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EXPLOSIVES RESEARCH AND DEVELOPMENT ESTABLISHMENT

TECHNICAL MEMORANDUM No. 9/M/65

Plastic Propellant - The Effect of Composition on Slump Resistance : Part 1 : Summary of Work to June, 1964 [C]

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WALTHAM ABBEY
ESSEX

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(10) J. Scrivener and G.J. Spickernell,

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1. SUMMARY

This memorandum summarises existing information concerning the effect of composition on the tendency of large charges of plastic propellant to deform, or slump under their own weight.

Studies of the current type of formulation have indicated that further improvements in slump resistance are possible as the result of changes in solids content, particle size, or binder viscosity. Brief investigations of the use of polymerisable polyester plasticisers and a modified "plastisol" process have given encouraging results. These and other means of modifying the binder will be further investigated.

Present evidence suggests that an increase in tensile strength of 100 to 200 per cent is quite feasible, but this may be accompanied by some loss of extensibility.

2. INTRODUCTION

The tendency of a plastic propellant charge to slump, under its own weight on storage or when subject to the set-back forces arising on acceleration, will increase with motor diameter. Experience has shown that requirements for motors up to about 20 inches in diameter can be met with current formulations. However, there is an interest now in the use of plastic propellant in motors up to 54 inches in diameter, and this will undoubtedly require the introduction of mechanical supporting devices or compositions with increased strength and slump resistance.

The development of improved compositions is proceeding along two main lines. One approach is to investigate the effect of compositional changes on the physical properties of the current type of propellant, which is based on a high viscosity polyisobutene binder. The second approach is to consider binder modifications which might be introduced to give improved strength, without detracting significantly from the advantages afforded by plastic propellant such as relative ease of manufacture, rework capability and high specific impulse.

In the past, various investigations, many not previously reported, have yielded information which is relevant to a study of the effect of composition on slump resistance. This first report brings together the data from these earlier studies.

/3. .....

3. PLASTIC PROPELLANT COMPOSITIONS OF THE CONVENTIONAL TYPE

3.1 Factors Affecting Physical Properties

In addition to the chemical composition and extent of particle breakdown and packing, two other factors have been shown to have a marked influence on propellant physical properties. These are the time and conditions of subsequent storage, and the degree of consolidation of the propellant (see, for example, references 1 and 2 for a fuller discussion). Thus, propellants made under specific mixing conditions must also be subjected to carefully controlled subsequent handling and test-sample preparation if reproducible and comparable results are to be obtained. Failure to allow for the effect of one or more of these factors probably accounts for a large proportion of the inconsistency and irreproducibility observed in the past.

Aging tends to improve slump resistance, but makes processing more difficult. Avoidance of the complications introduced by aging, by measurement of propellant properties as soon as possible after mixing, de-aeration, and pressing, would seem to be the best approach during early assessment of the effect of composition on slump resistance. It must be borne in mind however that, because aging is also composition dependent, there could be some change in the relative merits of different compositions on aging.

3.2 Reported Work

The effect of propellant composition on the fundamental flow parameters, yield point and coefficient of plastoviscosity, has been studied (3) using the Ward Plastometer. No details of compositions were given but some general trends were outlined.

In an examination of the effect of binder content, three polyisobutene concentrations were used, 10, 12.5 and 15 per cent. The composition containing 10 per cent of binder showed a marked increase in yield value with increasing shear, whereas with the larger amounts of binder this type of shear-hardening was small. With all three compositions there was a rapid increase in plastoviscosity with increasing shear. Contrary to what would be expected, the composition containing 15 per cent of binder had a greater plastoviscosity at all amounts of shear than the composition containing 12.5 per cent of binder. Increasing the viscosity of the binder from 5×10^5 to 10^6 poise, gave higher plastoviscosities at all shear values. With the higher viscosity binder, the yield value increased more rapidly with shear, but the initial yield values for each composition were similar. Replacement of lecithin by synthetic surface-active agents (S101 type) lowered the plastoviscosity other than at very small amounts of shear and raised the yield point. Replacement of ammonium perchlorate by ammonium picrate increased both the plastoviscosity and the yield value.

/Peers .....

