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MECHANICAL AND THERMOMECHANICAL PROPERTIES
OF NC BASE PROPELLANTS

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Report Title: Mechanical and Thermomechanical Properties of NC Base Propellants

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Abstract:
Compressive mechanical properties have been investigated as a function of temperature and strain rate for a "modified" double-base propellant (JA2) and comparison made with similar studies for a triple-base propellant. Measurements were made at strain rates of 10 sec(-1) and 10(-3) sec(-1) and temperatures of 80°C to -60°C. At the higher strain rate, the double-base propellant shows ductile behavior at 20°C and brittle failure at the low temperature. However, comparison with triple-base propellants indicates that this double-base propellant is much less brittle at the lower temperature, and considerably less fragmentation is observed for the double-base propellant. While the ductile-to-brittle transition can be related to a glass type transition temperature, the difference in fragmentation is apparently related to composition and structure. The relationship of ductile and brittle failure to abnormal burning and the related unsafe gun firing conditions is discussed.
ACKNOWLEDGMENTS

The authors are indebted to many colleagues for discussions pertaining to this work. Particular thanks are due to Y. Carignan and E. Turngren for detailed discussions of their results of studies of the low temperature phase transition and permission to use some of their unpublished results. Thanks are also due to J. Zucker for permission to use unpublished results and to D. Anderson for making TMA measurements.
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INTRODUCTION

The objectives of this work are: (1) to understand the mechanical properties of gun propellants, e.g., failure conditions as a function of composition, temperature, processing, etc.; (2) to determine the details of how mechanical failure can lead to undesirable and/or hazardous interior ballistics; (3) to use the understanding gained to modify composition, processing, etc., to eliminate/minimize undesirable properties within the constraints of required performance; and (4) to develop standard testing procedures to evaluate a given propellant relative to (2) above. The approach then is to study the mechanical properties as a function of the appropriate parameters such as composition and temperature, to determine the conditions for how mechanical failure can lead to undesirable and hazardous interior ballistics; to use the results and the understanding gained to make the appropriate modifications; and then to perform the necessary tests to demonstrate that a change has been accomplished. This report addresses 1 and 2.

One possible scenario for the role of mechanical failure in interior ballistic abnormalities involves the localized fracture of propellant grains with a resultant increase in the localized pressurization rate leading to the generation of pressure waves in the gun propellant chamber (ref 1). There is a correlation between the magnitude of the pressure waves and the maximum chamber pressure, with indications that under some conditions the peak pressure build-up is sufficient to cause breech blows.

EXPERIMENTAL

The investigations have proceeded in three general area: studies of mechanical properties; use of the standard closed bomb test to determine the effect of mechanical failure on the rate of pressurization; and studies of structural phase transitions and their relationship to mechanical properties.

Mechanical Properties Studies

All experiments were performed in compression with the samples machined from grains into right circular cylinders. The ends of the samples were lubricated to minimize friction. The ends of the samples were lubricated to minimize friction. Two strain rates were used, one leading to "failure" in the millisecond (interior ballistic) time frame and the other quasi-static. Measurements were made as a function of temperature between approximately 80 C and -60 C (refs 2 through 4).
Closed Bomb Studies

Standard closed bomb techniques were used to obtain dp/dt versus p and p versus t (refs 4 and 5).

Structural Phase Transitions

Standard thermal analysis techniques including thermal mechanical analysis (TMA) and differential thermal analysis (DTA) were employed.

