<table>
<thead>
<tr>
<th>UNCLASSIFIED</th>
</tr>
</thead>
<tbody>
<tr>
<td>AD NUMBER</td>
</tr>
<tr>
<td>AD468449</td>
</tr>
<tr>
<td>LIMITATION CHANGES</td>
</tr>
</tbody>
</table>

TO:
Approved for public release; distribution is unlimited.

FROM:
Distribution authorized to U.S. Gov't. agencies and their contractors; Foreign Government Information; 14 MAY 1965. Other requests shall be referred to British Embassy, 3100 Massachusetts Avenue, NW, Washington, DC 20008.

| AUTHORITY |
| DSTL, DSIR 23/32973, 8 Dec 2008 |

THIS PAGE IS UNCLASSIFIED
The Biaxial Tensile Properties of Plastic Propellant

(Mrs.) S. Cooke
J.H.C. Vernon

The recipient is warned that information contained in this document may be subject to privately-owned rights.
The Biaxial Tensile Properties of Elastic Propellant

by

(Mrs.) S. Cooke and J.H.C. Vernon

Approved: P.R. Freeman
P.R. FREEMAN
S.P.2

Approved for Circulation: G.H.S. YOUNG
P.S.(D)

14th May, 1965

WALTHAM ABBEY
ESSEX

UNCLASSIFIED
1. Summary

2. Introduction
   2.1 Multiaxial Stress Problems
   2.2 Aspects Peculiar to Plastic Propellant
   2.3 The Choice of Test System

3. The Bantam Motor

4. Spin Testing

5. Spin Testing of Bantam Propellants

6. Spin Testing and Consolidation

7. The Diaphragm Test

8. The Crack Test

9. Conclusions

10. Acknowledgements

11. References

Appendix

Figures 1 - 8
Reference: WAC/167/05

1. SUMMARY

In a case-bonded rocket motor the propellant is often subjected to biaxial tensile straining, arising from thermal or pressurisation effects. The behaviour of propellants under biaxial straining differs from that under uniaxial straining, and special biaxial test techniques have to be devised. Plastic propellants have some biaxial characteristics in common with other types of propellant, and others which are peculiar to this type of propellant. An account is given of the way in which biaxial strains arise in certain types of plastic propellant motor, and of the test techniques which have been developed or modified to suit the characteristics of the material. They include spin testing, diaphragm testing and a development of the standard crack test. The effects of time, temperature, age hardening and asymmetry on biaxial properties have been investigated. An account is given of the application of biaxial test results to design problems of the Bantam motor for the Skua meteorological rocket, and to the development of propellants for this application.

2. INTRODUCTION

2.1 Multiaxial Stress Problems

The mechanical properties of plastic propellants are currently measured in a variety of ways, depending on the purpose for which the information is required. Production batches are assessed by the traditional plasticity, crack and flow tests (1), more refined assessment is made in terms of plastoviscosity and yield point, while a wide range of rheological problems has been covered using various forms of tensile test. All these tests have one thing in common - they basically involve uniaxial strain conditions. The sample has one axis of symmetry along which it is strained, and behaviour transverse to this axis is not directly studied; it is assumed that transverse deformation will be such as to keep the volume of the sample constant and no further interest is taken in it.

In the rocket motor, however, deformations in the propellant charge are likely to be much more complicated, and in any analysis of mechanical behaviour the multiaxial nature of the stresses and strains must be taken into account. For some simple materials, behaviour under multiaxial conditions can be predicted from uniaxial behaviour but for plastic propellant this is certainly not the case. It is therefore necessary to study propellant behaviour under various types of multiaxial stress systems. One such system involves tensile testing under a confining pressure; this has already been extensively investigated for plastic propellant (2). Another aspect of considerable importance is behaviour under biaxial tensile conditions.
conditions. Such conditions occur in all case-bonded motors and, as will be shown, they are of particular significance with plastic propellant. Rainbird and Vernon (3) discuss in greater detail the requirements for biaxial testing, and describe the development of a successful test in which a thin membrane of propellant is expanded by pneumatic pressure until failure occurs. This test has been extensively used on polyurethane and cast double-base propellants. It has found some application for plastic propellants, but is not suitable for all the problems which arise, and other test systems have been devised. This report gives an account of these investigations.

2.2 Aspects Peculiar to Plastic Propellant

There are various reasons why plastic propellant has to be given special consideration in a biaxial test programme. Its low strength and modulus mean that microtomed slices are extremely fragile; this causes difficulties in the manipulation of the diaphragm test. The uniaxial extensibilities of many plastic propellants are very large, sometimes 200 or 300 per cent, but it is found that large uniaxial extensibilities are not matched by correspondingly large biaxial extensibilities. This in itself is an important reason for carrying out biaxial tests, since it means that biaxial behaviour cannot be predicted from uniaxial testing. Strain levels in motors are usually much lower than measured uniaxial extensibilities; this is particularly true for plastic propellants, and yet plastic propellant motors fail due to inadequate extensibility, especially at low temperatures. Measured biaxial extensibilities are much more realistic, and offer an opportunity of obtaining laboratory results which correlate directly with motor behaviour.

Another difference between plastic propellant and other types of composite propellant is that the former has a much higher solids content. The rheology of most composite propellants is determined partly by the binder behaviour, and partly by interaction between binder and solids. The latter effect is more important with plastic propellant, because of the high solids loading and the low intrinsic strength of the binder. A third effect, interaction between the solid particles themselves, appears only at the very high loadings used in plastic propellant. There are differences in the way these three components react to a change from uniaxial to multi-axial conditions; solid-binder and solid-solid interactions are affected more than simple binder behaviour. The properties of plastic propellant are therefore markedly different in tension and compression and, for similar reasons, there are major differences between behaviour in uniaxial and biaxial tension.