Peers has investigated (4) the effect of certain compositional variables on the rheological properties of S101-containing propellants. The usual routine compression tests were used (see Appendix) giving so-called plasticity, crack and flow values. The variables were (i) the proportion of picrate - no consistent trend is apparent, (ii) binder viscosity (0.5×10^6 to 5×10^6 poise) - low plasticity values were obtained after 2 hours mixing using PIB of viscosity 5×10^6 poise, suggesting that this binder would not be suitable for larger scale processing. However, results after 3 hours mixing suggest that the use of high-viscosity material could give a gain in slump resistance with acceptable processing, (iii) the proportion of binder - again the results were rather variable, but it seems probable that the binder content could be reduced below 10 per cent, the lowest concentration investigated.

3.3 Slump Test Vessel

This holds a charge 3 feet in diameter and 1 foot in depth, with a cylindrical conduit of diameter 8 inches. The charge is bonded only at the walls. Measurements are made of the slump occurring at the top of the conduit when the filled test vessel is stored vertically. A storage temperature of $32^{\circ}C$ is used.

The first composition examined was the lecithin - containing E3611 (40 per cent NH_4ClO_4, 41.5 per cent NH_4Pic, 5 per cent Oxamide, 1 per cent Lecithin, 12.5 per cent PIB). This fell out of the motor completely in 6 hours. In a 2 feet diameter vessel (other dimensions the same) the amount of slump was 0.13 inch in 5 weeks.

Compositions containing S101 and a higher solids loading have shown better performance (Table 1, p.4). Only the slump after storage for 3 weeks is quoted. Initially, slumping occurs relatively rapidly and then the rate decreases until after 3 weeks changes are occurring only very slowly.

A series of propellants was next tried consisting of E3668, E3756 and E3849 (compositions are given in Table 1) in which the binder content was increased from 10 to 11 per cent. This gave the surprising result that the propellant with the least binder slumped most; furthermore, repetition of the experiment with E3756 indicated reasonable reproducibility. Thus, further investigation is necessary. Some other results were obtained with the aluminised propellants E3833 (only 0.3 inch slump) and RD2422.

Further trials were carried out with the slump vessel on its side. Horizontal slumping was followed by measuring the fall at the lowest point of the conduit relative to the case. E3756 slumped 0.4 inch after 9 days, E3849 slumped 0.5 inch after 12 days.

These results are examined further in reference (5).

/TABLE 1 .....

TABLE 1

Composition Number	Composition, wt. %						Solids Loading		Amount of slump after 3 weeks, inches	Remarks
	NH_4ClO_4	NH_4Pic	Al	TiO_2	S101	B146	wt. %	vol. %		
E3668	43	45	-	1	1	10	89	80.3	1.8	First propellant filled into Westcott 3 ft motor. Required r_b was 0.14 inch/sec at 500 p.s.i. The solids volume loading was made as high as possible, just on limits of processibility.
E3756	43	44.5	-	1	1	10.5	88.5	79.5	1.1, 1.2 (2 expts.)	
E3849	43	44	-	1	1	11	88	78.7	1.1	
E3833	51	28	10	-	1	10	89	79.5	0.3	Similar to S1044 (E3878), the second propellant filled into Westcott 3 ft motor. Westcott required propellant with same r_b as E3668, but with Al. It was decided to allow a lower volume loading in view of reasonable behaviour of E3668, in order to achieve better processibility.
RD2422 (E3930)	64	13	12	-	1	10	89	79.0	1.1	For higher total impulse version of Westcott 3 ft motor. Required r_b was 0.43 inch/sec at 1000 p.s.i.; maximum specific impulse was also required.

/3.4 .....

3.4 Effect of Binder Viscosity

The propellant used for this study was E3090 (89 per cent NH_4ClO_4, 1 per cent S101, 10 per cent PIB), and the PIB binders examined were Oppanol B8, B11 (the usual grade), B14, B15 and Vistanex LM-MH. The mixing time was 1 hour in a 2 kg incorporator.

The physical properties measured were plasticity, crack and flow values, apparent yield value, apparent plastoviscosity, and constant load tensile properties (see Appendix). The binder viscosities and the extracted ammonium perchlorate mean specific surfaces were also determined. The results are shown in Table 2 and Figure 1. Propellant made with the highest viscosity binder was processable (the plasticity figure was 20; most propellant specifications call for a minimum plasticity of 12) and had about twice the yield value, twice the plastoviscosity and twice the stress for a 2000 sec breaking time as propellant made with the lowest viscosity binder. Thus, an increase in binder viscosity seems practicable (of. Section 3.2).

