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EFFECT OF BRANCHED GAP SYNTHESIS PARAMETERS ON MECHANICAL PROPERTIES OF ROCKET PROPELLANTS

by

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ABSTRACT

An experimental minimum-smoke low-vulnerability rocket propellant formulation was developed at DREV. This formulation contains phase stabilized ammonium nitrate (PSAN) as the oxidizer and an energetic binder based on branched glycidyl azide polymer (B-GAP). Different B-GAP polymers, obtained by varying some synthesis parameters, were incorporated into the formulation in order to study the effect on the mechanical properties of the resulting propellants. The reaction parameters investigated were the solvent and cleaving agent as well as the polyol used in the synthesis of B-GAP. The study highlighted the fact that some experimental parameters selected for the polymer synthesis have a strong influence on the mechanical properties of the propellant processed with B-GAP.

RÉSUMÉ

Une composition expérimentale de propergol pour fusée à vulnérabilité et fumée réduites a été développée au CRDV. Cette composition contient du nitrate d'ammonium stabilisé (NAS) comme oxydant et un liant énergétique à base de polyazoture de glycidyle ramifié (PAG-R). Différents polymères de PAG-R obtenus en faisant varier quelques paramètres de synthèse ont été incorporés dans la composition en vue d'étudier l'effet sur les propriétés mécaniques des propergols résultants. Les paramètres réactionnels étudiés étaient le solvant et l'agent de clivage ainsi que le polyol utilisé dans la synthèse du PAG-R. L'étude a souligné le fait que certains paramètres expérimentaux choisis pour la synthèse du polymère ont une influence marquée sur les propriétés mécaniques du propergol préparé avec le PAG-R.

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FIGURE 1

EXECUTIVE SUMMARY

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Conventional rocket propellants containing hydroxy-terminated polybutadiene (HTPB) binder and ammonium perchlorate (AP) oxidizer can generate a significant amount of secondary smoke due to the formation of hydrogen chloride. One way to reduce the smoke production is to replace AP by another oxidizer without chlorine but less energetic such as phase-stabilized ammonium nitrate (PSAN) and compensate the energy loss by using energetic polymers and plasticizers in the binder.

Following this trend, DREV has initiated the development of a rocket propellant with reduced smoke and low vulnerability based on glycidyl azide polymer (GAP) as a substitute for the inert HTPB. Recently, an improved process has been developed at DREV for the preparation of GAP with a branched structure (B-GAP). This one-step process involves the simultaneous degradation and azidation of a commercial rubber with sodium azide in the presence of a polyol and cleaving agent at elevated temperature in a polar organic solvent. GAP currently produced in the USA is relatively expensive and is prepared according to a two-step process involving two distinct chemical reactions.

Research on B-GAP has been conducted at DREV in cooperation with ICI Explosives Canada under the Defence Industrial Research (DIR) program. A preliminary study has proven the feasibility of formulating rocket propellants with B-GAP. The purpose of this study is to investigate how the variation of some B-GAP synthesis parameters (such as the polyol and solvent) could improve the mechanical properties of propellants processed with this energetic polymer.

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NOMENCLATURE

AN	ammonium nitrate
AP	ammonium perchlorate
BDNPA/F	50/50 mixture of bis (2,2-dinitropropyl) acetal and bis (2,2-dinitropropyl) formal
B-GAP	branched glycidyl azide polymer
CH_3OLi	lithium methoxide
DBTDL	dibutyl tin dilaurate
DMA	dimethyl acetamide
DMSO	dimethyl sulfoxide
E	Young's modulus
f	hydroxyl functionality
GAP	glycidyl azide polymer
HT	hexanetriol
HTPB	hydroxy-terminated polybutadiene
IPDI	isophorone diisocyanate
Me	hydroxyl equivalent weight
MW	molecular weight
M_W	weight average molecular weight
NaOH	sodium hydroxide
NAS	nitrate d'ammonium stabilisé
NCO/OH	isocyanate to hydroxyl equivalent ratio
PAG-R	polyazoture de glycidyle ramifié
PE	pentaerythritol
PECH	polyepichlorohydrin
PEG	polyethylene glycol
PSAN	phase-stabilized ammonium nitrate
TMP	trimethylol propane
ε_r	rupture strain
σ_m	maximum stress

1.0 INTRODUCTION

Composite rocket propellants traditionally developed and produced in Canada are based primarily on ammonium perchlorate (AP) dispersed in an inert hydroxy-terminated polybutadiene (HTPB R-45M) binder. Depending on the atmospheric conditions, such propellants can produce a significant amount of secondary smoke (due to the production of hydrogen chloride) which is undesirable for certain applications. One approach to avoid the production of this smoke is to replace AP in the propellant by another oxidizer containing no chlorine atoms. For this purpose, studies began in the last decade with phase-stabilized ammonium nitrate (PSAN) as a replacement for AP in rocket propellants. Unfortunately, ammonium nitrate (AN) is not as oxygen-rich or as dense as AP. Therefore, to regain the energy loss, it is necessary to replace both the inert polymer and plasticizer components in the formulation by energetic counterparts.