One property of plastic propellant makes the testing problem easier; the elastic component of any deformation is small, and decays rapidly to zero. After a few seconds, all deformation is permanent. This makes it possible to set up test systems, such as spinning tests, in which

/ deformation ......

- 2 -
deformation can be measured accurately at the end of the experiment, rather than having to attempt measurement while the test sample is still spinning.

The lack of elastic recovery poses certain problems in interpreting the rheology of plastic propellant. With rubbery propellants, tested uniaxially, the tensile stress/strain curve usually shows an initial peak and a "ductile" region in which the load remains constant, or falls slightly, until the sample eventually breaks. Two elongation values, at maximum load and at break, may then be reported. It has been found that the "strain capability" of the propellant (which is, in effect, the strain which can be withstood in the motor) is closer to the elongation at maximum load than the elongation at break. The "constant strain" test value (or maximum uniaxial strain which can be withstood over a long period) is usually similar. Now for plastic propellant the shape of the stress/strain curve resembles that of a rubbery propellant, except that the portion between the two elongation values is usually greater. However, constant strain tests do not give meaningful results, because the propellant does not fail under steady strain conditions. The question arises, therefore, whether the strain capability of a plastic propellant is related to elongation at break instead of elongation at maximum load. While there are several further aspects to be considered (the most important being the effect of fatigue) part of the dilemma can be resolved by analysis of biaxial behaviour.

For certain applications, a case-bonded cigarette-burning charge has considerable advantages over the usual radial burning designs, including simpler geometry, and longer burning times. The main disadvantage is the large strain level at the burning surface due to the effects of temperature change, combustion pressure, or both. This strain is biaxial in nature and increases with length/diameter ratio. Its magnitude is such that a design of this type can only be contemplated for a very extensible propellant such as plastic propellant. Biaxial tensile properties are thus of direct interest in the assessment of propellants for charges of this type.

2.3 The Choice of Test System

Various types of biaxial test are now in use, or under investigation, in the United States. A popular one is the strip biaxial test (4), in which a very wide J.A.N.A.F. test piece is pulled in the usual way. Near the centre of the test piece there is little shrinkage in the plane of the sheet; the longitudinal extension is balanced by compression in only one transverse direction, instead of both. The test is convenient to adopt, since test samples usually are cast in wide bars which are then cut into individual tensile test pieces; the uncut bars are suitable for the strip biaxial test. It has been confirmed, by experiments in which grids are painted on test samples, that the predicted conditions occur in the central region, and one of the strains is zero. The test is not a biaxial tensile test; indeed, it is a matter of opinion whether it may properly be called a

/biaxial .....
bially test at all. It could equally be regarded as a uniaxial test with strain inhibited in one direction. In the rocket motor, one of the tensile strains is usually much larger than the other. Asymmetric biaxial tests (for example the expansion of elliptical diaphragms) can be set up to reproduce this. The strip biaxial test could be regarded as an extreme case of an asymmetrical biaxial test; its conditions are therefore fairly close to those in some rocket problems. It does not, however, give all the information required for plastic propellant, where in some cases the problem concerns equal biaxial tensions. Another objection to the use of this test for plastic propellant arises because of the large extensibility at which the approximations involved cease to be valid.

Another biaxial test sometimes used in the United States is the Fitzgerald diametral compression test (5). This has been extensively analysed and does provide a method of producing stress fields which are substantially different from uniaxial. It is less useful for a propellant for which strain, rather than stress, controls fracture behaviour. It cannot be used for plastic propellant, which is so extensible that it can rarely be caused to fracture in this test, and then only at deformations too large for the theory of the method to be valid.

A test method being developed at I.M.I. for cast double-base propellants involves the stretching of short thin-walled cylinders (6). While the results for such a system would probably be valid for plastic propellant, there would be considerable difficulty handling thin-walled cylinders of this material.

The test systems which have been found practicable are spinning tests, and the diaphragm test. It has also been demonstrated that under certain circumstances the crack test can give information about biaxial behaviour. Details of these tests are given later in this report.

3. THE BANTAM MOTOR

The Bantam motor was developed by R.P.E. as the sustainer motor for the successful Skua Meteorological rocket. Its development has been described by Larlham and Rolfe (7). Requirements for this motor included a long burning time and a small diameter. This can be achieved with a radial burning charge and a propellant of low burning rate, or a cigarette-burning charge and a very fast burning propellant. The latter solution was adopted, and a suitable propellant was developed at E.R.D.E. Plastic propellants can be given very high burning rates by incorporating fine grades of ammonium perchlorate. The propellant chosen, designated RD.2423 (formerly S.1031), contains ammonium perchlorate with an initial specific surface of 8000 cm$^2$/cm$^3$, compared with 2000 cm$^2$/cm$^3$ in conventional plastic propellants. It has a burning rate of 1.50 inches/sec. at 1000 p.s.i.a.
In general, propellants containing finely ground oxidisers are less extensible than usual. They are also more prone to age hardening, and their extensibilities tend to decrease with time (S). The reasons for this are connected with the poor packing achieved with fine powders, and the difficulties of obtaining a favourable particle size distribution by further breakdown during the manufacturing process.