TABLE 2

	Binder Grade		B8	B11	Vistanex LM-MH	B14	B15
Binder Properties	Dynamic Viscosity, poise	25°C	134,000	531,000	1,140,000	2,530,000	4,230,000
		60°C	11,800	43,400	90,600	175,000	397,000
	Temperature Coefficient of Viscosity		3,000	3,090	3,130	3,300	2,920
Propellant Properties	Plasticity/Crack/ Flow Values, % Compression		30/66/21	40/75/12	31/76/12	34/72/12	20/64/11
	Apparent Plastoviscosity, 10^6 poise		5.1	3.9	5.8	4.3	9.9
	Apparent Yield Value, 10^4 dynes/cm^2		7.5	8.7	8.7	10.8	14.6
	Specific Surface of Extracted NH_4ClO_4, cm^2/cm^3		4,250	5,300	5,300	5,850	5,850

/Rounce .....

Rounce and Vernon (6) determined the Bingham plastoviscosity of a series of propellants containing 12.5 per cent binder over a tenfold range of binder viscosity. They found that the Bingham plastoviscosity was proportional to the square root of the binder viscosity. This was confirmed by an analysis of the temperature coefficients of these two properties.

4. PLASTISOL - PIB BINDER

In this approach, the propellant contains a solid polymer and a compatible plasticiser in addition to the usual ingredients. Normal processing is achieved by regarding the solid polymer as part of the total solids, and by selecting a higher viscosity PIB, which with the plasticiser gives a binder having a similar viscosity to the usual grade of PIB. During a subsequent "cure" period at an elevated temperature, swelling of the solid polymer by the plasticiser occurs, to give a general increase in the viscosity or elasticity of the binder.

4.1 Polyethylene

Polyethylene was chosen initially as it is a polymer which can be plasticised by PIB. Swelling of the polymer by the usual grade of PIB, Vistac (a low molecular weight PIB of viscosity 1000 poise) or ethyl oleate at 60°C was found to be very slow and the project was abandoned.

4.2 Polyvinyl Chloride

The most efficient plasticiser for PVC in general use is di-octyl phthalate (DOP). However, DOP was found to show limited compatibility with PIB, so mixtures of DOP with either di-octyl sebacate (DOS) or ethyl oleate (EtO) were used.

The following binders were examined in composition E3813 (83 per cent NH_4ClO_4, 5 per cent PVC, 1 per cent S101, 11 per cent binder),

	B100	B11	EtO	DOS	DOP	Viscosity at 25°C, 10^6 poise
	Parts by Weight					
B201	3	3	1	-	3	4.0
B202	3	3	1	-	4	3.5
B203	3	3	1	-	5	2.75
B204	3	3	-	1	4	-
B205	3	3	-	2	4	-

/The .....

The above binders on standing all exuded a considerable amount of plasticiser.

No heat was used during the incorporation (but work-heating raised the temperature to about 60°C), de-aeration or pressing. The mixing time was 1 hour in a 2 kg incorporator. Test pieces cut from pressed sheets were stored at 100°C. After conditioning at 25°C and -25°C, these were tensile tested at a strain rate of 2 inches/inch.min.

The load/extension curves were similar in shape to those obtained with polyurethane propellants. The maximum stress, elongations at maximum stress E_1 and E_2, and permanent extension E_4 values are shown in Table 3, which includes typical figures for current plastic and polyurethane propellants. It will be noted that increases in strength were achieved only at the expense of some loss of elongation.

TABLE 3

Binder	Cure Time at 100°C, hours	Properties at 25°C				Properties at -25°C	
		Maximum Stress, p.s.i.	Elongations, %			Maximum Stress, p.s.i.	Elongations $E_1 = E_2$, %
			E_1	E_2	E_4		
B201	140	41	20	30	9	215	9
B202	140	39	19	30	16	195	10
B203	110	29	17	19	4	130	11
B204	140	29	16	17	4	140	11
B205	140	23	28	37	12	120	12
Plastic Propellant		10 - 20	Elongations at break 50 up to several hundred			100 - 200	E_1 = 15 - 30
Polyurethane Propellant (U.188)		50	35	45	0	250 (at -40°C)	E_1 = 20 (at -40°C)

The "pot life" was 2 to 3 days at ambient temperature.

/Various .....

Various grades of PVC from Messrs British Geon Ltd. were examined in composition E3813 using binder B205, to determine the effect of particle size on the physical properties of fully cured material, and the rate at which these were achieved. The results, shown in Table 4, indicate that the coarsest grade of PVC gives the best increase in propellant strength and the fastest cure time.

