellant's mechanical behavior depends upon local filler particle size variation, filler–binder bond strength, and binder crosslink density. Ultrasonic studies of the quantitative damage development prior to final rapid crack growth in this material have been reported in a number of papers by Knollman et al.[1–3] Measuring the change in wave speed and attenuation through the thickness of propellant specimens, they found that these parameters correlated well with vacuole development resulting from the dewetting phenomenon. A model[3] was developed to relate ultrasonic parameters to the damage state of a solid propellant. In this model, a damaged region in a propellant is regarded as a spherical cavity in a homogeneous, isotropic medium. When the propellant is strained and in a stage of vacuole formation and growth, the propellant is considered to have a system of randomly distributed, isolated spherical voids. As ultrasonic waves travel through the damaged propellant, wave attenuation caused by wave scattering from the isolated voids is related to the damage state of the propellant. A detailed description and the mathematical formulation for the model can be found in Ref. 3. Thus, quantifying internal damage using ultrasonic technique would provide insight into the deformation and fracture processes. When characterizing material properties in a quasistatic tension test, valuable information can be obtained by measuring the damage development in the material along the direction of applied load. Ultrasonic wave speed measurements made along this direction are compounded in error by reflections at the specimen boundaries and by the material's internal composition and damage. An alternative method is the quantification of the wave signal in terms of spectral moments obtained from analysis of the temporal- or spectral-domain information. This approach, known as the acousto-ultrasonic (AU) technique, has been discussed in some detail by Kiernan and Duke[4] and Duke et al.[5] The purpose of this study is to apply the AU technique to determine the damage state in solid propellants.

AU Technique

The basis of the AU technique is the quantification of a material's mechanical properties by means of small-magnitude mechanical excitation. The AU technique was originally proposed by Vary and Bowles[6] for evaluating the integrated effect of distributed material degradation. Its potential has been demonstrated for a number of different applications.[7] The technique involves exciting a low-intensity ultrasonic stress wave into the component of interest by means of a piezoelectric ultrasonic transducer and then detecting the resulting mechanical disturbance using an acoustic emission sensor, located a short distance away from the transmitting transducer on the same surface. The material in between the two transducers acts as a waveguide for the ultrasonic stress wave. Any changes in the mechanical condition of the material along the path that the stress wave propagates will be integrated in their effect on the detected stress wave. The acoustic emission sensor monitors the mechanical disturbance as modified by the material, and signal analysis yields[4,5] moment-related parameters such as the zeroth moment M_0, which is directly proportional to the energy of the received signal, and the central

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*Research Scientist.

†Senior Material Research Engineer, RKAP, 4 Draco Drive. Member AIAA.

‡Head, Department of Engineering Science and Mechanics.

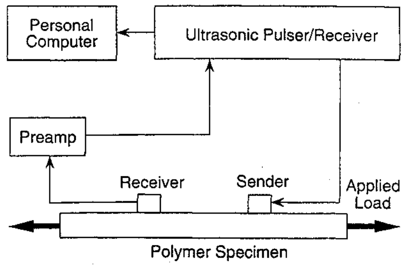

Fig. 1 Schematic diagram of the experimental setup.

frequency, which is the quotient of the first moment M_1 and the zeroth moment M_0. Various moments of an AU signal are given by

$$M_n = \int_0^\infty S(f) f^n \, \mathrm{d}f \qquad (1)$$

Advantages of the AU technique include 1) high sensitivity, permitting the detection of early damage processes, 2) fast response, permitting rapid and automated inspection, and 3) need for access to only one surface of the test article. The AU technique has successfully located weak regions in composite laminates that cannot be detected with sophisticated ultrasonic c-scan equipment.[8] The AU technique is a valuable tool in investigating damage and in-service performance of materials and engineering components.

Specimen Preparation and Testing Procedure

Inert propellant specimens of dimensions 4.0 × 1.5 × 0.25 in. (10.16 × 3.81 × 0.635 cm) were attached to aluminum end tabs with a two-part epoxy, resulting in a gauge length of 2.25 in. (5.72 cm). The specimens were tested in an Instron testing machine. A 0.5-in., 2.25-MHz broadband ultrasonic transducer and a low-frequency acoustic emission transducer were used as the transmitting and receiving transducers, respectively. These transducers were mounted centrally, 0.5 in. (1.27 cm) apart, along the gauge length of the specimen. When the transducers were mounted, a viscous gel was applied to the transducer surfaces, thereby coupling ultrasonic waves between the transducers and the specimen. The transducers were allowed to move with the specimen when it was stretched under loading.