DREV has initiated the development of a minimum-smoke and low-vulnerability propellant (Refs. 1-2) in which the binder is based on glycidyl azide polymer (GAP), as a replacement for the inert HTPB, mixed with one or two commercially available nitroplasticizers. GAP is a hydroxy-terminated aliphatic polyether containing alkyl azide groups. This polymer can be used as an energetic binder in low-smoke solid rocket propellants, composite explosives, gun propellants and pyrotechnics to enhance the performance and stability, reduce the vulnerability and improve the physico-chemical properties of the energetic formulations.

GAP currently produced in the USA (Ref. 3) is relatively expensive and is prepared according to a two-step process involving two distinct chemical reactions (polymerization and azidation). Recently, a novel process was developed at DREV (Refs. 4-7) for the preparation in one step of GAP with a branched structure (B-GAP) and with variable and controlled molecular weight (MW). Because of the structure and the unique synthesis process of B-GAP, many advantages over other energetic binders are expected.

The research program on B-GAP has been conducted at DREV since 1985, and also at ICI Explosives Canada Technical Centre since early 1990 under the Defence Industrial Research (DIR) Program (Refs. 8-11). In order to demonstrate in a typical application the B-GAP under development, work was performed at DREV to incorporate this energetic polymer in rocket propellant formulations (Refs. 11-15). The object of this report is to describe the correlation between some synthesis parameters of B-GAP and the mechanical properties of rocket propellants processed with this polymer.

This work was performed at DREV between September 92 and May 94 under PSC 32C, Rockets and Missiles.

2.0 EXPERIMENTAL

2.1 Branched GAP Samples

The B-GAP samples used in this study are listed in Table I; the polymers were synthesized according to the improved process described in Refs. 4-7. The chemical reaction involves the simultaneous degradation and azidation of a commercial rubbery polyepichlorohydrin (PECH) with sodium azide and a basic cleaving agent in the presence of a polyol at 120°C in a polar organic solvent.

All the GAP samples were prepared in dimethyl sulfoxide (DMSO) with lithium methoxide (CH_3OLi) as cleaving agent except Sample N76 which was obtained in dimethyl acetamide (DMA) using sodium hydroxide (NaOH) for chain cleavage. All the polymers were synthesized with a reaction time of 16 h at the laboratory-scale in lots of 170 g except Sample GBP-092 which was obtained after 30 h at a lot size of 800 g for scale-up purposes. The following polyols were used in the synthesis: trimethyl propane (TMP), pentaerythritol (PE), hexanetriol (HT), glycerol and polyethylene glycol (PEG) with MW 600. The reaction was carried out with one polyol or a blend of two polyols and in one case (Sample N115) without polyol. As shown in Table I, the weight average molecular weight (M_W) of the B-GAP samples were in the range 7830-13,250 and the hydroxyl equivalent weight (M_e) varied between 1290 and 1959. The polymers had a hydroxyl functionality (f) between 2.7 and 3.7 and a viscosity of 32,000-50,000 cP at 25°C.

2.2 Rocket Propellant Formulations

The rocket propellants were processed with the B-GAP samples listed in Table I. The propellant formulation selected as the basis for this study was previously reported by Lessard *et al.* (Refs. 1-2). However, the formulation required some modifications due to the nature of the B-GAP polymer (Refs. 11-15). The selected curing agent was isophorone diisocyanate (IPDI) at a NCO/OH ratio of 1.3/1.0 in all cases and dibutyl tin dilaurate (DBTDL) was added to the mixture as a cure catalyst. The energetic plasticizer used in the formulation consisted of a 50/50 mixture of bis (2,2-dinitropropyl) acetal and bis (2,2-dinitropropyl) formal, commonly designated as BDNPA/F at a plasticizer/polymer ratio of 1.0/1.0. The 68% by weight oxidizer used in the formulation consisted of a bimodal mixture of prilled and ground zinc oxide stabilized PSAN. Two percent each of a combustion catalyst and a stabilizer (diphenylamine) were also included in the formulation. The binder level was thus maintained at 28%. No bonding agents were included.

The propellants were processed in a Helicone 2CV Mixer from Atlantic Research Corporation at a batch size of 100-170 g. The mechanical properties were

determined at room temperature using an Instron apparatus (Model 1112) at a crosshead speed of 25 mm/min and miniature dogbones 6.3 mm thick and 41.3 mm long with a cross section of 6.3 mm X 6.3 mm and an effective gauge length of 35.56 mm.

