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Uniaxial Compressive Response of M26A1E1 as a Function of Temperature

George A. Gazonas
Michael G. Leadore

ARL-TR-79

February 1993

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13. ABSTRACT (Maximum 200 words)

A joint Hercules-Olin M14 Replacement Program for the M865 round considered M26A1E1 gun propellant as a candidate replacement for the current M14 gun propellant. This report outlines the results of uniaxial compression tests on M26A1E1 propellant as a function of temperature, -40, -20, 20 and 50 degrees Celsius and constant strain rate, 100 sec 1/s. The mechanical behavior of M26A1E1 is quantified using various mechanical response parameters such as yield stress, yield strain, compressive modulus, "failure" modulus, and absorbed energy density. In general, the mechanical behavior of M26A1E1 is more sensitive to temperature than M14. However, M26A1E1 is less susceptible to fracture damage than M14 over the temperature range -40 to 50 degrees Celsius. Scanning electron microscopy (SEM) of "cold-fractured" surfaces of M26A1E1 reveal the presence of undissolved nitrocellulose (NC) fibers on the order of 20 -58 micrometers. Some NC fibers "pull-out" during the cold-fracture process and leave behind a depression in the propellant surface. The surface of the depression reacts with the electron beam at high magnification forming blisters and extensional cracks.

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1. INTRODUCTION

The Fracture Mechanics Team (FMT) at the BRL became involved in a joint Hercules-Olin M14 Replacement Program whose objectives were to find a propellant replacement for the M865 round. The proposed replacement propellant, M26A1E1 (Hercules experimental formulation, HES-9520.25), is a modification of the M14 propellant. Some advantages of the M26A1E1 propellant include, 1) low production cost, 2) low propellant hygroscopicity, 3) elimination of the carcinogen, dinitrotoluene, (DNT) from the M14 formulation, 4) successful use with the 152-mm Sheridan tank round, and 5) the production process is easily transferred to other propellant facilities.

This report outlines the results of uniaxial compression testing of M26A1E1 propellant as a function of temperature, -40, -20, 20, and 50 degrees Celsius and constant strain rate, 100 sec⁻¹. Of particular interest is whether fractures develop in the propellant as a result of uniaxial deformation, since the presence of fracture damage has been linked to both the vulnerability response (Lu et al. 1991) and enhanced apparent burning rate (Gazonas, Juhasz and Ford 1991) in propellants.

A comparison of the mechanical response parameters (e.g. yield stress and strain, compressive and failure moduli, absorbed energy density) of M14 and M26A1E1 as a function of temperature are also provided. Photographs of the deformed propellant are included as an aid in the description of the macroscopic deformation mechanisms and scanning electron micrographs (SEM) provide a morphological description of the undeformed propellant.

2. EXPERIMENTAL PROCEDURE

2.1 Specimen Preparation. The seven-perforation granular propellant (25 grams) was shipped from the Hercules, Inc., Kenil, New Jersey facility in an explosion-proof cylinder. The

granular propellant starting material (M26A1E1 lot # HES-9520.25) was cut into right-circular cylinders using an Isomet double-bladed saw. A double-bladed saw was used to simultaneously cut both specimen ends parallel to each other and to help maintain coaxial deformation with the cylinder axis. Specimen aspect ratio, length-to-diameter (l/d), is about 2/1. The inert lubricant, molybdenum disulfide, MoS_2 , was applied sparingly to the specimen ends to reduce end friction effects and test variability (Gazonas and Ford 1992). The M14 specimens (lot # RAD-PD-066-1) were prepared and tested in the same fashion in an earlier study (Lu et al. 1991). Propellant chemical compositions appear in Table 1.

Table 1. Chemical Compositions of M14 and M26A1E1 Propellants

Composition (%)	M14 ^a	M26A1E1 ^b
NC (13.15%N) Nitrocellulose	89.0	65.55
DNT-Dinitrotoluene	8.0	-
NG-Nitroglycerin	-	23.51
DBP-Dibutyl Phthalate	2.0	9.75
DPA-Diphenylacetate	1.0	-
EC-Ethyl Centralite	-	0.98
Carbon Black	-	0.21

^aLu et al. 1992

^bFurrier 1992

2.2 Test Apparatus, Data Acquisition and Data Reduction. The High Rate 810 MTS material test system (Figure 1) consists of a conventional two-pole press with a servohydraulically actuated ram that operates from quasi-static velocities to a maximum velocity of about 12 meters/sec; the maximum velocity imparts a strain rate of 1200 sec^{-1} on a 10 millimeter long specimen. A Thermotron temperature controller, Model 5200, permits thermal conditioning of specimens from -85 to

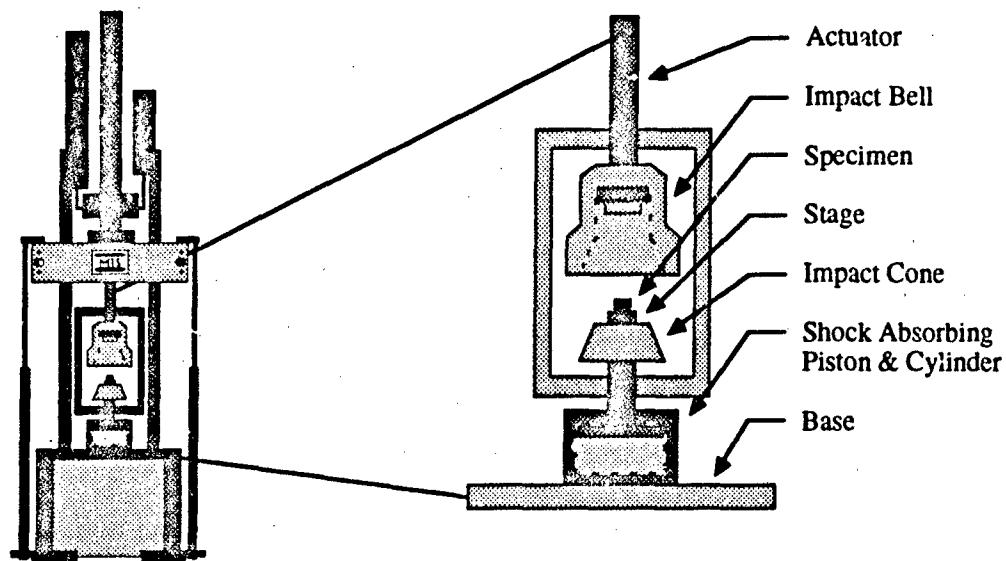


Figure 1. Servohydraulic Test Apparatus.

