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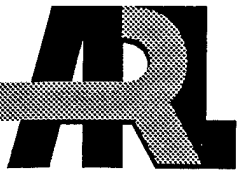

Mechanical Response and Morphological Characterization of Gun Propellant

Robert J. Lieb

ARL-TR-1205 September 1996

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13. ABSTRACT *(Maximum 200 words)*

The mechanical and morphological characterization of energetic materials (gun propellants in particular) has been progressing steadily for many decades. Progress has accelerated in the last 5 years, and several principal goals have been attained. This report outlines the objectives and methods of mechanical and morphological characterization of gun propellants, provides general characterization results for the four basic propellant types, and lists references to more detailed information. Fracture failure characterization is also addressed, and the methods for determining fracture-generated surface area are presented, along with results for the two most friable propellant types. The importance of morphological information is discussed, and an example of how the information can be used is provided.

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1. INTRODUCTION

It has long been realized that propellant breakup due to fracture causes performance degradation and increases the violence of propellant response to vulnerability threats. The perceived role that the mechanical response has played has varied throughout the past decade and a half. Early efforts focused on issues such as: determining whether gun propellants could even be characterized; developing procedures to prepare specimens and extract meaningful information; identifying properties that are desirable or undesirable; and developing predictive procedures. These issues have been mostly resolved, and procedures have been developed so that questions concerning the nature and scope of mechanical response in the gun propellant arena can be meaningfully probed. Recent work has addressed attempts to establish the relationship between gun performance and vulnerability, and mechanical response measurements. These efforts have been integrated with propellant morphological studies using scanning electron microscopy (SEM), and together, much progress has been made during the last 5 years. [1-11]

Early results revealed that high-rate, compressive measurements could be made reliably and that conventional engineering properties were able to characterize the propellant response over the temperature range of ballistic interest (-40 to 60° C). These studies also supported the assumption that the mechanical response of the propellant did not affect combustion until after mechanical failure of the material. This led to the design and purchase of new equipment that would permit the development of methodologies and parameters that characterize propellant failure in a fashion thought to relate to the combustion and vulnerability response of the material.[4] It was known, for example, that during an abnormal ignition event, a brittle propellant, subject to a pressure wave within the propellant bed, can suffer fracture in such a way that will support the growth of that pressure wave and lead to catastrophic gun failure.[12] The search for a simple mechanical parameter that could measure the propensity for a propellant to generate surface area upon mechanical failure resulted in the development of a failure parameter called the failure modulus,[3] E_f, that measures the rate at which the material strength is lost as a function of strain after material failure has begun.

This failure parameter has been put to pragmatic use in the development of new propellants (e.g., XM39, M43, and currently PAP1, a CL-20 based propellant) to ensure that improvements in formulation and processing did not degrade the mechanical response characteristics of the material. It has also been successfully used to evaluate the relative fracture susceptibility among various propellant lots, or between unconditioned propellant and propellant that has been subjected to special conditioning that may affect its mechanical response (e.g., thermal cycling). Methodology for the use of SEM was also developed to aid in the interpretation of mechanical properties measurements and to determine the morphological state of propellants undergoing development and processing changes.[18]

The failure modulus, however, showed indications of direct application to brittle failure when changes in the vulnerability response to shaped charge jet attacks were related directly to changes in this parameter.[2] The correlation was found for propellant beds at low temperature subject to a shaped charge jet attack and led to other studies that attempted to make the correlation more direct. The relationship, however, was made between *changes* in both responses, rather than directly relating the mechanical and vulnerability responses themselves. In addition, the mechanical response measurements were performed at rates of about 100 s^{-1}, whereas the rate of mechanical deformation during the jet interaction is estimated to be between 10^5 and 10^6 s^{-1}.

The previous observation led to studies in which compressive stress relaxation measurements[7] were performed and time-temperature shift factors were deduced to obtain master curves for all the basic propellant types. This enabled the determination of the temperature shift required to simulate the mechanical response characteristics of the propellant undergoing deformation at the corresponding higher strain rate. From this, the mechanical responses of propellant could be estimated for strain rates outside the limits of measurement by adjusting the propellant temperature. The augmentation of the vulnerability response due to mechanical considerations can be evaluated with this information. In addition, two questions were answered that helped establish much greater confidence in the use of shift factors. First, it was validated that the mechanical behavior was accurately simulated when these shifts were applied, and second, it was further shown that the time-temperature correspondence extended beyond the level of strain at which the relaxation measurements were made (i.e., into the region of failure).[8] Not only did the shift correctly predict the modulus, as expected, but the stress at failure, maximum stress, and method of failure were predicted, as well. These results greatly extended confidence in the application of the shift factor to simulate higher rate behavior.

Most recently, a direct link has been established between this failure parameter and a measure of fracture-generated surface area produced when M30, a conventional composite propellant,[10] and M43, a highly filled nitramine composite,[11] were uniaxially compressed. The failure modulus was measured, and damaged grains were later burned in closed-bomb firings to determine the surface area generated by the grain damage. These results showed that the surface area for the initial 10% of the fraction burned was directly related to the strain level and the logarithm of the failure modulus. This led to the prediction of the effective surface area profile, as a function of the fraction of the propellant charge burned, for any combination of failure modulus and strain level within the fracture domain. These results provided a method for assessing fracture damage for the M30 and M43 propellants by means of a simple mechanical measurement.

This paper presents the most current results of mechanical response measurement and the associated morphological technology. The results from the application of this technology help to provide critical information when assessing malfunctions and undesired performance characteristics, and provide critical guidance for propellants undergoing development to ensure that changes made during development do not increase performance at the expense of safety.

Many new areas of investigation have become viable because of the progress made in mechanical response measurement. The possibility of realistically modeling breechblow scenarios is brought closer because of the ability to express the fracture behavior of propellant grains as a function of a failure parameter and the strain state of the grain. Other questions remain, however. For instance, the full effect of fracture damage on performance and vulnerability response may include dynamic processes that become inactive if ignition and combustion are delayed, even for a very short period. There is evidence of partial decomposition on fracture surfaces, and it is known that fracture in polymeric materials releases radicals, ions, and other charged particles in various excited states. Plans are under way to continue to address these developing concerns through new instruments and procedures.