Plastic propellant is invariably case-bonded and its thermal coefficient of expansion is greater than that of the metal from which the case is made by a factor of ten-fold. The thermal and pressurisation strains which arise with all case-bonded motors are particularly severe for cigarette-burning motors. On cooling, the propellant will shrink relative to the motor tube, and the initially flat burning surface will dome inwards. Similar doming will occur on ignition, due to the combustion pressure. If the strains are too large, the charge will fail either by pulling away from the motor wall, or by crazing in the centre of the dome. The greater the length/diameter ratio, the greater is the strain level for a given temperature change (with radial-burning motors, the length/diameter ratio has very little effect). The Bantam motor has an eleven to one length/diameter ratio. The earlier Ladybird motor, on which experience with this type of motor was gained, had a ratio of only three.

At any temperature other than the pressing temperature, the free end will become domed, due to differential thermal expansion. The dome is very nearly spherical, and in the absence of information about the true profile it will be assumed to be exactly spherical. Any error due to this assumption will be small, except at very high strain levels.

Let  $a =$ radius of charge  
$R =$ radius of curvature of dome  
$x =$ height of dome  
$m = x/a$  
$V =$ volume of dome  
$L =$ length of charge  
$\alpha =$ the difference between the volumetric thermal expansion coefficients of propellant and motor  
$T =$ temperature

The volume of the dome is thus the volume of a spherical segment; thus

$$V = \pi x(x^2 + 3a^2)/6 \quad \quad \quad 1$$

$$= \pi a^3 m(m^2 + 3)/6 \quad \quad \quad 2$$

/If  

- 5 -

UNCLASSIFIED
If the dome is produced by differential thermal contraction, then

\[ V = \pi a^2 L a \Delta T \]  \hspace{1cm} \text{----- 3}

\[ m(m^2 + 3) = 6L a \Delta T / a \]  \hspace{1cm} \text{----- 4}

The relation between strain level and dome dimensions is not known for this particular problem; however, the relation was extensively investigated for the case of the pressurised diaphragm by Feraday, Long and Vernon (9). They found that, at the centre of the sample the two biaxial strains each had a value given by:

\[ \epsilon = 2m^2 / 3 - 2m^4 / 15 + 2m^6 / 35 \]  \hspace{1cm} \text{----- 5}

This relation is shown in Figure 1.

For plastic propellant in a metal case, \( \alpha = 3 \times 10^{-4} \text{ deg C}^{-1} \). Figure 2 shows the relation between strain level and temperature change for three values of \( L / a \), one of which is the value for the Bantam motor. The strain level does not increase linearly with temperature change; it builds up slowly at first and then increases rapidly.

For the Bantam motor itself, the values include:

\[ L = 55 \text{ inch} \]
\[ a = 2.46 \text{ inch} \]

The proposed temperature range for the motor is 25 deg C. For a 25 deg C drop in temperature from the pressing temperature, the dome volume, \( V \), is 7.84 inch\(^3\), \( m = 0.32 \) and the strain level is 7 per cent. Standard procedure, however, is to press the charge at the mean operating temperature. The maximum strain now becomes 2\( \frac{1}{2} \) per cent.

On pressurisation, the tube bulges and the volume available to the propellant increases by about 10 inch\(^3\). Thus the strain level due to pressurisation is approximately 12 per cent.

Biaxial strains are not additive; the total strain for thermal and pressurisation effects combined with both acting in the same direction, (i.e., that due to a volume differential of 13.9 inch\(^3\)) is not 14\( \frac{1}{2} \) per cent, but 19 per cent.
An early motor cracked badly on temperature cycling; the propellant in this motor had become hard, and clearly had inadequate extensibility. Various investigations were started at R.P.E. to overcome this difficulty. It was decided to press a half-inch layer of another propellant (RD.2424, formerly S.1052) on the burning surface. This burns more slowly than S.1031 and therefore gives a lower initial pressure, and a slower rate of application of pressurisation strains. Current firings with this system are successful. At E.R.D.E., studies included the checking of the extensibility of RD.2423, of the specially developed RD.2424, and of alternative propellants which might have had improved properties. Because of the nature of the problem, biaxial testing, chiefly spin testing, was extensively used in the assessment. Some of the results of these tests are given in Section 5. Studies on Bantam propellants did, of course, include investigations other than biaxial tests, but these are outside the scope of this report.

4. SPIN TESTING

The simplest way to deform a propellant sample biaxially is to use it as a cigarette-burning charge in a small "motor" which is then spun about its own axis. A MSE major centrifuge has been in use for some years for the study of gravitational effects on plastic propellant motors (10). Provision was made for a motor to be spun about its own axis, mainly in support of investigations on spinning rockets, such as the gun assisted rocket. The biaxial test was developed from this system.

The motors used for this work have an internal diameter of 1.875 inches; the charge length is normally 2 inches. Spinning speeds of up to 6000 rev/min. are available. It has been established that plastic propellant shows no significant elastic recovery over the time scale involved, so that the centrifuge may be stopped, and the deformation measured with a suitable gauge. It has further been established that interruption of the spinning process does not affect the deformation rates. A motor spun for 3 periods of 5 minutes, with pauses for measurement, will deform as much as a motor subjected to continuous spinning for 15 minutes.

Normally, the only measurement required is the depth of the centre of the dome; from this the biaxial strain level is calculated according to equation 2.

A problem which arises with all types of biaxial testing concerns the difficulty of establishing when failure has occurred. At some strain level, not usually well defined, a network of small cracks appears on the propellant surface. Later, some of these cracks open out to give a crazed appearance. Little quantitative information is available about the depth and number of cracks needed to cause a catastrophic increase in burning surface. It is known from R.P.E. work on cordite that a flame will penetrate a capillary

/0.007 inch ......
0.007 inch in diameter; however, a charge with cracks considerably wider than this may fire successfully if the cracks are not deep.