90 degrees Celsius in an air-circulating oven/refrigerator that houses both upper and lower pistons during testing. Specimens are uniformly heated/cooled and thermally conditioned at the test temperatures -40, -20, 20 and 50 degrees Celsius for at least 30 minutes before each test. Uniaxial compression tests are performed at a constant strain rate of 100 sec^{-1} by computer control of the piston velocity via feedback from an externally-mounted, linear-variable-differential-transformer (LVDT), MTS Model 244.11. Force is measured with a 60 kN, quartz-piezoelectric, force transducer, Kistler Type 9031A, that is mounted on the upper moving piston. Apparatus stiffness is measured at $91.87 \pm 4.8 \text{ kN/mm}$ (Gazonas 1991).

The raw force and displacement data are acquired, stored, and analyzed using an IQ-300 multichannel processing digital oscilloscope. The raw force and displacement data are reduced to engineering stress, σ , versus strain, ϵ , by normalizing measured quantities to initial specimen area and specimen length respectively. Force data are corrected for temperature changes since the force transducer and piston assembly are housed within the thermal conditioning chamber.

An automatic data reduction program was written for the IQ-300 processing oscilloscope in

an effort to reduce the arbitrariness and operator-dependent preferences involved with picking points from stress-strain curves. For example, in this study, the yield stress is defined as the stress level where the material most rapidly loses its ability to sustain load; the yield stress level is determined by finding the minimum in the second-derivative of stress with respect to time. The second-derivative stress versus time data are somewhat noisy because of the particular finite-difference algorithm employed for calculating derivatives of oscilloscope trace data. However, the derivative data are substantially improved by two successive data smoothings. Satisfactory results are obtained if the first derivative of stress is first smoothed with a twenty-point moving average and the second derivative of stress is then smoothed with a twenty-five-point moving average. Automation of stress-strain data analysis through software programming permits determination of unbiased, operator-independent estimates of mechanical properties.

Equally arbitrary definitions of the yield stress (Malvern 1969), such as the proportional limit definition (stress level at the end of the linear range) or offset method definition (stress level after 0.2 percent offset strain), were not viable as yield stress definitions, since for the former definition a suitable linear range is difficult to determine for these materials, and for the latter definition the yield would occur at fractions of a percent of maximum stress and the strain dependence of yield could not be investigated. Other mechanical response parameters include, the yield strain, energy density absorbed per unit undeformed volume, and compressive and "failure" moduli (see Figure 2

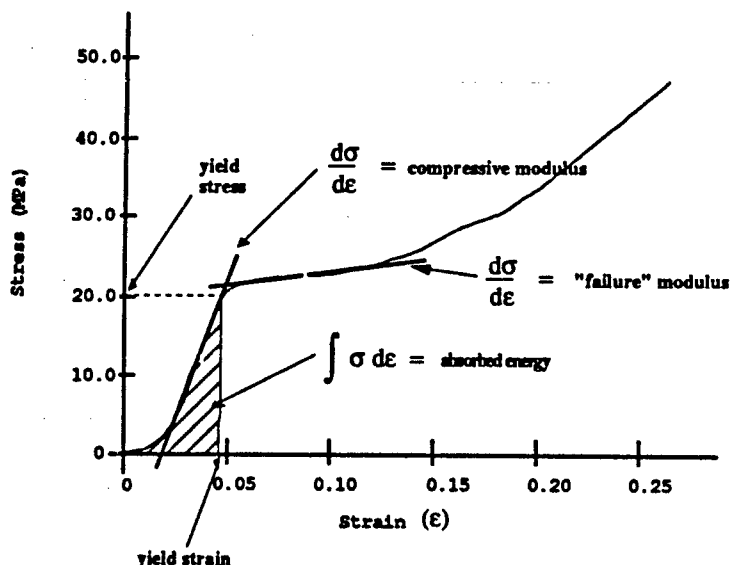


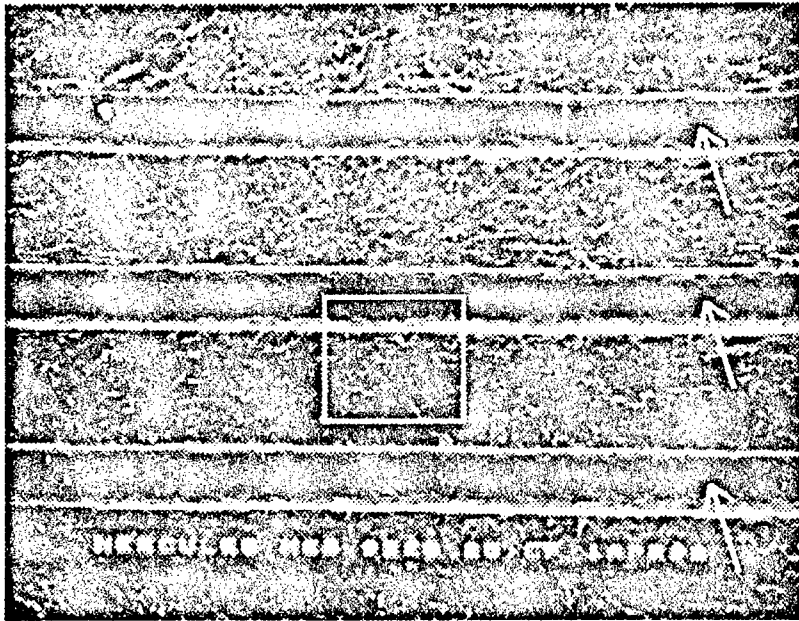
Figure 2. Definitions of Mechanical Response Parameters.

for a graphical illustration of the definition of these quantities).

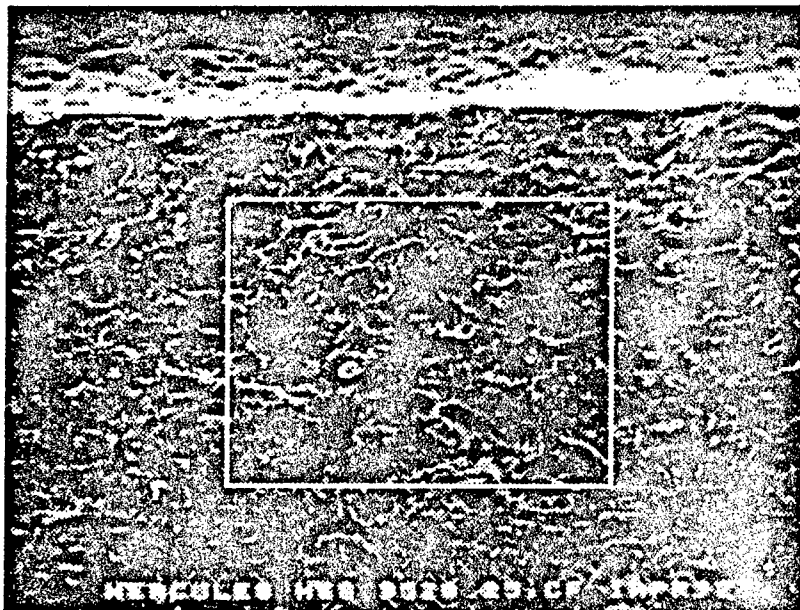
After data reduction is completed, the mechanical property data and other pertinent test information are transferred to a Compaq 286 personal computer via an RS-232 communications port. The data are then imported into a DBASE III Plus database library for subsequent comparison and correlation. A total of 31 mechanical property fields are stored and include propellant ID, lot number, date, compressive moduli, stress and strain at yield, energy absorbed at fixed strain levels from 0.025 to 0.25, specimen dimensions, test temperature, strain rate and a character array for physical description of the deformed propellant.