2. UNDERSTANDING THE ROLE OF PROPELLANT MECHANICS

2.1 Mechanical Response Measurement. The propellant response measurement technique at the U.S. Army Research Laboratory (ARL) has evolved from a method using a drop weight tester to the current device, a specially designed servohydraulic tester,[4] illustrated in Figure 1. When it was realized that prefailure characterization could not predict failure response, a new methodology using new equipment became necessary because the specimen's prefailure response affected the conditions evolving after failure in the drop weight machine. Since the required new parameter would characterize the failure response, this dependence had to be eliminated. The servohydraulic machine allows for controlled compression measurements at any rate up to 1,000 s^{-1} for a specimen about 1 cm in length. Compression ceases at contact between the impact bell and cone. Therefore, the amount of specimen compression can be accurately predetermined by setting the anvil height. Contact between bell and cone also shunts the force around the specimen. The nitrogen spring absorbs the residual mechanical energy and moderates the deceleration rate of the massive ram. The force applied to the specimen is measured using the gauge inside the impact bell. During compressive response measurements, displacement is measured with a linear variable differential transformer in the actuator column and is corrected for machine stiffness.

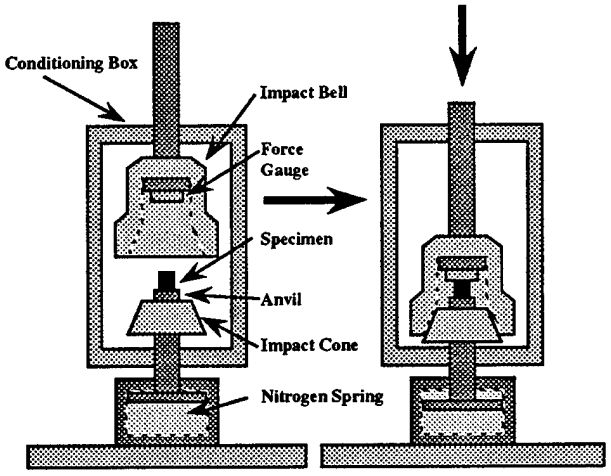


Figure 1. Servohydraulic Tester

The specimens are prepared from a variety of sources. The most common form is the multiperforated gun propellant grain. Since the fracture response depends on many factors, one of which is the propellant processing, using specially made grains or solid core samples will almost always result in specimens with fracture characteristics different from the propellant grain being fired within the gun. One cardinal rule that is employed in this facility is to never perform measurements that may have as much of a problem being related to the applied situation as the original problem being solved. Since the fracture response of multiperforated grains needs to be understood, specimens are made from samples of those same grains. The specimen preparation procedure begins by cutting the sample with a diamond saw to a length of about 1.00 cm. The ends are cut flat, parallel and perpendicular to the grain axis according to the specifications in an updated version of the test procedure entitled "Uniaxial Compressive Gun Propellant Test."[13] This specimen preparation is required to acquire overlapping stress-strain curves in repetitive testing, to acquire uniform failure stresses, and to minimize (or eliminate) the "toe" observed at the beginning of many response curves (see Figure 2). Temperature conditioning is achieved by placing specimens inside the environmental chamber for a time at least twice that needed to reach thermal equilibrium (a total of about 30 min). The specimen is then positioned and compressed. The testing takes place within the conditioning chamber, so no transfer from a conditioning environment is required, and therefore, no thermal disruption occurs.

As explained previously, the final strain to which the specimen is taken is determined by the distance between the anvil and the force gauge when the bell and cone surfaces mate. That distance is determined by placing a lead specimen on the anvil and performing a compression. This procedure allows for any dynamic affects to be taken into account that may be overlooked during a static measurement. The percentage strain used in these tests can be reliably selected from 2% to over 80% and is normally selected to be 50%. Failure of the gun propellant grains usually occurs between 2 to 8% strain, depending upon composition, temperature, and strain rate.

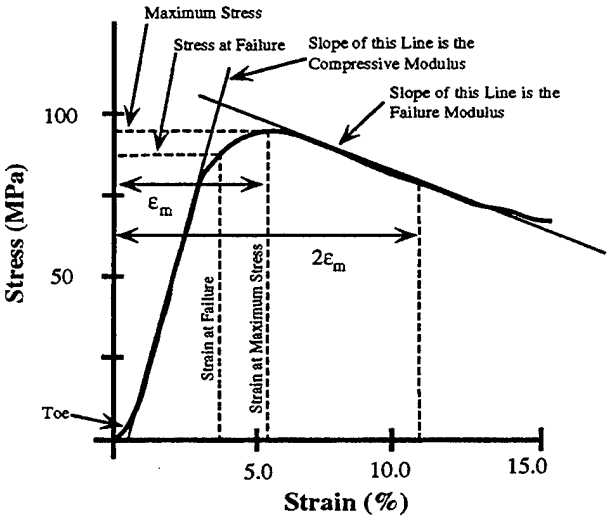


Figure 2. Mechanical Characterization Parameters

The specimen strain rate can be selected from quasi-static to as high as 1,000 s^{-1} and is chosen for routine testing to be 100 s^{-1}, which is the order of strain rate encountered by the grains during a normal ballistic firing.[23] Higher rates can be simulated using lower temperatures, as will be explained more fully later, with each decade of rate change being represented by a temperature shift of about 10° C.

The parameters measured during a response characterization test are the modulus (E), maximum stress (σ_m), strain at maximum stress (ε_m), stress at failure (σ_f), strain at failure (ε_f), and failure modulus (E_f). The definitions of these parameters are illustrated in Figure 2. While E, σ_m, and ε_m have the usual engineering definitions, the point of failure and the failure modulus have special definitions. The failure modulus is the slope of the stress-strain curve in the *near-linear* region between strain at maximum stress and twice that value. If no maximum stress occurs in the region of failure (because of work hardening and plastic failure), the failure modulus is measured between the strain at failure and three times that value. The failure point is determined by the intersection of the two lines that determine the modulus and the failure modulus. The strain at that intersection point is called the strain at failure, and the stress at failure is the corresponding stress on the response curve. All these parameters are needed to characterize material. The modulus describes how quickly the stress rises with strain, the stress and strain at failure show where the material begins to yield, the maximum stress and strain at maximum stress provide ultimate strength information, and the failure modulus describes the nature of the failure process. For a given propellant, this failure process most strongly affects the performance and vulnerability response. The failure modulus, therefore, is the parameter of greatest interest. The reasons for this are outlined below.