In biaxial testing, it is customary to report the strain level at which small cracks appear, and the level at which they open out, as the points of "cracking" and "failure" respectively. It has to be accepted that these are approximate values and their relation with motor failure is not precisely established (and cannot be established except by firing a considerable number of end-burning motors with crazed burning surfaces, at considerable cost in motor tubes). However, the degree of uncertainty is no greater than that arising from lack of reproducibility of properties of any solid propellant.

If the test motor, instead of being spun about its own axis, is placed in the bottom of a centrifuge bucket and subjected to longitudinal acceleration, failure may occur in one of several ways, including shear at the wall and peeling at the upper corner (10). It may also fail by crazing at the centre of the end faces. The stress pattern will be different, but under long-time loadings, and if the failure is strain-dependent, the effect will be the same. An advantage of this test system is that the deformation can be reversed, and then repeated, so that the effect of fatigue can be investigated.

5. SPIN TESTING OF BANTAM PROFELLANTS

The main propellant for the Bantam motor is RD-2423. Early spinning tests showed cracking at a biaxial extension of 25 per cent at 20°C, but failure did not occur over the strain range of which the test was capable. The propellant was known, from uniaxial tests, to be prone to age hardening, and so a series of spinning tests was carried out on aged and unaged samples. The effect of test temperature also was investigated. Results are given in Table 1.

/TABLE 1 .......

- 8 -

UNCLASSIFIED
TABLE 1
Spinning Tests on RD.2423

<table>
<thead>
<tr>
<th>Aging</th>
<th>Test Temperature, °C</th>
<th>Final Spin Speed rev/min.</th>
<th>Mean Biaxial Strain, per cent at:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cracking</td>
</tr>
<tr>
<td>Nil</td>
<td>20</td>
<td>5000</td>
<td>27</td>
</tr>
<tr>
<td>1 day at 60°C</td>
<td>20</td>
<td>5000</td>
<td>13</td>
</tr>
<tr>
<td>3 days at 60°C</td>
<td>20</td>
<td>5000</td>
<td>19</td>
</tr>
<tr>
<td>Nil</td>
<td>-20</td>
<td>6000</td>
<td>5</td>
</tr>
<tr>
<td>1 day at 60°C</td>
<td>-20</td>
<td>6000</td>
<td>8</td>
</tr>
<tr>
<td>3 days at 60°C</td>
<td>-20</td>
<td>6000</td>
<td>4</td>
</tr>
<tr>
<td>1 day at 60°C</td>
<td>60</td>
<td>5000</td>
<td>10</td>
</tr>
</tbody>
</table>

The unaged samples were not spun until failure occurred, since in these early experiments it was assumed that even small cracks constituted failure. The criterion was changed when more experience was gained of the test, and comparisons could be made with actual motors.

The results suggest that the propellant would be satisfactory at 20°C, and probably at the lower temperature also, and after severe age hardening, failure would occur at the lower temperature.

It is well established that propellant subjected to repeated straining will fail at strain levels much lower than those for first time failure. It was therefore decided to introduce an element of fatigue into the spinning tests. Axial acceleration was used, and the acceleration direction was reversed from time to time so as to keep reforming the domes. The intention was to cycle between values of m of ± 0.3, corresponding to a strain level of about 7 per cent. Two canisters were tested; after consolidation at 1000 p.s.i. they were aged for 18 hours at 60°C and then spun in the centrifuge bucket at 400 rev/min. (thus subjecting them to 45 g ) for long enough to reach this strain level.

The results for the two canisters were very similar, and the mean results are given in Table 2.
### TABLE 2

Fatigue Spinning Tests on RD.2423

<table>
<thead>
<tr>
<th>Run</th>
<th>Spinning Time, minutes</th>
<th>Biaxial Strain, per cent</th>
<th>Cracks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Convex End</td>
<td>Concave End</td>
</tr>
<tr>
<td>1A</td>
<td>5</td>
<td>3(\frac{1}{2})</td>
<td>5</td>
</tr>
<tr>
<td>1B</td>
<td>3</td>
<td>6</td>
<td>4(\frac{1}{2})</td>
</tr>
<tr>
<td>2A</td>
<td>3</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>2B</td>
<td>2</td>
<td>9</td>
<td>7</td>
</tr>
<tr>
<td>3A</td>
<td>1(\frac{1}{2})</td>
<td>5(\frac{1}{2})</td>
<td>4</td>
</tr>
<tr>
<td>3B</td>
<td>1(\frac{1}{2})</td>
<td>9</td>
<td>6(\frac{1}{2})</td>
</tr>
<tr>
<td>4A</td>
<td>1</td>
<td>4(\frac{1}{2})</td>
<td>4</td>
</tr>
<tr>
<td>4B</td>
<td>1</td>
<td>7(\frac{1}{2})</td>
<td>6</td>
</tr>
<tr>
<td>5A</td>
<td>3(\frac{1}{4})</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>5B</td>
<td>3(\frac{1}{4})</td>
<td>7(\frac{1}{2})</td>
<td>6</td>
</tr>
</tbody>
</table>

In the A runs, the end at which the consolidation pressure had been applied was uppermost on spinning, and became concave. In the B runs the canisters were the other way up.

The strain levels did not differ significantly between ends in any one experiment. In every case, cracks occurred only at the convex end. The cracks sealed completely when the acceleration direction was reversed. It was not possible to work at a higher strain level, and so cause cracks on the concave surface, because of shear failure at the wall. Previous experiments indicate that behaviour on the concave end on cracking would be the same as on the convex end, only at a higher dome size. Although the cracks seal up on each cycle, they grow bigger on the next cycle. A strain level of about 7 per cent appears to be safe for a concave surface.