3. UNDEFORMED STARTING MATERIAL: SEM MORPHOLOGY

The M26A1E1 propellant is longitudinally cold-fractured after the specimen is thermally conditioned in dry-ice for five minutes. The fracture surface is smooth with no observed cracks or voids (Figure 3a). Subsequent SEM micrographs (Figures 3b through 3j) are sequentially magnified for illustrative purposes. The specimen contains undissolved nitrocellulose fibers, not uncommon in highly nitrated nitrocellulose. The diameter of the nitrocellulose fibers range between 20-58 micrometers. Also observed are very small particles with diameters less than 2 micrometers (possibly carbon-black). Some nitrocellulose fibers appear to have been pulled out when the specimen was prepared by cold-fracturing. The pull-out regions appear as depressions or "footprints" of undissolved nitrocellulose fibers (Figures 3g and 3h). A blister of roughly circular shape which contains surface extensional cracks forms when the electron beam is focussed over the area of the "footprint" (Figure 3j). The area immediately adjacent to the depression shows less reactivity to the electron beam. The inhomogeneous reaction sensitivity of the propellant surface might be explained by, 1) a higher concentration of energetic material (possibly nitroglycerin) in the depression or 2) surface roughness or topographic differences between the depression and adjacent areas. The high degree of surface reactivity is unusual for double-base propellants and further research is needed to fully understand the phenomenon.

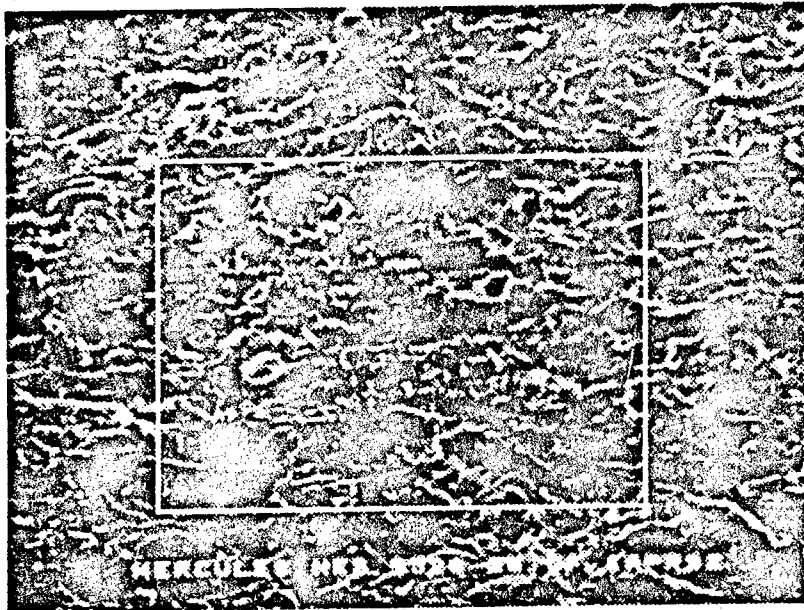


a) Longitudinal section showing cross-section through three perforations (100 μ = 1mm). Highlighted rectangle is magnified in subsequent photographs.

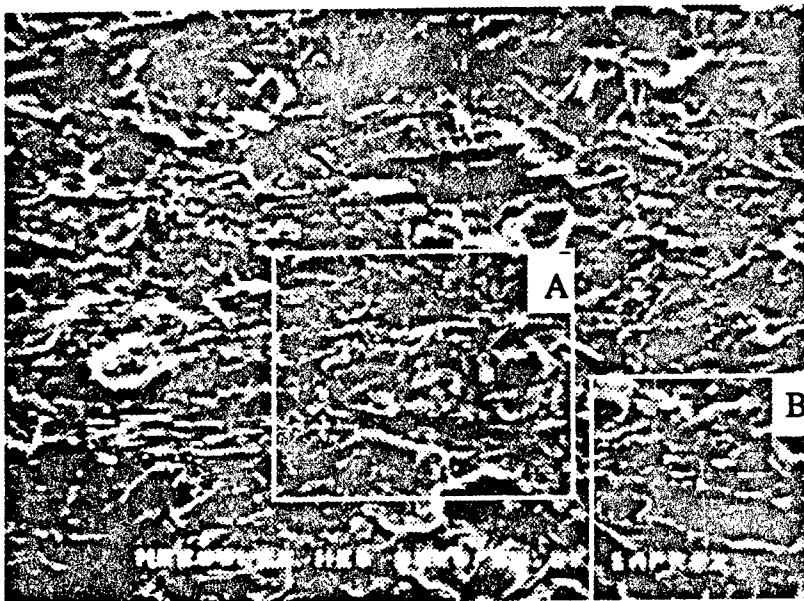


b) Enlarged section of Figure 3a) (100 μ = 5mm).

Figure 3. SEM micrograph of longitudinally "cold-fractured" propellant grain.

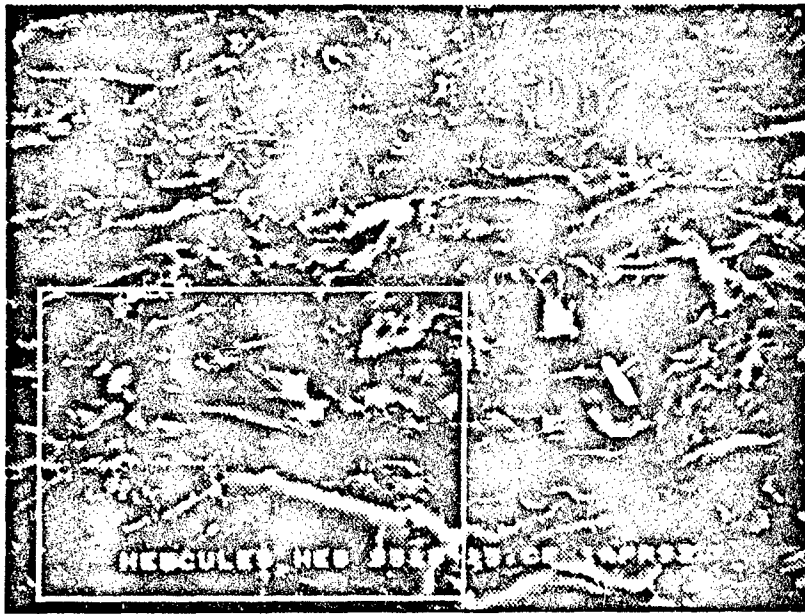


c) Enlarged section of Figure 3b) ($100 \mu = 10 \text{ mm}$).