Two mechanical tests, fracture and loading-unloading tests, were performed. A Panametrics 5052A pulser–receiver, a Panametrics preamplifier, and a PC-based digital acquisition board (Sonix two-channel, 25-MHz real-time sampling STR*825 board) were used to generate and record the AU signals. A block diagram of the ultrasonic setup in presented in Fig. 1. A modified experimental setup using a tone burst signal required the replacement of the pulser–receiver with a function generator. For the fracture test, the specimen was loaded in tension at a strain rate of 2.2%/min until fracture while AU signals were simultaneously obtained. In this test, 8-MHz pulses were sent to the transmitting transducer from the Panametrics pulser. In the modified method, the data were obtained by sending in 22-kHz, 60-V (peak to peak) tone bursts. The transducers and excitation frequencies were selected to optimize the signal-to-noise ratios of the receiving signals. The loading–unloading test used 8-MHz pulses like the fracture test, but the specimen was unloaded near one-half its failure strain (as determined by the fracture tests); hence, the specimen never reached fracture. Signal analysis for energy content (M_0) and central frequency (M_1/M_0) was performed using developed software.[9]

Results and Discussion

Results of the fracture test are shown in Figs. 2–4. The stress–strain curve for the specimen exhibits the basic shape observed with other propellant materials.[10] Initially, the slope of the curve is high until a plateauing, where the stress remains nearly constant with increasing strain. Near the end of the specimen's life, the stress decreases rapidly until the point of fracture. At the plateauing region, the dominant damage mechanism in the material is believed to be the growth of vacuoles, which contributes significantly to volume dilatation. Figures 2 and 3 show that the energy content of the

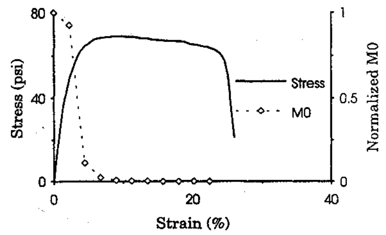

Fig. 2 Stress, strain, and energy measurements for the fracture test.

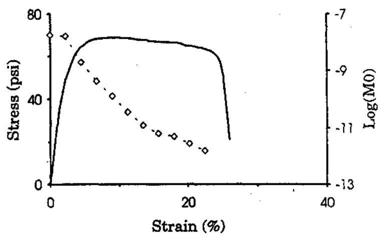

Fig. 3 Plot of log M_0 for the fracture test.

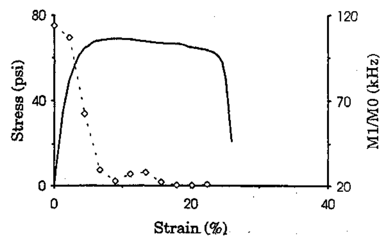

Fig. 4 Central-frequency plot for the fracture test.

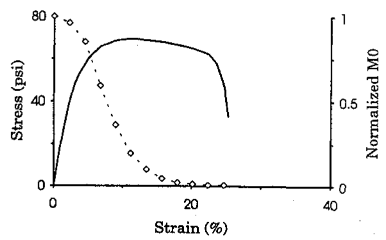

Fig. 5 Stress, strain, and energy measurements for the tone burst test.

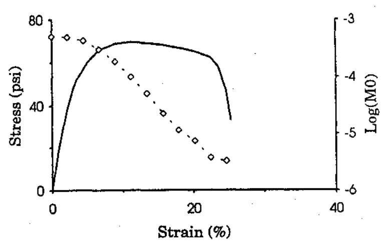

Fig. 6 Plot of log M_0 for the tone burst test.

AU signal decreases during the specimen's life. The initial rapid decrease in energy content may be due to the absorption and scattering effects of the dewetting and vacuole formation processes. Smaller energy-content differences between consecutive measurements are observed during the latter portion of the specimen's lifetime, when vacuole growth and coalition and macroscopic crack formation are believed to dominate. Figure 4 illustrates the relation between central frequency and percentage of strain. It is believed that the generation of vacuoles in the material, which filters out the high-frequency portion of the input signal, contributes to the change in central frequency. In general, the energy content of the AU signals seems to be a more consistent parameter for material characterization. For the tone burst test, the behavior of energy content in relation to increasing strain (Figs. 5 and 6) is similar to that observed in the fracture test. The energy content in the tone burst test decreases at a slower

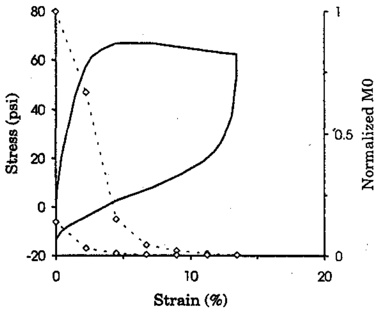

Fig. 7 Stress, strain, and energy measurements for the loading–unloading test.