The failure modulus is a parameter that describes how quickly the material is losing strength after achieving a maximum stress. Its value is determined by dividing the change in stress by the corresponding change in strain, in the linear region immediately following maximum stress. If brittle fracture occurs and the material is no longer able to support the applied load, a dramatic loss in strength is observed, which results in very large negative values of E_f. If the sample fails in a plastic mode, the material may be able to support almost the maximum stress level well beyond the failure point. This results in a flattening of the response curve near maximum stress and failure modulus values very near zero (sometimes even positive). A more important observation is that values that

Table 1. Average Mechanical Property Values of Tested Propellants

Temperature	Propellant	Maximum Stress	Strain at Max Stress	Failure Stress	Failure Strain	Modulus	Failure Modulus
°C		MPa	%	MPa	%	GPa	GPa
-40	M14	207.8	8.5	202.0	5.8	4.30	-0.743
	JA2	-	-	123.5	5.5	2.72	-0.411
	M30	243.0	7.0	241.0	7.0	4.69	-12.900
	M43	145.8	3.2	122.0	2.75	5.97	-18.40
-20	M14	181.8	7.0	173.0	5.1	4.08	-0.894
	JA2	-	-	74.8	5.0	1.89	-0.086
	M30	171.0	7.5	169.0	7.0	3.20	-1.740
	M43	141.0	3.5	140.0	3.4	5.28	-12.50
0	M14	177.9	6.0	160.0	3.8	5.36	-0.296
	JA2	-	-	40.6	3.2	1.35	0.023
	M30	134.8	6.2	125.0	4.2	3.06	-0.530
	M43	130.9	4.0	124.0	3.3	5.53	-1.58
20	M14	121.7	7.0	115.0	4.8	2.79	-0.205
	JA2	-	-	21.0	3.0	0.82	0.025
	M30	73.9	5.0	68.9	3.2	2.26	-0.260
	M43	101.0	4.5	92.0	3.0	4.30	-0.430
50	M14	106.6	6.5	100.0	4.2	2.58	-0.140
	JA2	-	-	10.5	3.2	0.38	0.032
	M30	64.5	6.5	60.0	4.0	1.60	-0.190
	M43	60.0	4.0	58.0	2.7	2.74	-0.230

are lower in magnitude indicate a lower amount of fracture has occurred. The specimen, then, is able to continue to support a reduced load, and a lower amount of fracture surface area is exposed to the flame. Typically, brittle and plastic failures occur together, which results in corresponding, intermediate values of E_f.

Measurement of the characterization parameters determined from the response curves are given in Table 1 for the four basic propellant types (single, double, triple, and nitramine base). The measurements were made at -40, -20, 0, 20, and 50° C, and the reported values were determined from the average curve that was generated from the five response measurements, except in the case of very brittle responses (usually near -40° C) where curve averaging can be deceptive. Under brittle conditions, failure can occur over a very wide range of stresses and strains, which will cause artifacts in the *average curve*. In these extremely brittle cases, therefore, the parameters listed were first determined from individual response curves and then were averaged. Failure to use a proper averaging sequence can lead to misleading average response curves and erroneous values for the parameters determined from them.

Figure 3 shows examples of stress-strain curves for M30 propellant. Figure 4 shows the resulting specimens, which were compressed to 50% strain. Note that the appearance of the grain correlates well with the magnitude of the failure modulus. The relationship that seems to hold is: the lower the value of E_f, the greater the amount of surface area that is generated during compressive failure. This observation, along with the vulnerability response observations mentioned earlier, led

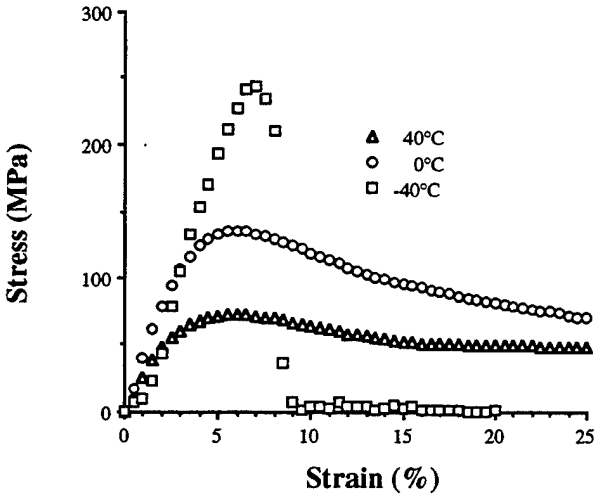


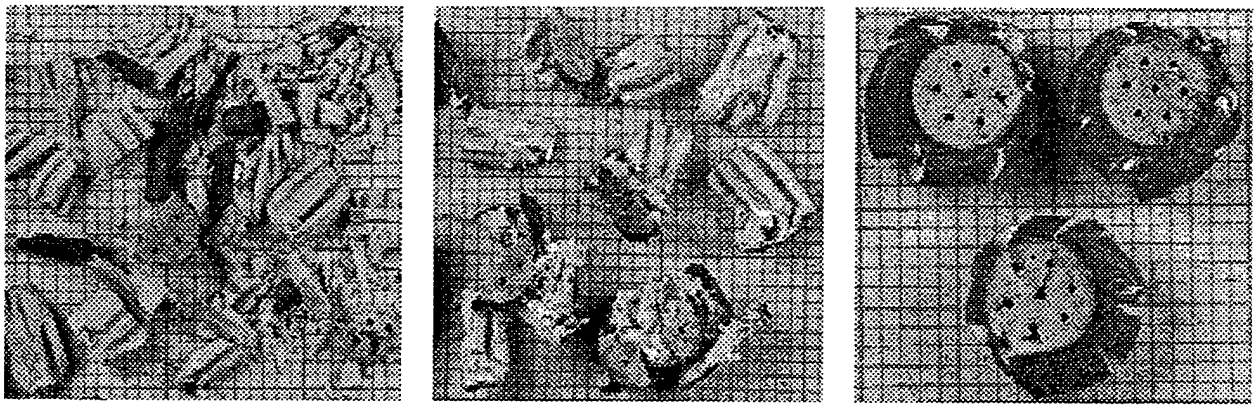
Figure 3. Stress vs. Strain Curves for M30 Propellant

to the establishment of a functional relationship between E_f and the generated fracture surface area.