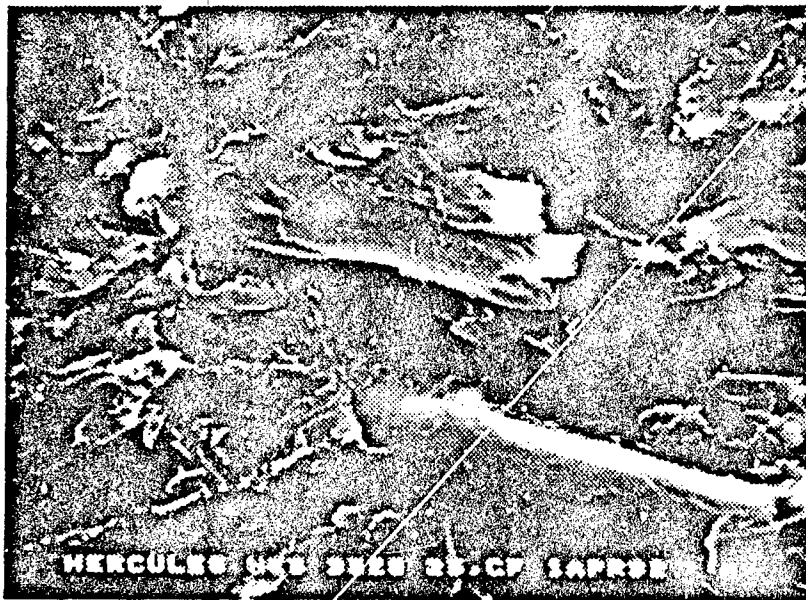


d) Enlarged section of Figure 3c) ($10 \mu = 2 \text{ mm}$).

Figure 3. SEM micrograph of longitudinally "cold-fractured" propellant grain (cont'd)

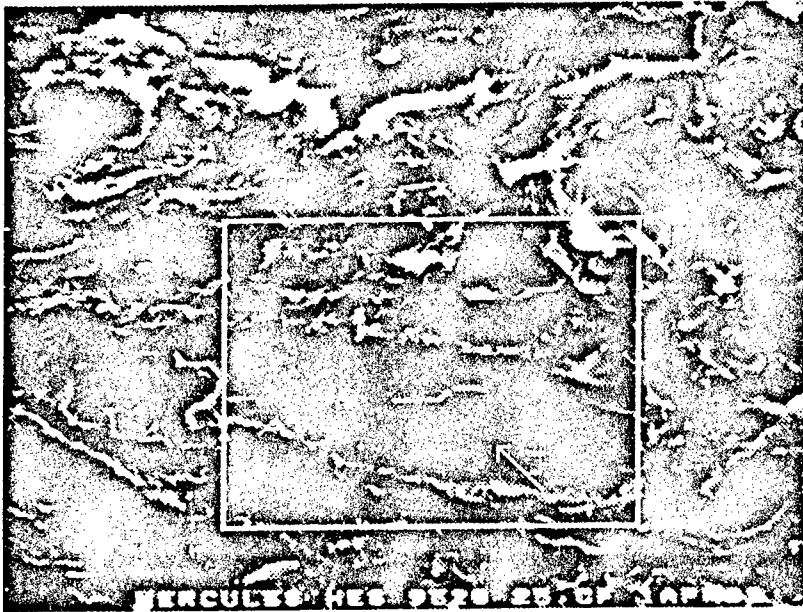


e) Enlarged section of Figure 3d-A) ($10 \mu = 5 \text{ mm}$).



f) Enlarged section of Figure 3e) ($10 \mu = 10 \text{ mm}$).

Figure 3. SEM micrograph of longitudinally "cold-fractured" propellant grain (cont'd)



g) Enlarged section of Figure 3d-B) showing surface depression ($10 \mu = 5 \text{ mm}$).

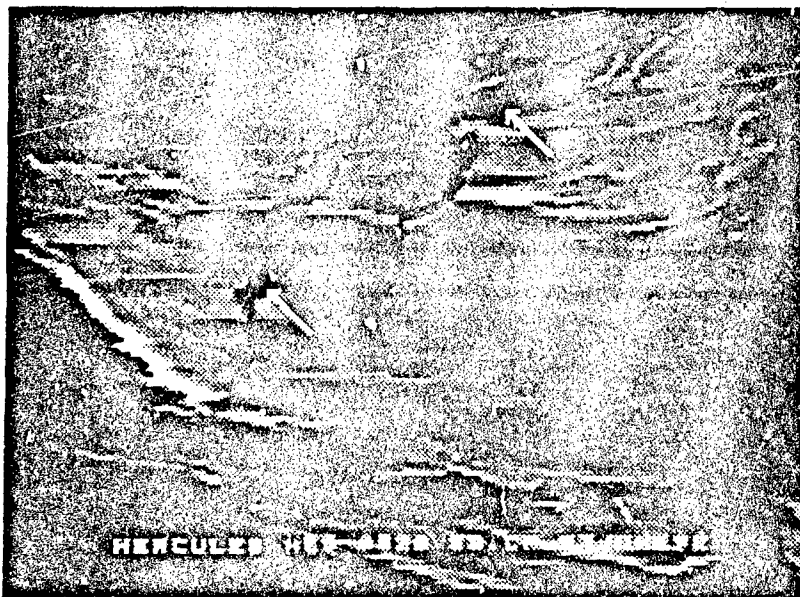


h) Enlarged section of Figure 3g) ($10 \mu = 10 \text{ mm}$).

Figure 3. SEM micrograph of longitudinally "cold-fractured" propellant grain (cont'd)



i) Circular blister with surface cracks in depression. The electron beam is focussed over the area in Figure 3j and is photographed at the magnification ($10 \mu = 10 \text{ mm}$) above.