TABLE 4

Geon PVC	Specific Surface cm^2/cm^3	Approximate Cure Time at 100°C, hours	Maximum† Stress, p.s.i.	Elongations†, %	
				E_1	E_4
101	1,240	Between 80 and 160	34	23	6
113	995	30	40	25	6
121	52,700	Between 80 and 160	29	21	24
202*	1,205	30	38	28	10
434*	605	30	32	45	25

*PVC/VA copolymers
†Properties at 25°C

The major drawback associated with this approach to binder modification would appear to be degradation of motor performance unless the amount of solid polymer can be kept very small, or an energetic polymer, e.g. plastisol NC is used.

5. CROSSLINKING OF THE BINDER

The initial approach has been to mix trifunctional materials, capable of being crosslinked with di-isocyanates, with polyisobutene.

Mixtures of poly(diethylene/trimethylolpropane isosebacate), trimethylolpropane (TMP), tolylene di-isocyanate (TDI), and PIB have been assessed in compositions E3954 (88 per cent NH_4ClO_4, 1 per cent S101, 8.8 per cent PIB, 2.2 per cent polyester) and E3955 (88 per cent NH_4ClO_4, 1 per cent S101, 6.4 per cent PIB, 4.6 per cent polyester). The stoichiometric amount of TDI was added. Mixing was carried out without external heating and the normal de-aeration process was used. The mixing time was 1 hour in a 2 kg incorporator.

/A .....

A summary of the tensile (2 inches/inch.min) properties obtained is shown in Table 5 (p. 10). These suggest that it may well be possible to achieve a marked improvement in propellant strength, while still retaining the ability to blend for a reasonable period after mixing. Again, some loss in extensibility may be expected.

6. CONCLUSIONS

6.1 Conventional Plastic Propellant

Information available on the effect of composition on propellant mechanical properties is scanty and often contradictory; there is a need for a much more detailed investigation utilising those laboratory techniques most appropriate to a study of slump resistance, (constant load tensile, bending beam, and centrifuge tests (2)) as well as the quality control tests hitherto employed. Laboratory investigations should be backed up closely by larger scale tests, for example by experiments using the slump test vessel.

Improved reproducibility should be sought, but the major concern should be to determine whether large increases (well in excess of normal variations) can be achieved.

Studies of the effect of solids content and particle size, binder viscosity, and mixing time, beginning with the simplest system NH_4ClO_4 - S101 - PIB, are indicated.

6.2 Modified Plastic Propellant

Investigations of modified binder systems for plastic propellant should be aimed towards achieving improved strength and slump resistance without serious loss of the advantages of the present system or drastic modification of processing methods. The advantages include high specific impulse (resulting from the use of a hydrocarbon binder and a high solids loading), easy processing (no need to exclude water or pre-dry ingredients), and rework capability (propellant may be reworked, blended, or pressed at any time subsequent to manufacture).

Two general approaches are evident. One is to cause some physical change to occur which gives a general increase in the viscosity or elasticity of the binder subsequent to processing. The results in Section 4 indicate that this may be achieved using a solid polymer - plasticiser - liquid polymer (PIB) type of system. The introduction of nitrocellulose as the solid polymer could prove interesting.

The other approach is to introduce some chemical crosslinks into the binder. The problem here is that there has been little commercial interest in weakly crosslinked hydrocarbon polymers or systems which will cure at temperatures below about 60°C, apart from that linked to the U.S. castable polybutadiene propellant programmes. Attention should be given to the possibility of obtaining liquid hydrocarbon polymers with reactive sites for crosslinking, and low-temperature curing agents.

/TABLE 5 .....