2.2 Relation Between E_f and Fracture Surface Area. Propellant grains, damaged by uniaxial compression while mechanical response characterization measurements were performed, were burned in a special, small-volume closed bomb[14] to determine the effect of mechanical damage on the amount of surface area generated. The pressurization within the bomb during combustion is controlled by the intrinsic burning rate of the propellant and its exposed surface. After burning, the acquired pressure-time curves were used to determine the evolving surface area ratio (the surface area divided by the initial area of an undamaged specimen, S/S_0) of the charge as a function of the fraction of the charge burned, using the propellant burning rate established from undamaged specimens.[15]

These results permitted the characterization of the surface area profile for the burning charge as a function of amount of applied strain and the propensity of the propellant to fracture, as measured by the failure modulus. Selected surface area profiles are shown in Figure 5 for M43 grains damaged to 50% end strain at various temperatures.

The surface area profile was then represented by a linear function that is weighted to take into account important features of the actual profile, the initial surface area and how it evolves as the charge burns. This effective surface area ratio for the first 10% of the fraction burned can be represented as a function of the amount of strain, ε, the failure modulus, E_f, and the fraction of charge burned, Z, by an equation of the form:



a. -40°C b. 0° C c. 40° C

Figure 4. Photographs of the M30 Propellant Specimens After Compression to 50% Strain

$$S/S_0\ (\varepsilon, E_f, Z) = [S_i/S_0\ (\varepsilon, E_f)] + [\text{Slope}\ (\varepsilon, E_f)]\ Z. \quad (1)$$

For M30,[10]

$$S_i/S_0\ (\varepsilon, E_f) = [-1.84 + 0.361\ \varepsilon + (0.392 + 0.116\ \varepsilon)\ \ln(-E_f)], \text{ and} \quad (2)$$

$$\text{Slope}\ (\varepsilon, E_f) = [20.6 - 3.40\ \varepsilon - (5.71 + 1.18\ \varepsilon)\ \ln(-E_f)], \quad (3)$$

where S_i/S_0 describes the initial surface area ratio and Slope (ε, E_f) Z describes the change in surface area ratio as the charge burns. These equations can be used as descriptors of the surface area profiles up to the first 10% of the charge burned for propellants that show fracture failure ($S_i/S_0 > 1.2$).

M43 propellant showed much more fracture and a more complicated dependence on the final strain and failure modulus.[11] The initial surface area ratio is described by either equation 4 or 5, depending on the amount of strain.

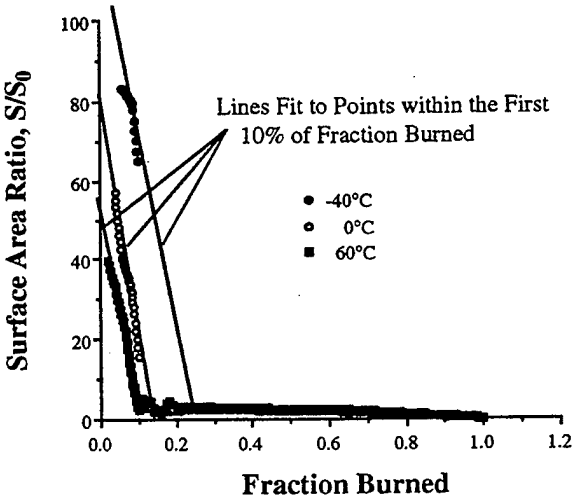


Figure 5. Surface Area Profiles for M43 Propellant Damaged at 50% Strain

For $10\% \le \varepsilon \le 20\%$, the initial surface area is given by

$$S_i/S_0\ (\varepsilon, E_f) = [3.9769 + 1.3336\ \varepsilon] + [-5.36 + 0.788\ \varepsilon]\ \ln(-E_f), \quad (4)$$

and for $\varepsilon > 20\%$, by

$$S_i/S_0\ (\varepsilon, E_f) = [3.9769 + 1.3336\ \varepsilon] + [10.27 + 0.007\varepsilon]\ \ln(-E_f). \quad (5)$$

For M43, somewhere above 20% strain, it seems as if the evolution of surface area loses most of its dependence on the initial structure of the propellant grain. This results in unpredictable evolution of S/S_0 above 20% strain, even though the initial surface area ratio has a predictable value. However, for 20% end strain and lower, the evolution of the effective surface area curve can be approximated, as previously stated, by placing the appropriate end strain and failure modulus values in the following equation.

For $10\% \le \varepsilon \le 20\%$,

$$\text{Slope}\ (\varepsilon, E_f) = [-159 - 6.05\ \varepsilon] + [41.2 - 7.07\ \varepsilon]\ \ln(-E_f)\ ; \quad (6)$$

for $\varepsilon > 20\%$ end strain, the evolution of the surface area profile seems to be erratic.

These equations can be combined to produce the effective surface area profile vs. fraction burned for the first 10% of the fraction burned for the following conditions.