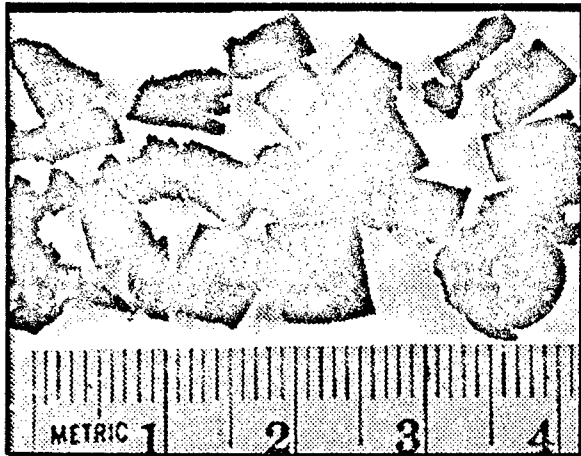
j) "Extensional" cracks in enlarged section of Figure 3i) ($1 \mu = 5 \text{ mm}$).

Figure 3. SEM micrograph of longitudinally "cold-fractured" propellant grain (cont'd)

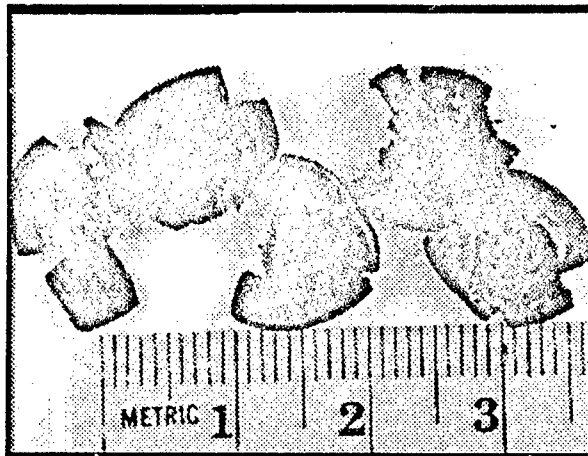
4. UNIAXIAL COMPRESSION TEST RESULTS

Photographs of damaged M26A1E1 specimens at each test temperature. -40, -20, 20 and 50 degrees Celsius, appear in Figures 4a) through 4d) respectively. All specimens are uniaxially deformed at a strain rate of 100 sec^{-1} to a strain level of about 40 percent. Specimens deformed at -40 degrees Celsius fracture through axial splitting and fragment into two or more pieces (Figure 4a). Fracture is also observed in specimens deformed at -20 degrees Celsius, yet specimen fragmentation does not occur (Figure 4b). Some axial splitting is also observed in specimens deformed at 20 and 50 degrees Celsius (Figures 4c and 4d).

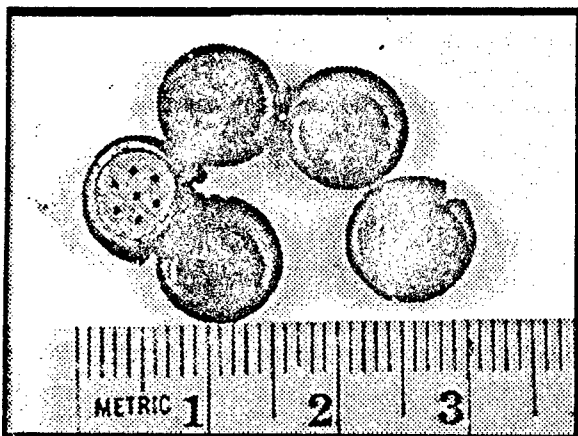
Summary plots (5 tests per curve) which illustrate the experimental results for the uniaxial compression of M26A1E1 and M14 at temperatures of -40, -20, 20 and 50 degrees Celsius and a strain rate of 100 sec^{-1} appear in Figures 5 and 6 respectively. Both propellants exhibit "softening" behavior as temperature increases. Thermal softening is common in materials with temperature-dependent deformation mechanisms. The time axes in these figures are readily converted to strain by multiplying by the strain rate, 100 sec^{-1} . The mechanical behavior of M26A1E1 is very similar to that of JA2 (Gazonas and Ford 1992), and exhibits continual workhardening behavior at all test temperatures except at -40 degrees Celsius where the material slightly worksoftens after reaching a maximum stress level (Figure 5). The macroscopic deformation response of M26A1E1 is ductile since the material maintains significant stress levels to strain levels of 40 percent. In contrast to M26A1E1, fragmentation, and a deterioration of the mechanical strength of M14 is observed at the colder test temperatures, -20 and -40 degrees Celsius (Figure 6).



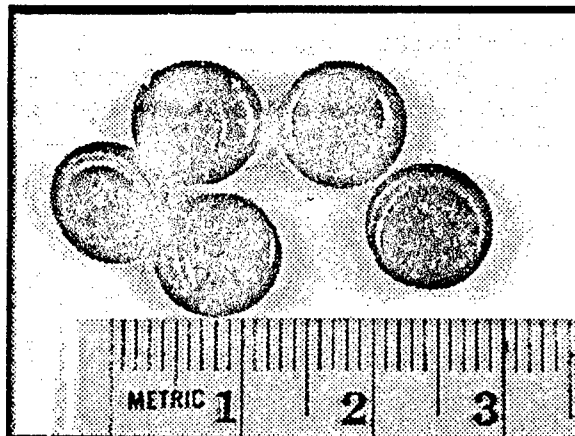
a) - 40 °C



b) - 20 °C



c) 20 °C



d) 50 °C

Figure 4. Photographs of M26A1E1 Propellant Uniaxially Damaged at Various Temperatures.