The spinning time required to produce a given strain level falls steadily throughout the experiment. This is due to the anisotropy induced by shear hardening. This is an important aspect of plastic propellant fatigue, and is being investigated separately (11). In effect, when a
sample of propellant is strained repeatedly in a given way, resistance to
deformation falls considerably at the second and subsequent deformations,
even if the propellant has been strained in some other direction meanwhile.
The effect appears to occur when the tensile or shear stress in a given
direction exceeds the bond strength between ammonium perchlorate and the
binder; it applies only to the direction in which the stress is applied and
is not reversible.

It was concluded that there is a fatigue threshold for biaxial strain-
ing, above which the cracks seal up as the strain is removed, but reappear
and open wider during the next half cycle. The strain level for this
threshold seems to be about 6 or 7 per cent, which is similar to that
reached in the Bantam motor on temperature cycling over a 25°C range, and is
only about a quarter the strain level at which cracking occurs on first
time straining.

An alternative propellant, E.3898, was formulated for the Bantam motor
containing one per cent titanium dioxide instead of two per cent copper
chromate. Although this might be expected to have a greater extensibility,
spin testing showed no significant improvement (in fact, extensibilities
both before and after age hardening were slightly lower than for RD.2423)
and the development of E.3898 was discontinued.

It was then decided by R.P.E. to press a layer of slower burning pro-
pellant on the initial burning surface in order to lower the initial
pressure, and hence the rate at which strains are imposed. Several pro-
pellants were assessed at E.R.D.E. as candidates for this purpose, or as
alternatives for the main propellant. Spin testing results on these pro-
pellants included the following:
### TABLE 3
Spin Testing on Candidate Bantam Propellants

<table>
<thead>
<tr>
<th>Propellant</th>
<th>Unaged Biaxial Strain, per cent, for:</th>
<th>Aged Samples Biaxial Strain, per cent, for:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cracking</td>
<td>Failure</td>
</tr>
<tr>
<td>E.3908</td>
<td>&gt;30</td>
<td>&gt;50</td>
</tr>
<tr>
<td>E.3912</td>
<td>8</td>
<td>28</td>
</tr>
<tr>
<td>E.3913</td>
<td>13</td>
<td>&gt;30</td>
</tr>
<tr>
<td>E.3914</td>
<td>9</td>
<td>23</td>
</tr>
<tr>
<td>E.3915</td>
<td>9</td>
<td>24</td>
</tr>
<tr>
<td>E.3916</td>
<td>9</td>
<td>32</td>
</tr>
<tr>
<td>E.3919</td>
<td>8</td>
<td>25</td>
</tr>
</tbody>
</table>

As a result of these and other tests, propellant E.3913 was selected for the initial layer, and designated RD.2424.

6. SPIN TESTING AND CONSOLIDATION

In early experiments, the test motors were consolidated by the standard method used for preparing small test pieces of propellant. A kneaded plug of propellant of approximately the required shape was dropped into a mould, and pressed into final shape with a ram, or drift, under a pressure of 1000 p.s.i. Frequently there was scatter in the results of centrifuge tests; for example, in longitudinal acceleration experiments there would be a difference in the behaviour of two motors spun in opposite buckets, even though the test samples were identical and inevitably subjected to exactly the same acceleration-time profile. A major reason for this was eventually found to be due to an asymmetry in the test sample, the end where the drift was applied being softer than the other end. The differences arose when one canister happened to be spun drift-end upwards, and the other drift-end downwards. Similarly, when a motor was spun about its own axis, the drift-end invariably deformed more. When a floating base plate was used in the consolidation process, so that in effect there was a drift at both ends, the trouble was cured.

/The
The problems associated with degree of consolidation, and the relation between consolidation and rheological behaviour have been extensively studied, and will be reported separately. The ratio of the biaxial strains at the two ends of a spun charge formed by application of a consolidation pressure from one end only may be several fold for a motor with a length/diameter ratio greater than one. Uniform behaviour is obtained if the L/D ratio is less than 0.5. However, the effect is not merely one of L/D ratio; increasing the size of the system improves the uniformity of consolidation for a given L/D ratio. Increasing the consolidation pressure from 1000 to 3000 p.s.i., or the time of consolidation from 30 seconds to 10 minutes has no effect. Variations in consolidation tend to affect yield stress rather than plastic viscosity, and hence have their greatest effect on flow behaviour when the stress levels are small. They affect biaxial behaviour more strongly than uniaxial behaviour, and are considered to be associated with solid-binder interactions. They must be allowed for in reporting and recording biaxial test results. Conversely, the biaxial technique gives most information about the factors which affect the consolidation of the propellant.

7. THE DIAPHRAGM TEST

In the diaphragm test, a microtomed membrane of propellant is expanded by pneumatic pressure until failure occurs. The test procedures are essentially the same as those described by Rainbird and Vernon (3), but modified where necessary because of the particular properties of this type of propellant.

A 2-inch cube of propellant is stuck to a base plate with clear Bostik, and mounted on a MSE flat bed microtome. Satisfactory slices can be cut with thicknesses ranging from 0.007 to 0.035 inches; a thickness of 0.013 or 0.020 inches is most suitable for the test. The blade is set to give an oblique cut, and the cutting angle is determined by experience. The softer the propellant, the faster the stroke required to give satisfactory slices. The first 20 or so slices are rejected; this removes any surface skin on the block of propellant, and makes the cut surface sufficiently flat to give undamaged slices. The cut slices are now removed from the blade with a camel hair brush, examined individually for faults, placed between sheets of paper and stored in a desiccator over silica gel.