For an end strain between 10 and 20%,

$$S/S_0\ (\varepsilon, E_f, Z) = [3.9769 + 1.3336\ \varepsilon + (-5.36 + 0.788\ \varepsilon)\ \ln(-E_f)] + [-159 - 6.05\ \varepsilon + (41.2 - 7.07\ \varepsilon)\ \ln(-E_f)]\ Z. \quad (7)$$

For end strains greater than 20%, only the initial surface area ratio is defined and is given by

$$S_i/S_0\ (\varepsilon, E_f) = [3.9769 + 1.3336\ \varepsilon] + [10.27 + 0.007\varepsilon]\ \ln\ (-E_f). \qquad (8)$$

The initial surface area ratios predicted from the previous equations are plotted in Figure 6 for both M30 and M43. For ease of viewing, isolines of S_i/S_0 are displayed by the dashed lines on the surfaces. As can be seen, the friability of M43 is much greater than for M30, even for the same value of failure modulus. This indicates that when equal amounts of load bearing capability are lost in these propellants, M43 produces much more fracture surface area. This characteristic contributes to the increased uncertainty in the surface area evolution for M43 propellant.

As mentioned earlier, the evolution of surface area needed to be extended to include a broader domain of conditions. Now that the generation of fracture surface area was expressed as a function

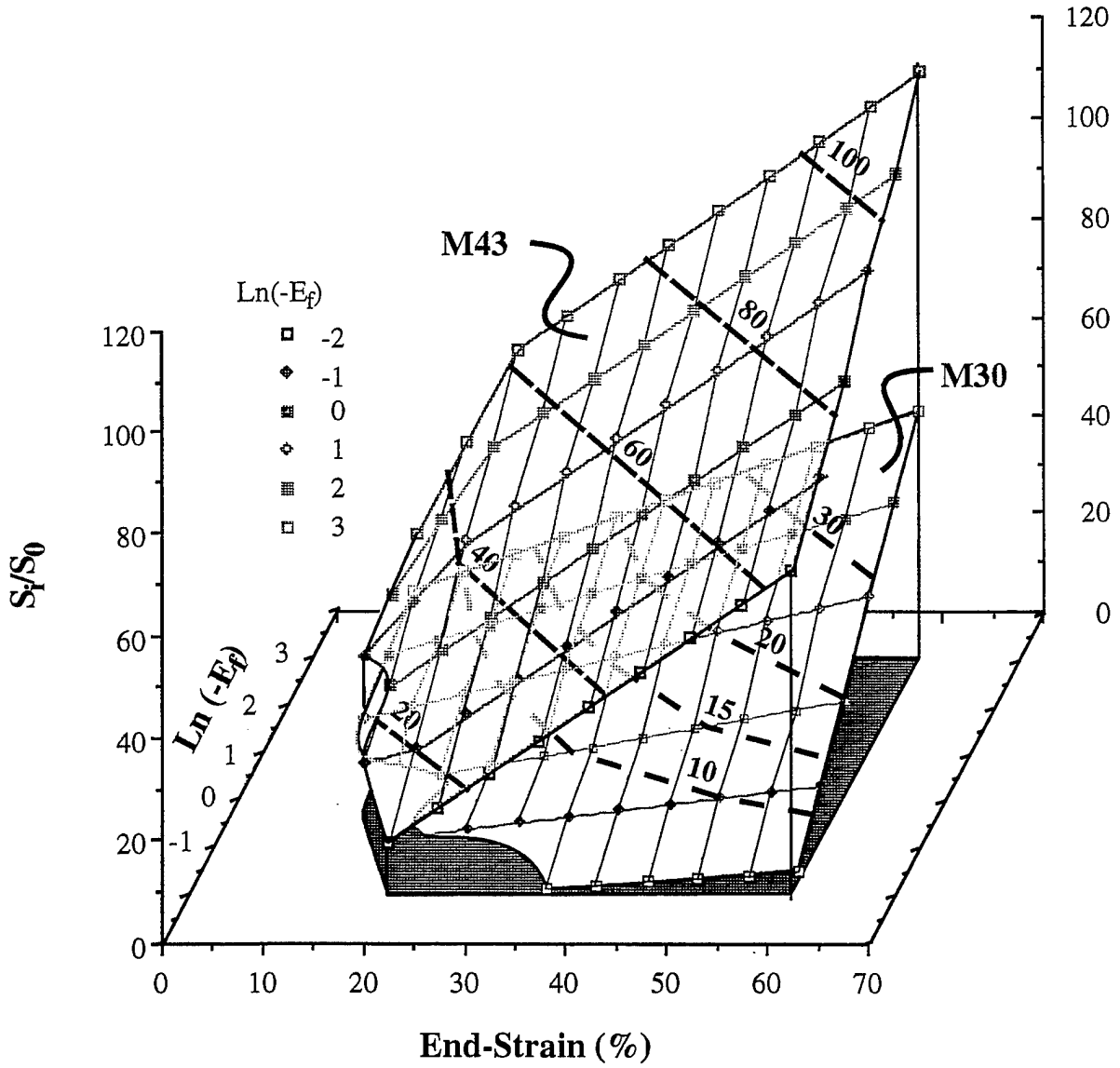


Figure 6. Initial Surface Area Ratio as a Function of Strain and Failure Modulus for M43 and M30

of strain and failure modulus, it became more important to find methods that would project the application of this dependence into regions not accessible to experiment. It was noticed that the rocket community has been successful in predicting long-term mechanical effects using high-temperature material properties extracted from stress relaxation measurements. We in the gun propellant community investigated the possibility of using the same method, but in the opposite direction (i.e., predict short-term behavior using the low-temperature relaxation response).

2.3 Time-Temperature Equivalency of Gun Propellants. A method that can be used to estimate mechanical response of materials at strain rates outside the limits of available equipment is to employ the time-temperature superposition principle[16, 17] by using stress relaxation measurements[7] to determine shift factors [A(T)] for the material. Once attained, material behavior can be approximated by shifting the temperature to simulate the response at the higher or lower rate. Relaxation measurements were performed on the four basic propellant types, single- (M14), double- (JA2), and triple-based (M30) propellants, and a nitramine composite (M43) gun propellant. Since the temperature range of interest for guns is -40 to 60° C, and testing can be easily performed in this range, relaxation measurements were made between these temperatures. The shift factors were determined from the relaxation stress plotted against the logarithm of time by creating a master curve for each propellant type. This procedure involved shifting the relaxation curves from the various temperatures, other than the reference temperature, along the Log (t) axis to form a single continuous curve. The amount of shift required was determined by acquiring the best linear fit for the overlapping points in adjacent temperature regions. This shift determined a factor (logarithmic differences are ratios of the argument) that corresponded to the temperature at which the relaxation was measured. The temperature difference was thus related to the ratio of the deformation rate. For example, an increase in strain rate by a factor of 100 (the corresponding shift in Log [(A(T)] is 2) can be approximated by a temperature shift of -24 C° (from the reference temperature of 21° C) for M43 propellant, as is shown Figure 7.