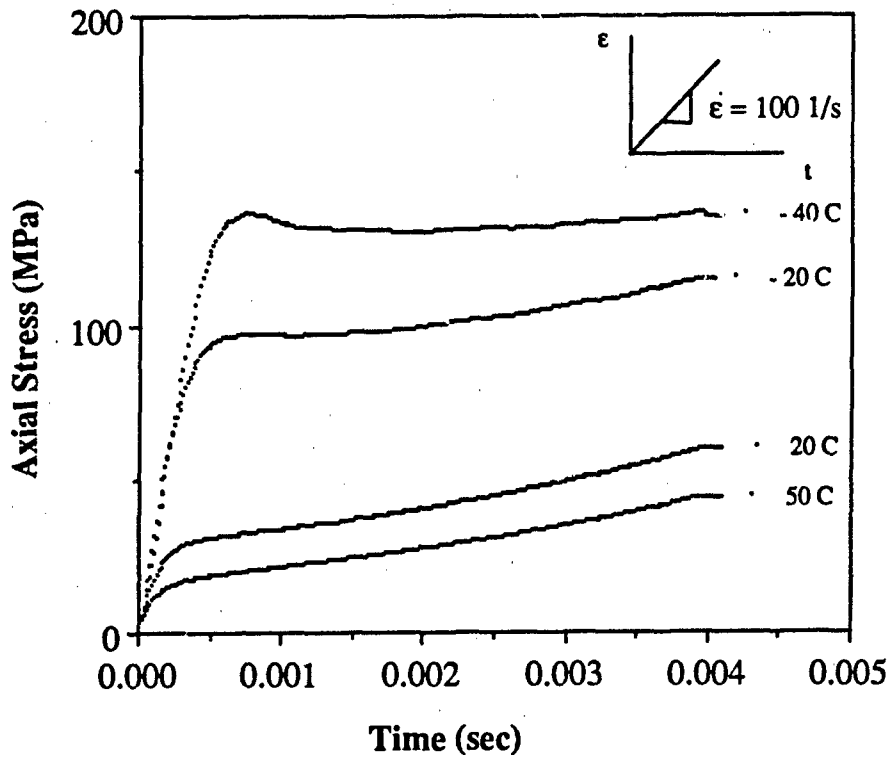


Figure 5. Axial Stress versus Time for Various Temperatures in M26A1E1 Propellant.

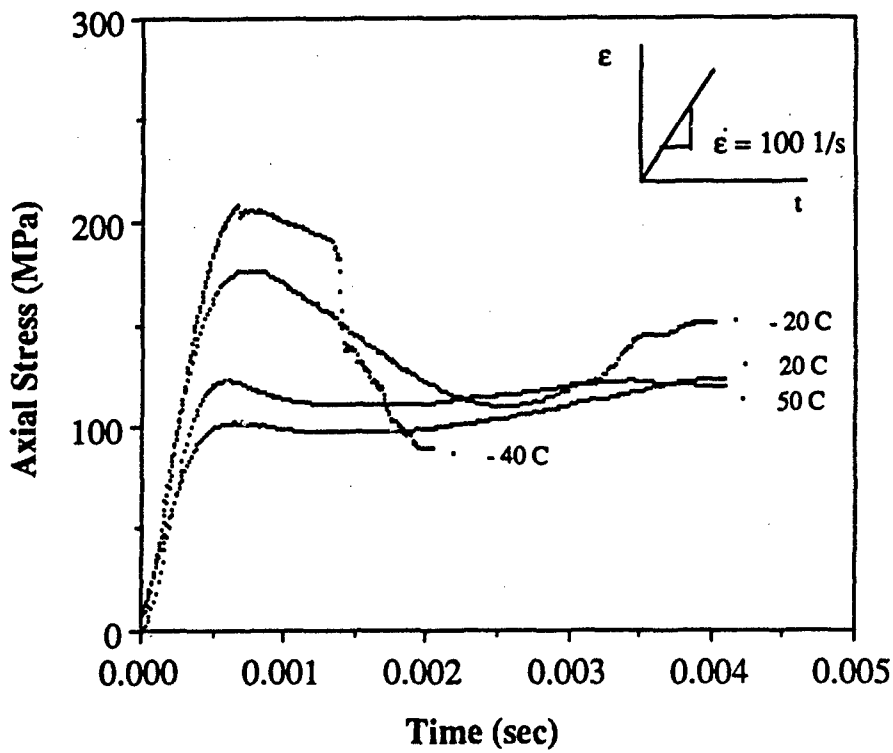


Figure 6. Axial Stress versus Time for Various Temperatures in M14 Propellant.

5. MECHANICAL RESPONSE PARAMETERS

Certain mechanical response parameters aid in quantifying the macroscopic deformation behavior of M26A1E1 and M14 propellants. These parameters were illustrated earlier (Figure 2), i.e., yield stress and strain, energy density absorbed per unit undeformed volume, and compressive and "failure" moduli, and appear as a function of temperature for M26A1E1 and M14 propellants in tabular form in Table 2.

Table 2. Comparative Mechanical Properties of M26A1E1 and M14 Propellants as a Function of Temperature, (a) M26A1E1 (HES-9520.25), (b) M14 (RAD-PD-066-1). Standard deviations are estimated from 5 tests.

Temp., °C	Yield Stress, MPa	Yield Strain, %	Modulus, GPa	Failure Modulus, GPa	Energy Density/Unit Vol., MPa
(a)					
-40	140.2 ± 5	5.5 ± 0.2	2.8 ± 0.2	-0.09 ± 0.01	31.5 ± 0.9
-20	89.5 ± 3	4.4 ± 0.2	2.05 ± 0.05	0.019 ± 0.002	22.6 ± 0.4
20	28.1 ± 1	2.7 ± 0.3	0.8 ± 0.1	0.057 ± 0.005	8.7 ± 0.3
50	16.6 ± 2	2.1 ± 0.2	0.51 ± 0.04	0.054 ± 0.003	5.6 ± 0.2
(b)					
-40	196.4 ± 16	4.8 ± 0.2	4.2 ± 0.5	-0.5 ± 0.2	19.1 ± 4
-20	166.5 ± 7	4.4 ± 0.5	3.7 ± 0.3	-0.36 ± 0.02	19.4 ± 2
20	118.3 ± 7	4.4 ± 0.4	2.9 ± 0.3	-0.14 ± 0.01	25.4 ± 2
50	100.8 ± 7	4.1 ± 0.3	1.9 ± 0.2	-0.031 ± 0.006	22.4 ± 2

The mechanical response parameters are also plotted in Figures 7 through 11. Polynomial expressions are included in each figure for determination of mechanical responses at temperatures other than those provided in Table 2. The yield stress decreases as temperature increases in both M26A1E1 and M14 propellants, yet there is a divergence of the yield stress at higher temperatures indicating that thermally activated deformation mechanisms are more important in M26A1E1 (Figure 7). The yield strain is also observed to decrease as temperature increases in both propellants (Figure 8). In M26A1E1, the yield strain is reduced by nearly a factor of three whereas in M14 the yield strain is reduced by only a factor of 1.2 over the temperature range from -40 to 50 degrees Celsius. Yield strain insensitivity to strain rate and temperature was also observed in previous uniaxial deformation studies on M30 gun propellant (Gazonas and Ford 1992). The compressive modulus of M26A1E1 is also seen to be more sensitive to temperature changes than the compressive modulus of M14 (Figure 9). At 50 degrees Celsius, M14 is about 3.5 times "stiffer" than M26A1E1. The opposite behavior is observed in the post-failure regime, where M14 propellant rapidly loses the ability to sustain load at colder temperatures due to crack coalescence and fragmentation of the specimen (Figure 10). Finally, the absorbed energy density per unit undeformed volume (obtained by integrating the area under the stress-strain curve to a strain level of 0.25, see Figure 2) is also relatively insensitive to temperature in M14 (Figure 11). In M26A1E1, the decrease in the absorbed energy density with temperature is primarily due to the decrease in stress level sustainable in the propellant as temperature is increased (Figure 5).