TABLE 5

Composition No.	Batch No.	TMP, +%	TDI, +%	Approximate Time to Full Cure at 60°C, hours	Properties at 25°C after full cure					Properties at -25°C* after full cure					Properties at 25°C after 100 hours at 25°C				
					S p.s.i.	E_1 %	E_2 %	E_3 %	E_4 %	S p.s.i.	E_1 %	E_2 %	E_3 %	E_4 %	S p.s.i.	E_1 %	E_2 %	E_3 %	E_4 %
E3954	1	0.55	1.625	20	66	16	-	45	22	605	13	13	13	0	48	23	-	88	53
	2	0.365	0.93	30	25	21	-	170	125	465	17	17	22	0	23	25	-	230	170
	3	0.185	0.575	30	23	24	-	150	130	520	17	20	26	5	24	39	-	175	175
	4	-	0.22	40	11	43	-	440	270	215	22	30	100	20	6	22	-	640	530
	5†	-	-	After 400 hours	8	26	-	440	385	170	18	24	140	50	5	30	-	715	660
E3955	1	0.7	1.795	20	63	29	40	53	15	645	13	13	13	0	26	48	94	105	45
	2	0.45	1.305	40	53	28	50	60	23	640	16	16	18	0	22	47	100	120	47
	3	0.2	0.825	40	36	28	42	55	18	420	15	15	24	5	14	38	99	125	53
	4	-	0.425	40	26	29	47	68	17	330	17	17	44	7	16	30	48	56	20
	5†	-		After 140 hours	25	19	24	32	5	220	12	12	49	10	-	-	-	-	-

S: Maximum Stress

E_1, E_2: Elongations at maximum stress

E_3: Elongation at break

E_4: Permanent elongation

†In each case, batch 5 showed aging characteristic of conventional plastic propellant

*E.3533 (88 per cent NH_4ClO_4, 1 per cent S101, 11 per cent PIB) has the following properties at -25°C:

S = 140 p.s.i., E_1 = 25 per cent, E_2 = 35 per cent, E_3 = 250 per cent, E_4 = 100 per cent

/An .....

An alternative method would be to mix with the PIB another system which will undergo crosslinking. There is considerable experience with polyurethane propellants at E.R.D.E., and it has already been demonstrated (Section 5) that the addition of polyesters and di-isocyanates could give worthwhile improvements. The aim should be to minimise performance losses by keeping the amount of polyester as low as possible (i.e., a highly trifunctional material is indicated) and to obtain a polyester which is miscible with PIB. Combination of the U.S. carboxy-terminated polybutadiene/imine- or epoxy-cured system with PIB or, perhaps, liquid polybutadiene would also be a worthwhile study.

Inevitably some loss of ease of processing is bound to occur if the binder is to change between mixing and its final state. However, the aim will be to retain a reasonable period for blending and processing with the addition, if necessary, of a cure stage at an elevated temperature.

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/APPENDIX: .....

APPENDIX:

Physical Property Tests

The first five tests are carried out on small cylindrical samples of propellant, 2 cm high and 1.5 cm diameter. Fuller details are given in reference 7.

1. Plasticity Value is the percentage decrease in height when a sample is subjected to a load of 1500 g for 30 sec at 25°C.

2. Crack Value is measured using the same sample as in 1. The sample is further compressed slowly, with the addition of manual pressure, until cracks begin to appear on the surface. The crack value is the percentage decrease in height at which this occurs.

3. Flow Value is the percentage decrease in height when a sample is subjected to a load of 200 g for 16 hours at 60°C.

4. Apparent Yield Value is obtained by subjecting a sample at 25°C to a load of 1500 g until no further deformation occurs. Then,

$$\text{Apparent Yield Value} = \frac{Wh}{2V} = \frac{Wh}{2A_o h_o}$$

where W = load
V = volume of sample
A_o = initial cross-sectional area of sample
h_o = initial height of sample
h = final height of sample

This apparent yield value refers to material which has been subjected to a shear of $\ln h_o/h$.

5. Apparent Plastoviscosity is obtained by measuring the decrease in height, at short intervals of time, for the early stages of compression of a sample subjected to a load of 1500 g at 25°C. A plot of compressive strain (amount of shear) $\ln h_o/h$, against time is extrapolated to zero time; the initial slope then gives the initial rate of shear, and this is divided into the shear stress $W/2A_o$ to give an initial apparent plastoviscosity.

/6. .....

6. Constant Load Tensile Test A dumb-bell specimen of 1 cm square cross-section and 3 cm gauge length is used. The stress value is the load divided by the initial cross-sectional area.

7. Constant Strain Rate Tensile Test A Hounsfield tensometer is used. The cross-head movement is 2 inches/min giving a strain rate of approx. 2 inches/inch.min. The test specimen used is the same as in 6. A typical load-extension curve shows the load rising to a maximum, and then remaining constant for a while before falling. The elongation at the point at which the maximum load is first reached is designated E_1, and that at which it begins to decrease, E_2. E_3 is the elongation at break and E_4 is the permanent elongation. The stress is the load divided by the initial cross-sectional area.