The test system is shown in Figures 3 and 4. The propellant section, 1½ inches in diameter, is mounted in a cell, and a clamping ring is fixed above it. The inner edge of the clamping ring is radiused so that failure occurs in the centre of the diaphragm and not at the edge; the required radius was found in a series of preliminary experiments. Compressed air is introduced at the required rate and the expansion of the diaphragm is observed under a low power microscope. The thickness of the sample is such as to make it translucent, so that the behaviour of the filler-binder bond can be observed, but thick enough for there to be a layer of filler particles

/undisturbed .......

- 13 -

UNCLASSIFIED
undisturbed by the cutting process. Tests over a four-fold range of thickness gave identical results, indicating they are representative of the behaviour of the material as a whole. The extensibility is calculated from equation \( \text{2} \). Provision is made, as described in reference (3) for controlling humidity, temperature and strain rate.

The biaxial extensibility of plastic propellant varies with time after cutting; this effect is not observed with other types of propellant. For example, a batch of B-3838 was tested and found to fail at 14 per cent extension immediately after cutting, 20 per cent four hours later, 30 per cent next day, and 30 per cent after six weeks storage. The effect is not one of age hardening (which would reduce the extensibility); overnight storage at 50°C made no difference to the results. Newly cut samples have a rough surface, which becomes glossy, and sticky, after a few hours as binder migrates to the surface; the cutting process is apparently rather drastic for this material, which is more sensitive than other propellants to surface conditions. The final value is accepted as the "true" value, and membrane tests are normally carried out on the day after the sections are cut.

Extensibility varies with the strain rate, to a greater extent than is observed with other types of propellant. A sample of RD.2423 Bantam propellant had an extensibility of 48 per cent in a slow test, taking four minutes, and 60 per cent in a test which took 10 seconds.

Attempts were made to compare biaxial and uniaxial extensibilities. The main problem here is to determine which uniaxial extensibility to use in the comparison. Many propellants have two well defined points on the stress/strain curve, which can be quoted as the extension at maximum load, and the extension at break. At low rates, plastic propellants give a stress/strain curve with no sharp yield and a broad maximum. Uniaxial tests were therefore carried out in which the extension was noted when cracks began to appear on the test piece surface. This criterion for failure is similar to that used for the biaxial observations. A correction was made for gauge length effects; it was assumed that true extensibility was equal to 60 per cent of nominal extensibility, since the true gauge length has been found to exceed the nominal value by this factor.

The results included the following:

/Table ******

- 14 -

UNCLASSIFIED
## Extensibility

<table>
<thead>
<tr>
<th>Propellant</th>
<th>Biaxial to tear</th>
<th>Uniaxial to crack</th>
<th>Uniaxial at break</th>
</tr>
</thead>
<tbody>
<tr>
<td>E.3833</td>
<td>25</td>
<td>45</td>
<td>60</td>
</tr>
<tr>
<td>S.1007</td>
<td>25</td>
<td>50</td>
<td>120</td>
</tr>
<tr>
<td>S.1031</td>
<td>48</td>
<td>80</td>
<td>150</td>
</tr>
</tbody>
</table>

The values for uniaxial extension to crack are approximate since the cracks do not appear suddenly; they are first observed at about the peak in the stress/strain curve. It thus appears that, as with other propellants, the biaxial extensibility is approximately half the uniaxial value. This correlation is not obtained if the uniaxial extension at break is used in the comparison.

The biaxial extensibility falls at low temperatures (the values for polyurethane and cast double-base propellants are almost independent of temperature). Thus RD.2428 has a biaxial extensibility of 25 per cent at 20°C and 12 per cent at -2°C. RD.2421 has values of 25 per cent at 20°C and 8 per cent at -2°C.

For radial burning motors, the strains in the critical region are biaxially tensile, but not equal. At the conduit, the hoop strain is several times greater than the longitudinal strain. Accordingly, tests were carried out on elliptical diaphragms giving strain ratios of up to 10:1. For other types of propellant, such as polyurethane propellant, it was observed that the total biaxial extensibility was a constant for different ellipses (3). Tests were made on plastic propellant RD.2423 with the following results:

<table>
<thead>
<tr>
<th>Ratio Major/Minor Axis</th>
<th>Failure Strain, per cent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>on major axis</td>
</tr>
<tr>
<td>1</td>
<td>13</td>
</tr>
<tr>
<td>1.5</td>
<td>9</td>
</tr>
<tr>
<td>2.0</td>
<td>8</td>
</tr>
<tr>
<td>3.0</td>
<td>6</td>
</tr>
</tbody>
</table>

/Plastic
Plastic propellant differs, therefore, from the other types of propellant investigated in that the total is not constant, but increases with eccentricity. This implies that plastic propellant has a different failure criterion from other propellants, and is more sensitive to biaxiality.

8. THE CRACK TEST

For many years the failure behaviour of plastic propellants has been assessed by a simple test known as the crack test, which gives a measure of the cohesive strength of the propellant. A cylinder 2 cm. high and 1.5 cm. diameter is prepared by the usual moulding technique, and is then compressed axially until failure occurs (1). For many propellants this is a straightforward uniaxial compression to break test. The failure pattern is similar to that for many solid materials subjected to a crushing test, with fracture cones extending from the centre of the sample to each of the flat surfaces.

With softer propellants however, such failure may not occur, owing to the extreme ductility of the propellant, and the sample may compress to a thin disc without internal failure. The only failure observed will then be a surface cracking on the equator of the sample. Such failure can, under certain circumstances, be regarded as biaxial rather than uniaxial.