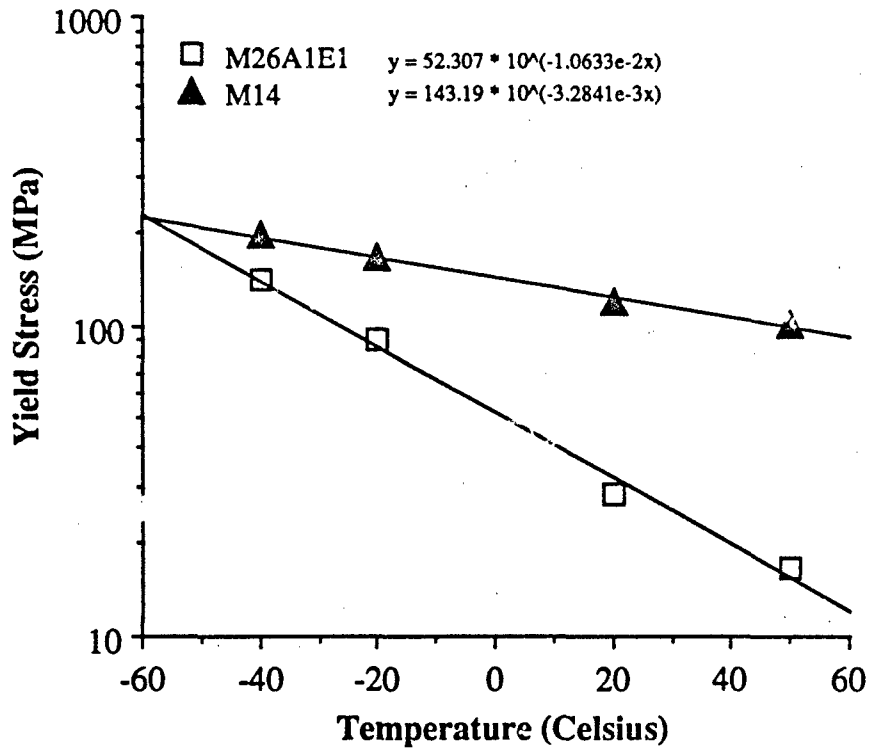


Figure 7. Yield Stress versus Temperature.

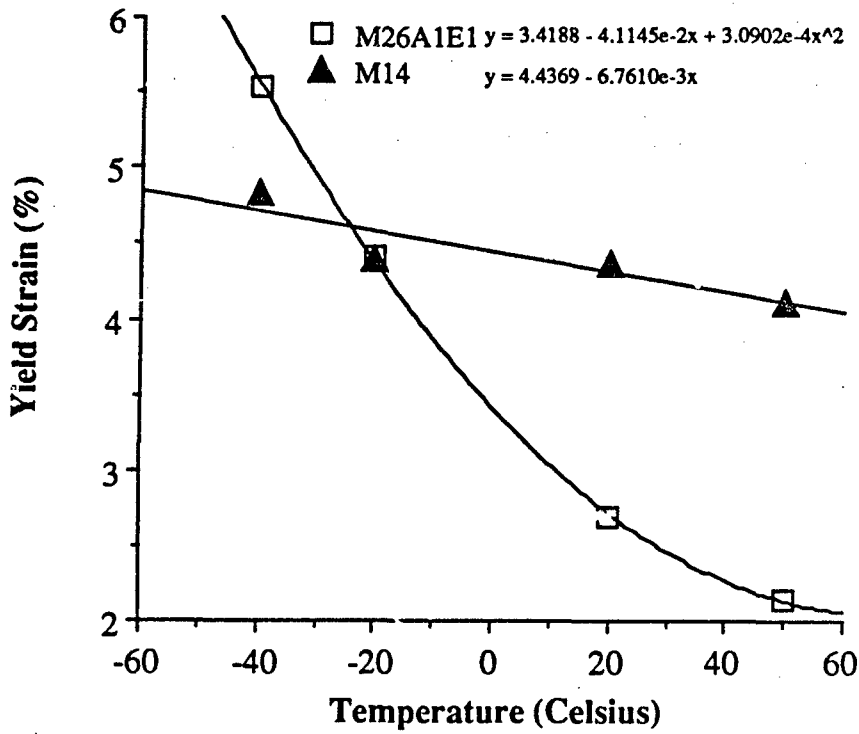


Figure 8. Yield Strain versus Temperature.

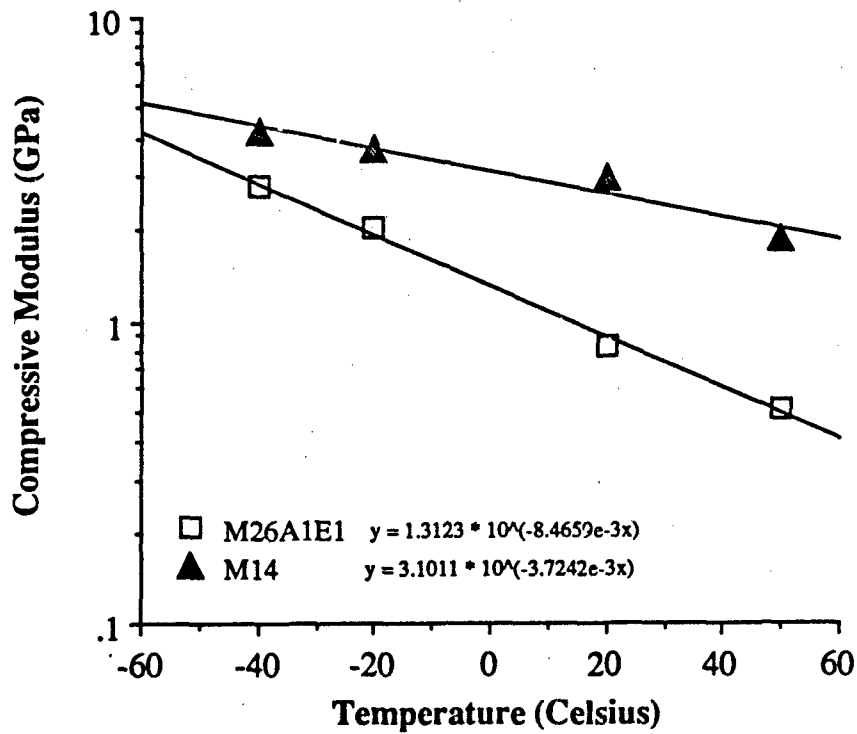


Figure 9. Compressive Modulus versus Temperature.