8. Temperature Coefficient of Viscosity (PIB)

This is defined as,

$$\frac{\log \eta_2 - \log \eta_1}{1/T_2 - 1/T_1}$$

for $T_1 = 333^{\circ}K$, $T_2 = 298^{\circ}K$ this becomes,

$$2838 \log(\eta_2/\eta_1)$$

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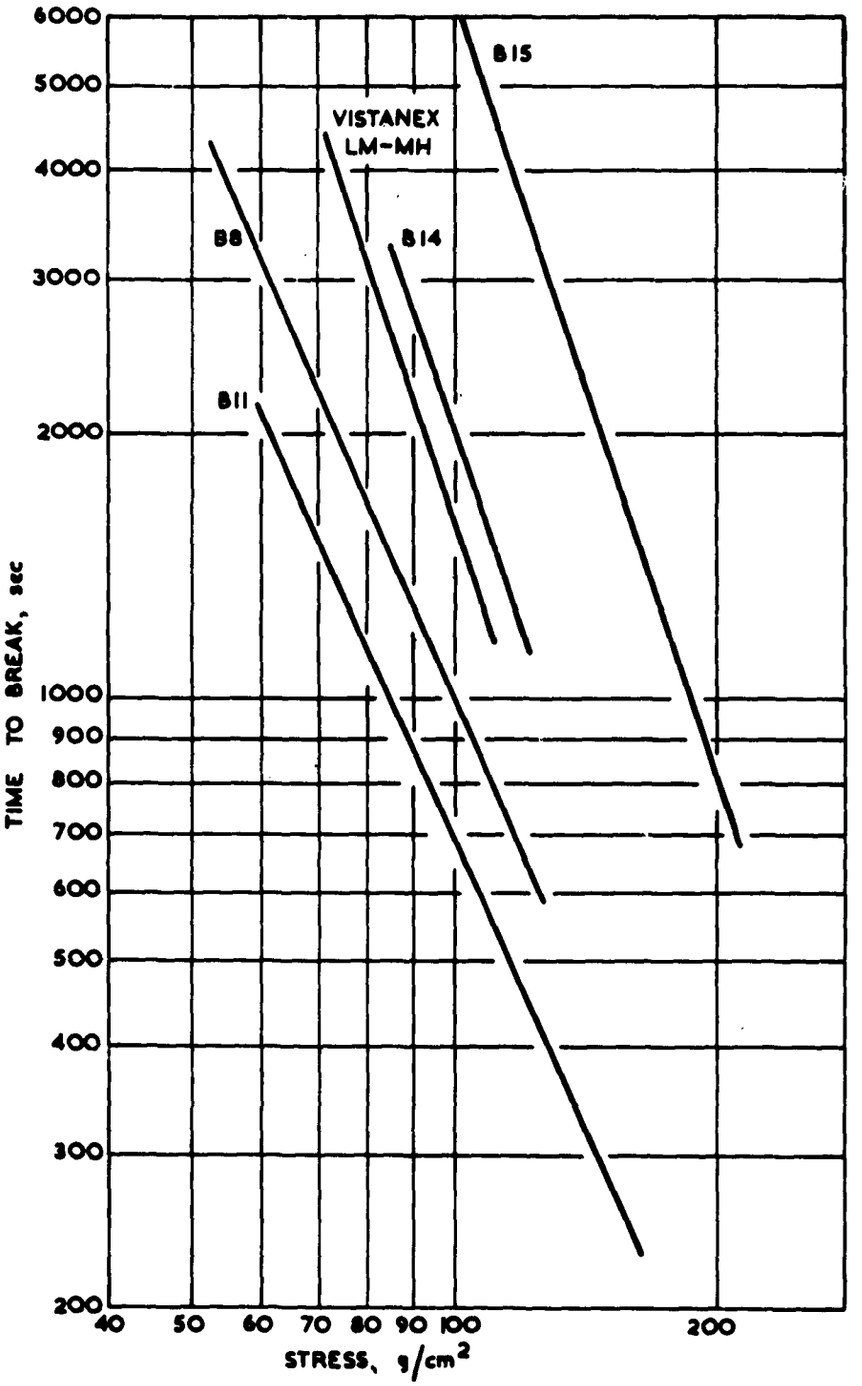

CONSTANT LOAD TENSILE TEST. FIG. 1.

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