Table 1. Table of propellants studied

<table>
<thead>
<tr>
<th></th>
<th>M1</th>
<th>M8</th>
<th>M26</th>
<th>JA2</th>
<th>M30A2</th>
</tr>
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<tbody>
<tr>
<td>Nitrocellulose</td>
<td>85.0</td>
<td>52.2</td>
<td>67.5</td>
<td>63.5</td>
<td>27.0</td>
</tr>
<tr>
<td>% Nitrogen</td>
<td>13.15</td>
<td>13.25</td>
<td>13.15</td>
<td>13.00</td>
<td>12.57</td>
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<tr>
<td>Nitroglycerin</td>
<td>---</td>
<td>43.0</td>
<td>25.0</td>
<td>14.0</td>
<td>22.5</td>
</tr>
<tr>
<td>Nitroguanidime</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>46.3</td>
</tr>
<tr>
<td>Ethyl centralite</td>
<td>---</td>
<td>0.6</td>
<td>6.0</td>
<td>---</td>
<td>1.5</td>
</tr>
<tr>
<td>Diethylene glycol dinitrate</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>21.7</td>
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</tr>
<tr>
<td>Dinitrotoluene</td>
<td>10.0</td>
<td>---</td>
<td>---</td>
<td>---</td>
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</tr>
<tr>
<td>Dibutylphthalate</td>
<td>5.0</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Diethylphthalate</td>
<td>---</td>
<td>3.0</td>
<td>---</td>
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</tr>
<tr>
<td>Potassium nitrate</td>
<td>---</td>
<td>1.20</td>
<td>0.75</td>
<td>---</td>
<td>2.75</td>
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<tr>
<td>Barium nitrate</td>
<td>---</td>
<td>---</td>
<td>0.75</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Magnesium oxide</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>0.05</td>
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<tr>
<td>Akardite II</td>
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<td>---</td>
<td>---</td>
<td>0.05</td>
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<tr>
<td>Graphite</td>
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<td>0.30</td>
<td>0.07</td>
<td>---</td>
</tr>
<tr>
<td>Graphite glaze, max</td>
<td>---</td>
<td>---</td>
<td>0.15</td>
<td>0.50</td>
<td>0.15</td>
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</table>
Studies have been made of several propellant formulations. The compositions of propellants considered in this report are given in table 1. While the emphasis to date has been on triple-base propellants, investigations have also been made of double-base and single-base propellants.

RESULTS AND DISCUSSION

Mechanical Properties

The results for a triple-base propellant are presented and compared with those for a double-base propellant. Compressive stress versus strain curves are given in figure 1 as a function of temperature for M30A2 at the higher strain rate. The results are typical of a polymer/plasticizer system with plastic flow apparently occurring at the higher temperatures. With decreasing temperature the apparent modulus and ultimate strength increase while the strain at failure and the work to produce failure decrease. A ductile-to-brittle-like change in grain failure occurs between 0°C and -15°C as evidenced by the sample breakup (fig. 2). These photographs show the sample and/or fragments after compression at various temperatures. At room temperature only moderate cracking is observed, even for large compressive strains, while at low temperature severe fragmentation occurs at small strains (fig. 1).

Compressive stress versus strain curves are given in figure 3 for the same propellant over approximately the same temperature range but at the lower strain rate. The results show large strains at -45°C and indicate the importance of strain rate for the occurrence of brittle fragmentation-type failure in this propellant. It must be emphasized, however, that in no case have samples been sectioned to search for evidence of internal cracking. All comments pertaining to failure relate to external appearances and the stress versus strain curves.

Stress versus strain is given in figure 4 for the modified double-base propellant JA2 at two temperatures and at the higher strain rate. JA2 is made using a solventless process, while M30A2 (and the other propellants of table 1) is made using solvents. A very limited number of samples of this propellant were available; therefore, the results must be taken as somewhat preliminary. As for the triple-base propellant, there is evidence of plastic flow at the higher temperature and a fragmentation type failure at -45°C; some plastic flow may occur at -45°C. The degree of brittleness encountered at lower temperatures for M30A2 is not observed for JA2 as evidenced by the photographs in figure 5 for the two temperatures. Side and top views of the samples are

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1 J. Zucker, private communication.
given after compression. However, by going to a lower temperature and a slightly higher strain rate, brittle fragmentation was also observed for JA2. Plans have been made to study the fracture surfaces by scanning electron microscopy and x-ray photoelectron spectroscopy to determine the role of added solids in the failure of the triple-base propellants. Differences in plasticizers and processing may also account for some of the differences in mechanical failure characteristics of M30A2 and JA2.

Compressive stress versus strain curves for the double-base M26 propellant are given in figure 6 at two temperatures and the lower strain rate. The results indicate a large "softening" at the higher temperature. A phase transition has been observed by TMA in the vicinity of 40°C. This temperature is between the two temperatures of figure 6 and probably is associated with the differences between the two curves of this figure.

Closed Bomb Studies

Typical closed bomb results are given in figure 7 in the form of dp/dt versus p curves for undeformed grains of M30A2 propellant (refs 3 and 4). Also given are the results for fragments of grains of this propellant obtained by compression at -45°C at the higher strain rate. A large increase in dp/dt is observed, which is to be expected because of the large increase in surface area due to fragmentation. Significant increases in dp/dt were also observed for grains deformed at 20°C. These results lend support to the hypothesis that fractured grains leading to high localized dp/dt could contribute to the generation of pressure waves.