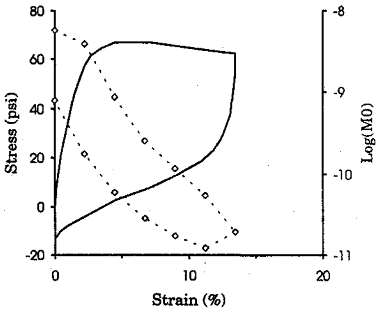

Fig. 8 Plot of log M_0 for the loading–unloading test.

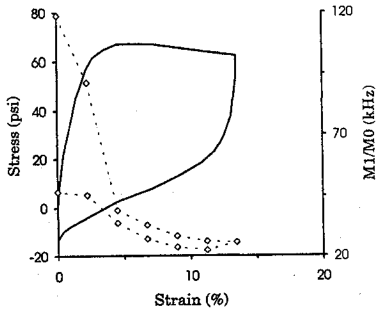

Fig. 9 Central-frequency plot for the loading–unloading test.

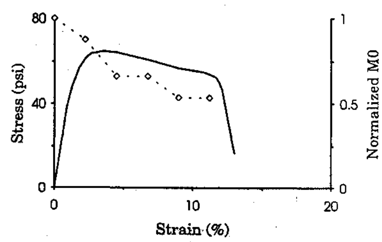

Fig. 10 Stress, strain, and energy measurements for the through-transmission test.

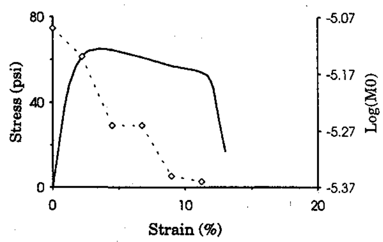

Fig. 11 Plot of log M_0 for the through-transmission test.

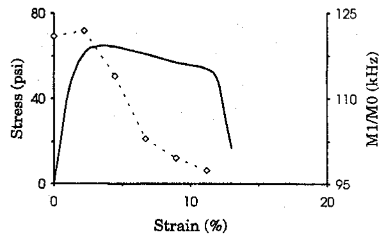

Fig. 12 Central-frequency plot for the through-transmission test.

pace than in the fracture test. As mentioned before, it is believed that the generation of vacuoles in the material filters out the high-frequency signals, which make up the major part of the input pulse in the fracture test. In the tone burst test, since the input signal is a low-frequency signal, we expect the energy content to decrease slowly.

Figures 7–9 show the results of the loading–unloading test. During the loading cycle, in response to the imposed strain, microscopic damage such as nucleation and growth of vacuoles occurred in the specimen. When the applied strain was reduced during the unloading cycle, the local stresses between particles might still be high enough to cause additional damage and/or to increase the size of existing vacuoles. In addition, the local time-dependent material response was out of phase and lagged behind the applied strain because of the viscoelastic nature of the material. Thus in the unloading cycle, the size of the vacuoles remained large in comparison with their size at the same strain level during loading, and this phenomenon is manifested in the energy-content and central-frequency measurements. According to these figures, once again, the energy content of the AU signals seems to be a more appropriate parameter for propellant characterization.

So far, the data have been obtained from transducers allowed to move with the specimens as they were stretched under tension. Changes in AU parameters are mainly due to damage development within the specimens. Other factors that may contribute to the changes are the large elongation of the material in the loading direction and the contraction of the material in the thickness direction. Figures 10–12 show the results of the ultrasonic through-transmission test. In this test, two transducers were mounted face to face on opposite sides of the specimen. Unlike Figs. 2–4, energy content and central frequency decrease slowly with applied strain in the through-transmission test. The material interrogated in this test is the 0.25-in.-thick area between the two transducers, which is less than the areas inspected in other tests.

The stress–strain curve of the last test is different from the previous stress–strain curves on account of the aging of the material. All the specimens were from the same block of inert propellant. The specimens were wrapped in aluminum foil and stored in a desiccator before test. The aging of the material creates a problem in comparing experimental results from different tests. Further work is needed to develop a theoretical model based on the model in Ref. 3 to relate AU parameters to the damage state of solid propellants.

Conclusions

In general, the energy-content parameter of the AU data effectively characterizes damage in inert solid propellant material, whereas the central-frequency parameter requires further investigation. The overall results from the tests are fairly consistent. Energy-content measurements derived using the AU technique should aid researchers to understand cumulative internal damage in filled polymers.

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E. A. Thornton
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