Plastic propellant does not hold a stress for any length of time; if it is strained, the stress completely decays in a second or so (12) and the failure is more closely related to strain than stress. Thus if strain levels are reproduced in different tests, the failure criteria will be comparable between such tests. This is why, for example, it is held that spinning tests can effectively reproduce strain behaviour due to thermal contraction in a case-bonded charge. The stress patterns in an elastic propellant would be different, but for a plastic propellant in the time-scale relevant to thermal straining the stresses would have fully decayed.

All strain systems are in a sense multiaxial. A tensile strain in one direction must be balanced by a compressive strain at right angles, otherwise the volume will change. If the extension ratios in three mutually orthogonal directions are \( \lambda_1, \lambda_2, \) and \( \lambda_3 \) (\( \lambda \) is greater than unity for tensile strains) then, if the volume does not change, \( \lambda_1\lambda_2\lambda_3 = 1. \)

In the uniaxial test \( \lambda_1 \) represents the imposed strain; \( \lambda_2 \) and \( \lambda_3 \) are compressive and balance this. In a biaxial test, as conventionally understood, two tensile strains at right angles are imposed; the remaining strain will, of course, be compressive. In the crack test there are, at places on the surface, positions where the two strains are tensile. Locally, therefore, a biaxial tensile state exists.
The geometry of the deformation in the crack test may be analysed as follows:

On compression, the test piece may, or may not, barrel out to some extent, depending on such things as the friction at the plates. Extreme cases are

(a) if the surfaces are fully lubricated, the test piece remains cylindrical,

(b) if there is considerable friction, the test piece barrels out, and the end radii do not increase.

Case (a). Initially the test piece is a cylinder of height $h_0$ and radius $r$; after compression its height is $h$, and its radius $r + x$.

Since the volume does not change,

$$\pi r^2 h_0 = \pi (r + x)^2 h$$

$$\therefore \frac{h}{h_0} = \frac{1}{(1 + x/r)^2}$$

The plasticity, $P$, is given by

$$h = h_0 / (1 - 0.01P)$$

$$\therefore (1 + x/r)^2 = 1/(1 - 0.01P)$$

Case (b). After compression, the test piece is barrel shaped. We assume that the minimum radius is $r$, as before, and the maximum radius is $(r + x)$, and the profile is circular.

The original volume = $\pi r^2 h_0$

The volume of the barrel = $\frac{1}{3} \pi h [2(r + x)^2 + r^2]$  

and these are equal.

Thus $h_0 = \frac{1}{3} h [2(1 + x/r)^2 + 1]$  

As before, $h = h_0 (1 - 0.01P)$

Equations 10 and 7 give the predicted relation between $x/r$ and $P$. 

/The
The relation between x/r and plasticity was investigated for propellant E-3898, using standard plasticity test practice, and measuring the diameter at intervals. The curve obtained, together with the two theoretical curves, is plotted in Figure 5. Clearly, the test piece remains very nearly cylindrical.

We now consider the strains at the "equator". Let λ₁ be the circumferential extension ratio and λ₂ be the radial extension ratio. Both are more than 1, so the strains are tensile.

\[ \lambda_1 = \frac{2\pi(r + x)}{2\pi r} = 1 + \frac{x}{r} \]

\[ \lambda_2 = \frac{r + x}{r} = 1 + \frac{x}{r} \]

Thus the two strains are equal: \( \epsilon_1 = \epsilon_2 = \frac{x}{r} \), and the strain is related to plasticity (or crack value) by equation 8 in the form:

\[ (1 + \epsilon)^2 = \frac{1}{(1 - 0.01F)} \]

Figure 6 shows the relation between crack value, biaxial strain and m.

The crack test is in some ways more convenient than the spinning or diaphragm test, although it, too, suffers from the drawback that with extensible propellants the onset of crazing is often difficult to observe. Equipment was set up so that it could be carried out on a Hounsfield Tensometer, at controlled temperatures and speeds. Figure 7 shows the effect of temperature on the crack value of E-3838/T1, an early, hard batch of RD.2423. The scatter is largely due to the difficulty of deciding when the first crack has occurred. The crack value is high above 40°C, and not very dependent on temperature in the range 0°C to -40°C. The effect of strain rate was also studied, this time on a new, softer batch, WMA 6, and the results are shown in Figure 8. The crack value appears to increase by about five units for each decade of strain rate. Cracking times ranged from 8 seconds to 9 minutes. A comparison may now be made between the biaxial extensibilities determined by three different methods.
<table>
<thead>
<tr>
<th>Time to Fail</th>
<th>Crack Value</th>
<th>Biaxial Extensibility, per cent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>calculated from crack value</td>
<td>from spinning test</td>
</tr>
<tr>
<td>10 seconds</td>
<td>60</td>
<td>59</td>
</tr>
<tr>
<td>4 minutes</td>
<td>52</td>
<td>43</td>
</tr>
</tbody>
</table>

It is very satisfactory that three different techniques, used by three different operators, have given results in such good accord. The crack test may be considered to be of particular value in the routine assessment of propellants for applications where biaxial conditions are of importance.