Structural Phase Transitions

Thermal analysis techniques have been used to investigate phase transitions in the polymer/plasticizer propellant system. The objective of this part of the program is to relate mechanical properties to other easily measured physical properties of the propellants. This can lead to further understanding of the mechanical properties, may provide a simple tool for characterizing these properties, and may also be used as a guide to propellant modifications for improved mechanical properties.

DTA data are given in figure 8 for nitroglycerin (NG) and M8 propellant. The onset of a "phase transition" at approximately -70°C for NG and at a somewhat

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2M. Mezger, unpublished results.

3E. Turlgren and Y. Carignan, private communication.

4The phrase "phase transition" as used in this report refers to a glass transition. See, for example, J. Jackie, Phil. Mag., B65, 113, 1987.
higher temperature for M8 strongly suggests that the phase transition in M8 is related to the NG. Other studies indicate that NG undergoes a phase transition in this temperature range from a "glassy" solid to a highly viscous liquid.\textsuperscript{3}

TMA data are given in figure 9 for M30A2 propellant. Again a transition (change of thermal expansion coefficient) is observed in the same low temperature range, suggesting a relationship to NG. Reference to figure 1 shows no indication of plastic flow in this low temperature range. Therefore, there may well be a relationship between this phase transition and the severe embrittlement observed at low temperatures. Additional work is clearly necessary to clarify these matters. It must be noted that the TMA data are taken at an order of magnitude lower strain rate than the data of figure 1. The relationship of this low temperature phase transition to the apparent ductile-to-brittle transition between -15°C and 0°C is also not apparent.

An additional phase transition is observed at about 40°C for M30A2 (not shown in fig. 9). This phase transition is reproducible only under certain conditions of sample thermal history. However, transitions at approximately the same temperature are observed by TMA for M26 and M1 suggesting that they are due to nitrocellulose (NC). This phase transition may account for the large difference in mechanical properties of M26 above and below 40°C (fig. 6). Phase transitions have not been detected in the low temperature range (above -80°C) for M1 and the results for M26 are inconclusive, i.e., the experimental results do not definitely establish or rule out the existence of a phase transition in the vicinity of -70°C. These results are consistent with the low temperature transition being associated with NG since M1 does not contain NG while M26 does contain NG. Clearly, more work is necessary to understand these phase transitions and to relate them to mechanical properties.

**SUMMARY**

The mechanical properties of propellants exhibit the general temperature and strain rate dependencies to be expected of polymeric systems. A triple-base propellant has been found to have a ductile-to-brittle-like transition between 0°C and -15°C at strain rates leading to failure in the millisecond time frame (interior ballistic time frame). A modified double-base propellant also shows embrittlement at low temperature but does not give the extreme fragmentation characteristic of the triple-base propellant. Closed bomb tests indicate that the brittle fragmentation type failure could lead to high localized pressurization rates and so to abnormal and hazardous interior ballistic conditions. Thermal analysis studies indicate two structural phase transitions which can be tentatively identified with propellant composition.

\textsuperscript{3}Ibid.
Figure 1  High strain rate compressive stress versus strain curves as a function of temperature for M30A2 propellant
Figure 2. Deformation and fracture of M30A2 propellant at several temperatures due to high strain rate compression.
Figure 3. Low strain rate compressive stress versus strain curves at various temperatures for M30A2 propellant (J. Zucker, private communication)
Figure 4. High strain rate compressive stress versus strain curves at two temperatures for JA2 propellant
Figure 5. Deformation and fracture of JA2 propellant at two temperatures due to high strain rate compression.
Figure 6. Low strain rate compressive stress versus strain curves at two temperatures for M26 propellant
PROPELLANT: M30A2
LOADING DENSITY: 0.2 GM/CC
TEMPERATURE: 21° C
IGNITION: 0.5 GM BLACK POWDER M100 MATCH

Figure 7. Pressurization rate versus pressure for closed bomb burning of undeformed M30A2 propellant grains and M30A2 grain fragments obtained by high strain rate compression at -45°C
Figure 8. $\Delta T$ versus temperature for differential thermal analysis (DTA) of nitroglycerin and M8 propellant (E. Tourgren and Y. Caognan, private communication)
Figure 9. Linear dimensional change versus temperature for thermomechanical analysis (TMA) of M30A2 propellant (heating rate 0.2°C/min)
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