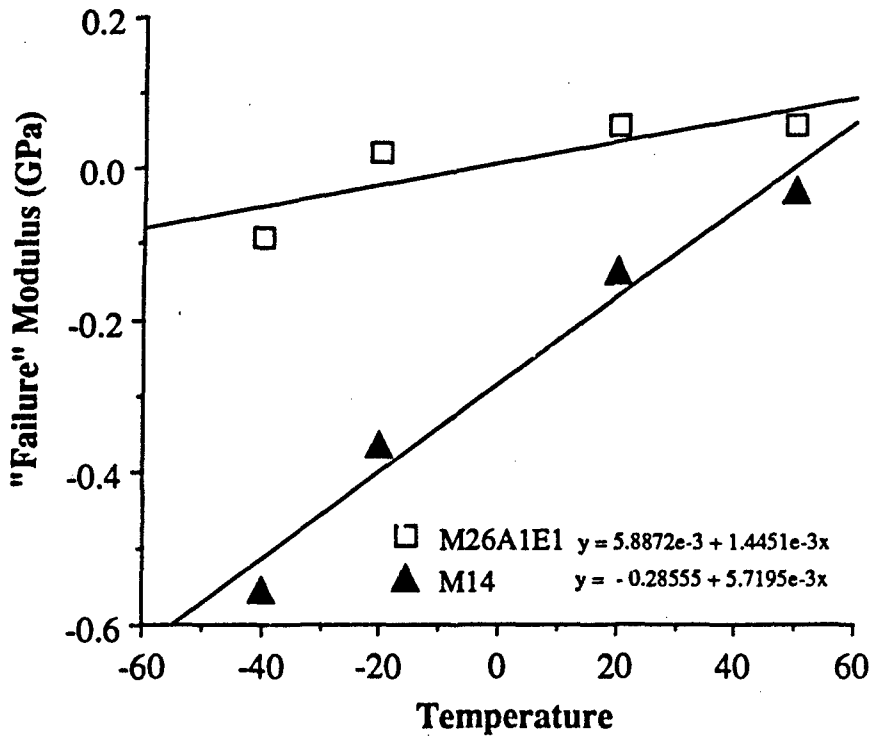


Figure 10. "Failure" Modulus versus Temperature.

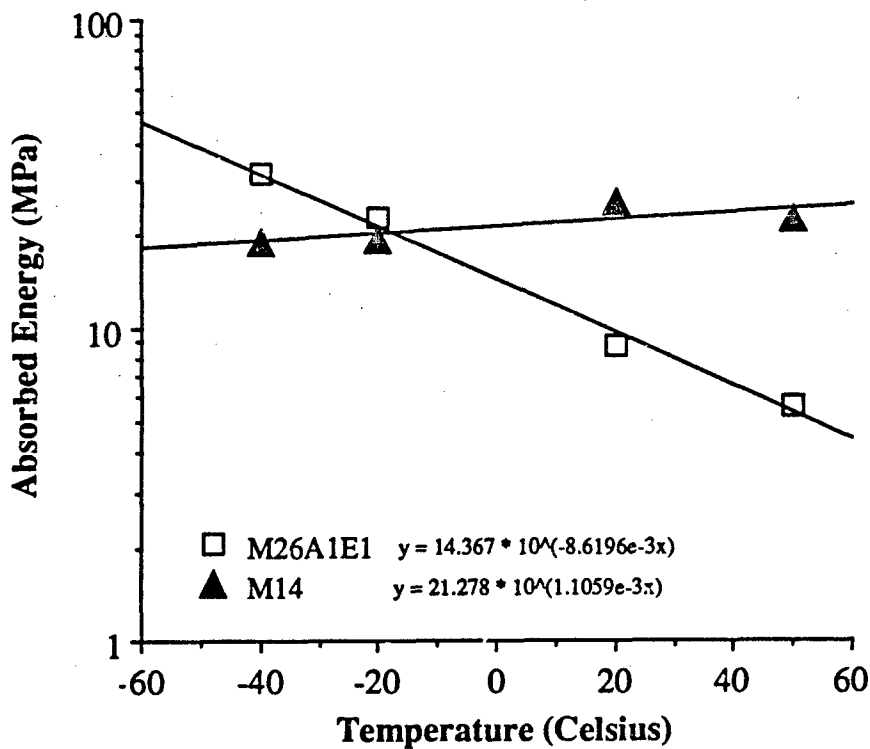


Figure 11. Absorbed Energy versus Temperature.

6. SUMMARY AND CONCLUSIONS

1) The uniaxial compressive response of M26A1E1 propellant is determined as a function of temperature, -40, -20, 20 and 50 degrees Celsius and constant strain rate, 100 sec^{-1} , and compared with the mechanical response of M14 propellant.

2) Axial splitting is observed in all M26A1E1 specimens deformed to 40 percent strain, but fragmentation into two or more pieces is only observed in those specimens tested at -40 degrees Celsius.

3) Scanning electron microscopy of longitudinally cold-fractured specimens of M26A1E1 reveals the presence of undissolved nitrocellulose fibers on the order of 20 - 58 micrometers. Depressions in the propellant surface form as a result of "pull-out" of the nitrocellulose fibers during the cold-fracture process. The depression areas are also highly reactive to the incident electron beam and surface decomposition by blistering occurs as the energy density of the electron beam is increased. Regions outside the depression show less reactivity and indicate that the depression surface might contain a higher concentration of energetic material (possibly nitroglycerin) than adjacent areas. It is also possible that surface roughness or topographic differences between the depression and adjacent areas cause the inhomogeneous surface reactivity. Further research is needed to study the phenomenon.

4) A comparison of the mechanical response parameters, yield stress and strain, compressive modulus, and absorbed energy density, between M26A1E1 and M14 propellants reveals that the mechanical response of M26A1E1 propellant is more sensitive to temperature than M14 propellant. However, the "failure" modulus in M14 propellant is more sensitive to temperature since fracture deformation mechanisms dominate in this material.

5) Since M26A1E1 propellant is less susceptible to fracture damage than M14 propellant over the temperature range -40 to 50 degrees Celsius and at a strain rate of 100 sec^{-1} , then M26A1E1 propellant will also be less likely to exhibit as significant an increase in apparent burning rate as M14 propellant. In addition, previous work has shown that propellant vulnerability is correlated to fracture damage (cold propellant is more vulnerable and mechanically friable than hot propellant).

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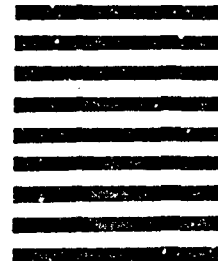
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