The temperature and rate equivalence for mechanical response was demonstrated for each propellant type[8] and is shown in Figure 8 for M43 propellant. The scatter in the values of the parameters for the different curves are within the scatter found for specimens tested under identical temperature and strain rate conditions. Therefore, the plots of stress vs. strain characterized the response as virtually identical. Mechanical response measurements were performed on each of the gun propellant types at four different strain rates (from 100 s^{-1} to 0.1 s^{-1}) and at the corresponding temperatures that were predicted from the shift calculation to provide an equivalent mechanical response. In each case, the mechanical response of the propellant type remained nearly identical. This was true for the response measured in the region of strain where the relaxation measurements were performed, and more importantly, this equivalent response was found to extend into the regions of strain corresponding

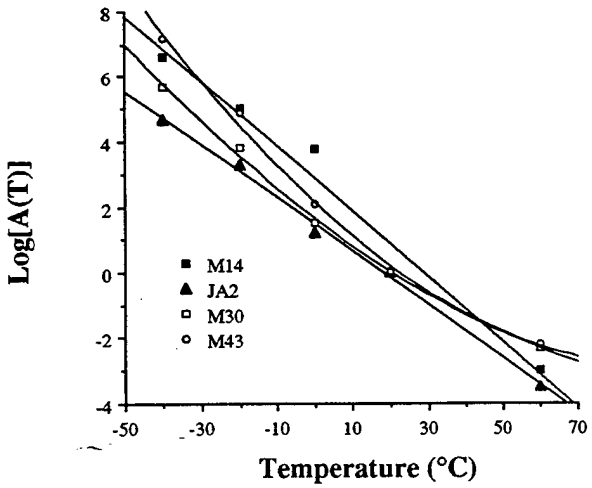


Figure 7. Shift Factors Needed to Generate Equivalent Time-Temperature Conditions

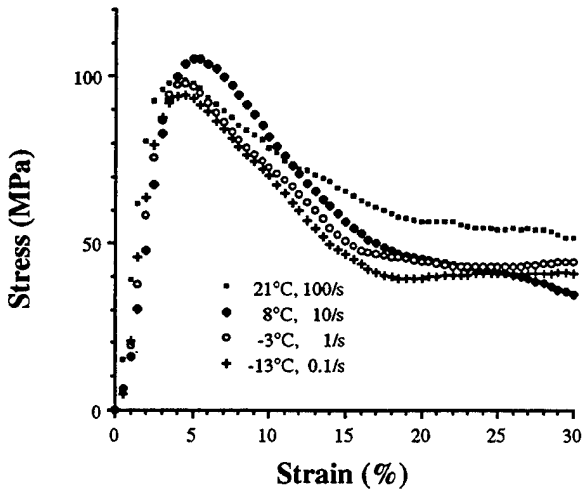


Figure 8. Mechanical Response of M43 at Equivalent Time-Temperature Conditions

to failure. For each propellant, very similar values for maximum stress, strain at maximum stress, yield stress and strain, compressive modulus, and failure modulus were observed for each propellant tested under the predicted equivalent conditions. The results provide great confidence in the ability to predict mechanical and failure response of materials at rates outside those available within the laboratory by employing time-temperature equivalence.

2.4 Propellant Morphology. The physical structure of a propellant is important for understanding the mechanical response in general and the failure response in particular. It can also expose processing problems and offer insights to appropriate corrective measures. Understanding the morphology can also aid in the evaluation of associated propellant properties, such as burning rate.

To glean as much information as possible from the morphological structure, it is important to maintain the integrity of the intrinsic morphology. Specimen preparation is therefore a very important step in the process of exposing features of interest. If the process alters or masks critical indicators, the purpose of the procedure is defeated. The primary tool used for documenting the structure is the SEM, which can be used to quickly determine features of exposed surfaces. In this manner, morphological properties, such as particle size distribution, binder-filler interaction, defect identification, defect concentration, and constituent structural uniformity (perforation and grain diameter), can be evaluated. If, for example, a material shows a lower stress at failure and greater friability than expected, and if it is also noted that the particle binder interaction was weaker than normal, or that voids had been introduced into the propellant during extrusion, then steps can be taken to change the formulation or processing to enhance the interaction or eliminate the voids. Once corrected, the mechanical response of the propellant with the desired morphology can be evaluated. If the formulation had favorable chemical properties, the mechanical response may improve sufficiently to keep it from being eliminated from consideration simply because of poor processing.

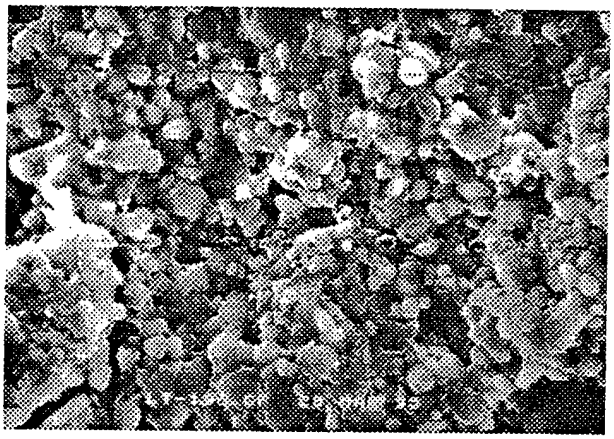
As mentioned previously, it is essential that the preparation procedure preserves the intrinsic morphology in specimens, that a record keeping system is in place that allows easy retrieval of recorded information, and that SEM techniques are developed that expose important morphological features. Such a set of procedures for gun propellants is outlined in Kaste, Ceasar, and Lieb.[18]