The 25°C thermal cycling requirement of the Bantam motor, a biaxial strain of 7 per cent, thus requires a crack value of 12. The pressurisation strain is 12 per cent, equivalent to a crack value of 20. The combination represents a strain of 28 per cent, or a crack value of approximately 41. Such values have to be measured at the appropriate temperatures and time scales. According to Figure 8, the biaxial extensibility increases with rate. It may, therefore, seem surprising that the practical solution to the Bantam pressurisation problem is to lower the strain rate on ignition. A possible reason for the success of the two-layer propellant system is the better biaxial extensibility of the R/D.2424. Another possibility for an actual charge behaving rather better than expected from laboratory tests is that the surface layer of propellant is burning away, and may burn faster than the cracks can form and spread. Again it could be that the extensibility may reach a maximum at a certain rate, and fall at higher rates. The uniaxial extensibility is a maximum for a breaking time somewhat less than one second, and falls at shorter breaking times. It has not so far been possible to develop a high speed biaxial test.

9. CONCLUSIONS

Biaxial test systems have been successfully devised for plastic propellants, and test results are applicable to rocket motor problems. Uniaxial extensibilities of plastic propellants are usually much higher than strain levels met with in rocket motors. Values based on biaxial test results, however, are more realistic, and may be used directly in motor calculations; this is perhaps the first time this has been possible for plastic propellant.
Future work on biaxial behaviour will concern a study of the effects of composition, so that propellants can be developed for special applications, without too great a penalty in rheological properties, and the provision of numerical results as a basis for the development of a failure theory for plastic propellant. Meanwhile, there is a requirement for a high speed biaxial test, giving failure in a time similar to the pressurisation time of a motor (say 0.03 second), and for a biaxial adhesion test. Work on these will be reported in due course.

10. ACKNOWLEDGEMENTS

The crack tests were carried out by Mr. D.E. England. The membrane tests were by Mr. England and Mr. R.W. Rainbird. The authors acknowledge valuable discussions with Mr. P.R. Freeman and Mr. G.J. Spickernell of E.R.D.E., and Dr. J. Rolfe of R.F.E.

11. REFERENCES


APPENDIX
APPENDIX

The compositions of propellants referred to in this report include the following:

<table>
<thead>
<tr>
<th>Composition</th>
<th>NH$_4$ClO$_4$</th>
<th>NH$_4$ Picrate</th>
<th>Al</th>
<th>SiO$_2$</th>
<th>Cu Chromate</th>
<th>S101</th>
<th>B146</th>
</tr>
</thead>
<tbody>
<tr>
<td>RD.2421</td>
<td>64</td>
<td>10</td>
<td>15</td>
<td></td>
<td></td>
<td>1</td>
<td>10</td>
</tr>
<tr>
<td>RD.2423</td>
<td>84.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>12.5</td>
</tr>
<tr>
<td>RD.2424</td>
<td>85</td>
<td></td>
<td></td>
<td>0.5</td>
<td></td>
<td>1</td>
<td>13.5</td>
</tr>
<tr>
<td>RD.2428</td>
<td>74</td>
<td></td>
<td>14</td>
<td></td>
<td></td>
<td>1</td>
<td>10</td>
</tr>
</tbody>
</table>

The compositions of other, experimental, propellants referred to are classified Restricted. They can be obtained, if required, from the Director, E.R.D.E.
The relation between \( m \) and biaxial strain.

**FIG. 1.**
THE RELATION BETWEEN TEMPERATURE DROP & STRAIN LEVEL FOR A CASE-BONDED CIGARETTE-BURNING CHARGE.

FIG. 2
THE DIAPHRAGM TEST

FIG. 3.
THE INCREASE IN DIAMETER OF A COMPRESSED CYLINDER

**FIG. 5.**

UNCLASSIFIED
THE RELATION BETWEEN $m$, BIAXIAL STRAIN AND CRACK VALUE.

FIG. 6
EFFECT OF TEMPERATURE ON CRACK VALUE

FIG. 7.
No. 7/R/65   (Mrs.) S. Cooke and J.H.C. Vernon  August, 1965

In a case-bonded rocket motor the propellant is often subjected to biaxial
tensile straining, arising from thermal or pressurisation effects. The
behaviour of propellants under biaxial straining differs from that under uni-
axial straining, and special biaxial test techniques have to be devised.
Plastic propellants have some biaxial characteristics in common with other
types of propellant, and others which are peculiar to this type of propellant.
An account is given of the way in which biaxial strains arise in certain types
of plastic propellant motor, and of the test techniques which have been
developed or modified to suit the characteristics of the material. They
include spin testing, diaphragm testing and a development of the standard
crack test. The effects of time, temperature, age hardening and asymmetry
on biaxial properties have been investigated. An account is given of the
application of biaxial test results to design problems of the Bantam motor for
the Skua meteorological rocket, and to the development of propellants for this
application.

21 pp., 8 fig., 6 tables.

21 pp., 8 fig., 6 tables.
Defense Technical Information Center (DTIC)
8725 John J. Kingman Road, Suite 0944
Fort Belvoir, VA  22060-6218
U.S.A.

AD#:  AD468449

Date of Search: 8 December 2008

Record Summary: DSIR 23/32973
  Title: Biaxial tensile properties of plastic propellant (ERDE Rep 7/R/65)
  Availability Open Document, Open Description, Normal Closure before FOI Act: 30 years
  Former reference (Department) ARC 27136
  Held by The National Archives, Kew

This document is now available at the National Archives, Kew, Surrey, United Kingdom.

DTIC has checked the National Archives Catalogue website (http://www.nationalarchives.gov.uk) and found the document is available and releasable to the public.

Access to UK public records is governed by statute, namely the Public Records Act, 1958, and the Public Records Act, 1967. The document has been released under the 30 year rule. (The vast majority of records selected for permanent preservation are made available to the public when they are 30 years old. This is commonly referred to as the 30 year rule and was established by the Public Records Act of 1967).

This document may be treated as **UNLIMITED**.