As an example of a recent investigation, consider the case of new thermoplastic elastomer (TPE) propellants that are being developed. Morphological investigations were performed on two composite candidates, one filled with RDX and the other filled with CL-20. Selected micrographs appear in Figure 9 and indicate a significant difference between the internal structures of the two propellants. However, before the micrographs were analyzed, similar morphologies were expected, since both propellants had the same percentage of filler, and, initially, the particle sizes were thought to have had the same distribution. It is clear that the representations in Figures 9a and 9b show a marked difference. The conclusion was drawn that the CL-20 underwent a transition that

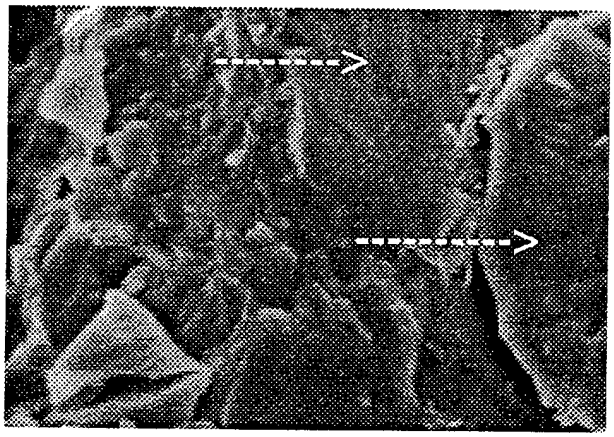
incorporated the particles into the binder. It was later realized that the size of the CL-20 particles when they were mixed was near 100 μm, while the RDX distribution centered on 5 μm. If this were the only difference, then Figure 9c should appear similar to Figure 9a. However, it is also known that, given equivalent binder-filler interaction and crystal strength, fracture proceeds much more easily through larger crystals than smaller ones. There are indications in the CL-20 propellant of a few exposed fractured crystals (indicated by arrows), but not nearly enough to account for the large percentage of filler in the propellant. However, unlike the poor adhesion between RDX and most binders, the particle-binder interaction in the CL-20 filled propellant is much stronger. This makes it possible that the crack could proceed through the binder alone, thereby occluding the view of the particles. The split particles observed in Figure 9b are very well integrated with the binder, indicating good binder-filler interaction. However, these few particles that show may also be indicative of some metamorphosis. In any case, the CL-20 propellant appears much more homogeneous than the RDX. This information helps to explain the "noncomposite" mechanical response of the CL-20 propellant, offers insight into favorable burning rate behavior, and may provide additional information regarding the processing techniques being established for this class of propellant.

To resolve issues raised by these observations, the morphology of extinguished CL-20 propellant grains will be examined, as was done in a previous study.[19] Quickly extinguishing burning surfaces by rapid pressure reduction preserves many features of the surface exposed to the flame. By examining the solidified melt layer and probing the morphological disruptions under the extinguished surface, much insight can be gained into the nature of the combustion process.

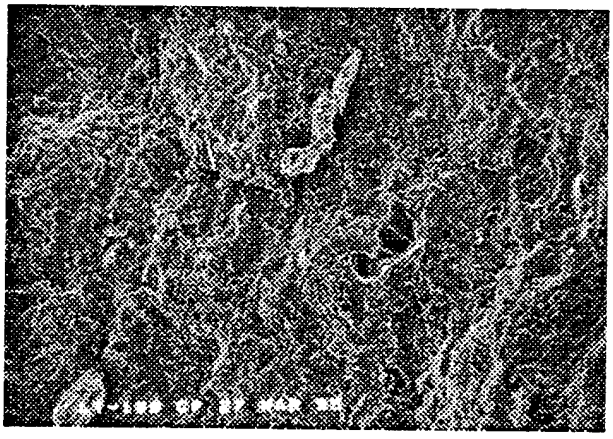
It is easy to see that propellant morphology can provide valuable information concerning constituent compatibility, processing tech-



a. RDX Filled (700X)



b. CL-20 Filled (700X)

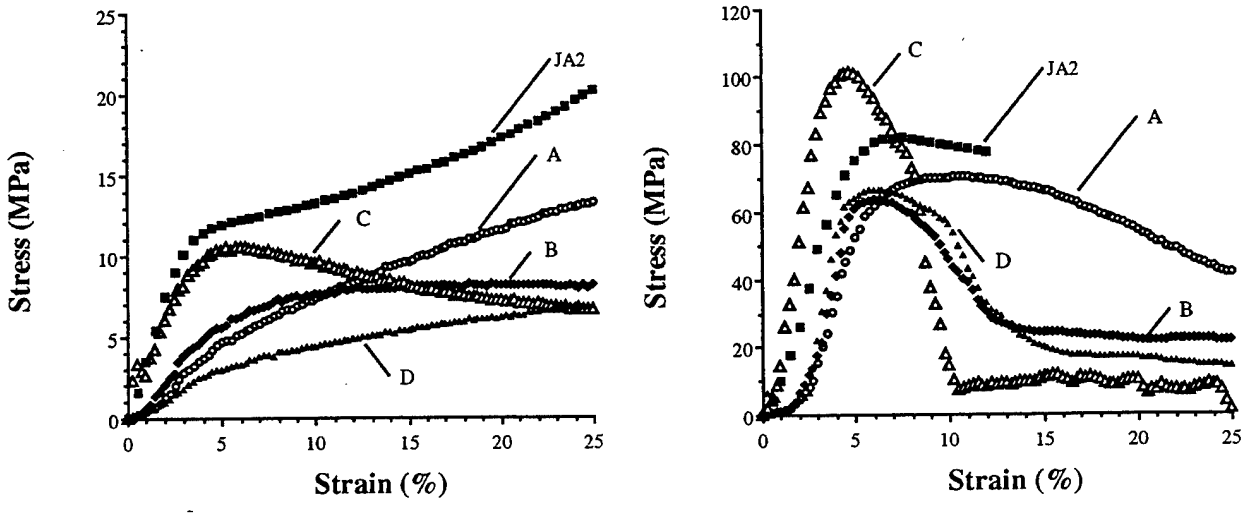


c. CL-20 Filled (30X)

Figure 9. SEM Micrographs Showing the Difference in Morphology Between RDX and CL-20 Filled Propellant

niques, mechanical response, and combustion characteristics, as well as the presence and nature of defects. As experience is gained in interpreting micrographs, more patterns are recognized, and the application of the technique is enhanced.

2.5 Application of Mechanical Response and Morphological Information to Advanced Propellant Formulation Selection. The application of these technologies to the selection of an advanced propellant from several candidate propellant formulations is outlined next. Four formulations of a new TPE propellant were evaluated at selected temperatures (-20° C, 20° C, and 49° C). These temperatures were chosen so the mechanical response of the materials could be sampled across the temperature range of ballistic interest. The propellants were designated as JA2, A, B, C and D, which, except for the JA2, were all highly filled composites. JA2 is a TPE with excellent mechanical properties. The goodness of the JA2 response is indicated by increased strength after failure, a sign of work hardening, and no brittle failure at temperatures above the glass transition temperature, which is near -20° C. The propellants were all found to be softer than JA2, which is the softest conventional gun propellant. Figure 10a shows the response curves for these propellants at 49° C (the JA2 curve was taken at 50° C) and shows the relative softness of the propellants. The only propellant that shows indications of a brittle-type response is C. However, high-temperature creep is a concern for propellants softer than JA2. The propellant that shows the most strength, other than JA2, and is characterized by a response very similar to JA2 is the A propellant. This gives A the best response at high temperatures among the new propellant formulations. The response curves at -20° C are shown in Figure 10b. All the curves except JA2 and A show brittle responses. The A propellant is softer than JA2 and shows loss of strength at high strain, but it is able to maintain load bearing capability up to 25% strain. The new propellant with the best response, compared to the other new formulations, is therefore the A formulation. If the softness of the propellant remains a concern, the level of filler can be increased or the ratio of hard to soft blocks in the co-polymer binder can be increased to increase stiffness.



a. 49° C

b. -20° C

Figure 10. Mechanical Response Curves of Advanced Propellant Formulations Compared to Conventional JA2 Propellant

The amount of material required for the morphological and mechanical response evaluation was less than 25 g, which makes these tests attractive for experimental lots. However, it should be kept in mind that the method of processing helps determine propellant response and morphology. Small batch processing often produces weaker propellant with greater numbers of defects, even though essential features of the mechanical response are usually indicated in the smaller batches. Marked improvements in mechanical response and morphological structure are noted when processing lot size is increased. In this fashion, the mechanical response of emerging propellants can be evaluated early in development, and sound choices can be made to enhance the propellant performance and safety.

3. SUMMARY AND CONCLUSIONS

The mechanical and fracture responses of the four basic types of conventional gun propellant have been characterized over the temperature and strain rate ranges of ballistic interest. In addition, these techniques can be used to characterize the response of new propellant formulations, so that the response during development can be monitored and improved. Fracture surface area profiles have been characterized for the two most friable propellant types (triple-base and nitramine composite), and surface area generation has shown a strong dependence on the level of strain suffered during deformation and, to a lesser degree, on the failure modulus [$\log(E_f)$]. However, the magnitude of the failure modulus clearly indicates the propensity of the material to fracture, and mechanical response measurements show the stress and strain levels at which failure occurs. The measurement of the shift factors for the various propellant types and the demonstration of time-temperature equivalency extend the range of response measurement into areas previously unavailable because of equipment limitations. This equivalency was also shown to extend into the failure arena, which permits a much broader application of the failure characterization. Techniques have been developed for and applied to the examination of the morphological features of energetic materials. Processing problems indicated by the presence of defects (voids, agglomerations, foreign particles, incorrect particle size, etc.), or other evidence of undesirable morphologies (poor mixing, weak particle-filler interaction, preferential fracturing, etc.) can be detected and corrected early in the propellant development. Mechanical response characteristics are made clearer by understanding the morphology, and the burning processes can be better understood by examining surface features of both virgin and extinguished specimens. Many tools have been developed that detect and minimize the deleterious effects of material failure in energetic materials. These tools can be employed at low cost to characterize the mechanical and fracture response, and to check the morphological structure. This information can then be used to assess the influence the mechanical response will have on the performance and vulnerability responses in a variety of operational situations.

4. FUTURE DIRECTIONS

The investigation of two areas would serve to greatly enhance the value of the information accumulated thus far. The first is the establishment of a pressure-temperature equivalency, similar to the time-temperature equivalency recently established for gun propellants. It is known that the response of gun propellants changes significantly with pressure.[20,21] The material toughens and gets stronger as the ambient pressure increases. This property underscores the importance of rapid and uniform pressurization during the ignition phase of the ballistic cycle. The likelihood of fracture is significantly reduced under uniform and rapid chamber pressurization. Quantifying the pressure-

temperature equivalence would permit meaningful predictions of mechanical and fracture response under a much broader scope of interior ballistic conditions. Establishing this equivalence, however, will be difficult at high strain rates and typical large-caliber gun pressures (~700 MPa). The second area involves the measurement of the augmentation of propellant burning resulting from the newly fractured surface. It is known in the propellant and explosive communities that fracture is associated with the emission of ions, radicals, and molecular fragments. It has been observed in this laboratory and others[22] that cracks show patches of decomposition products on the fracture surface. The question arises as to how the presence of these energetic emission particles affects the ignition and combustion of the propellant. Analysis of damaged propellant for equivalent surface area, results of which are reported here, has always been performed after a significant time interval between the fracture and the burning. This allows the emission particles from the fracture to be swept away or changed, and the patches of decomposition products to cool. It seems reasonable that ignition of the propellant after these fracture processes have decayed would reduce the rate of reactivity as compared to the processes that would occur if the ignition of these surfaces were immediate. A study to quantify these possible effects would involve the use of a pressure chamber (called a dynamic closed bomb) in which the propellant could be fractured and the chards immediately ignited. The effective surface area could be compared to that of grains damaged in a similar fashion but burned at a later time. Ratios of the effective surface areas as a function of fraction of the charge burned would provide a measure of the magnitude of the increased combustibility due to the freshly fractured surface, and would indicate whether such considerations are of any significance.

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