3.0 RESULTS AND DISCUSSION

All the rocket propellants were prepared and tested using the same formulation and techniques. The only variables were the NCO/OH ratio and the type of B-GAP incorporated into the formulation, in order to study the effect of some reaction parameters in the polymer synthesis on the mechanical properties of the resulting propellants.

The NCO/OH ratio varied from one formulation to another in order to optimize the mechanical properties. The effects of the NCO/OH ratio on the mechanical properties of some rocket propellants are illustrated in Fig. 1. Representative results of the mechanical properties of propellants are listed in Table II; the rupture strain (ε_r), the maximum stress (σ_m) and the modulus (E) are reported for various propellants processed with B-GAP samples synthesized with different reaction parameters. The optimum mechanical properties of the formulations were obtained at a relatively high NCO/OH ratio of 1.3-1.5.

3.1 Effects of the Reaction Solvent and Cleaving Agent

Branched GAP Samples N76 and N75, prepared respectively in the system DMA/NaOH and DMSO/CH_3OLi using TMP as polyol in both cases, were incorporated in propellants # 1 and # 2, respectively, in order to study the effects of the system (solvent/cleaving agent) used in the synthesis on the mechanical properties of the resulting propellants.

The mechanical properties of Propellants # 1 and # 2 reported in Table II indicate that σ_m is higher (0.58 MPa versus 0.39 MPa), ε_r is lower (18% versus 32%) and E is higher (6.2 MPa versus 2.3 MPa) when using the system DMSO/CH_3OLi instead of DMA/NaOH. Consequently, a B-GAP polymer synthesized in DMSO/CH_3OLi will lead to a propellant with a higher strength but a lower elongation compared with a propellant obtained with a polymer prepared in DMA/NaOH. These findings could be explained by the fact that B-GAP Sample N75 (M_e = 1290) synthesized in DMSO/CH_3OLi has a higher hydroxyl content than Sample N76 (M_e = 1925) obtained in DMA/NaOH. As reported in Ref. 6, B-GAP samples synthesized in DMSO will contain relatively more OH groups than their counterparts prepared in DMA with similar experimental conditions.

3.2 Effect of Polyol Reactant

Various B-GAP samples prepared in DMSO in similar experimental conditions but with different polyols (except Sample N115 obtained without polyol) were incorporated into propellant compositions in order to study the effect of the polyol reactant used in the polymer synthesis on the mechanical properties of the formulations. Representative results obtained for different propellants are reported in Table II.

The mechanical properties of Propellant # 2 (obtained with Sample N75 prepared with TMP) are the most promising ones: σ_m = 0.58 MPa, ε_r = 18% and E = 6.2 MPa. Although the elongation is slightly low, the stress value is however clearly superior to the stress values obtained for the other propellants. The high strength measured for Propellant # 2 could be explained by the presence of three primary OH groups in TMP used as polyol in the polymer synthesis. Propellants # 3, # 4, # 5 and # 6 processed respectively with B-GAP samples GBP-092 (using glycerol), N98 (using PE), N110 (using PEG-600) and N116 (using HT) gave similar stress values (σ_m = 0.35-0.39 MPa). However, Propellants # 3 and # 4 had a higher modulus (E = 3.6-3.7 MPa) and a lower elongation (21-23%) compared with Propellants # 5 and # 6, which had E = 1.8-2.3 MPa and ε_r = 32-36%. Although Propellant # 4 was obtained with a polymer synthesized using PE (containing four primary OH groups), it had nevertheless inferior strength than Propellant # 2 processed with a polymer synthesized using TMP (with three primary OH groups) due probably to some steric hinderance involved in the case of PE. The relatively high elongation values observed for Propellants # 5 and # 6 could be caused by the longer chain of the polyols (PEG 600 and HT) used in the synthesis of the B-GAP samples incorporated in these propellants.

Propellants # 7, # 8 and # 9 were processed with B-GAP samples that were synthesized by using a mixture of two different polyols in the reaction in an attempt to increase the strength of the propellants while hopefully keeping the elongation as high as possible. The mechanical properties of Propellant # 7 (σ_m = 0.47 MPa, ε_r = 19%, E = 4.4 MPa) processed with B-GAP sample N112 (using 60% TMP-40% PEG 600) show intermediate values between those of Propellant # 2 (with Sample N75 using TMP) and Propellant # 5 (with Sample N110 using PEG 600). Propellant # 9 (σ_m = 0.42 MPa, ε_r = 23%, E = 2.9 MPa), which was processed with B-GAP sample N107 (using 60% PE-40% HT), has also intermediate elongation and modulus values but a slightly higher strength compared with Propellant # 4 (with Sample N98 using PE) and Propellant # 6 (with Sample N116 using HT). On the other hand, Propellant # 8 (σ_m = 0.51 MPa, ε_r = 17%, E = 5.1 MPa), prepared with B-GAP sample N111 (using 60% PE-40%PEG 600), gave higher than expected strength and modulus values but a lower elongation compared with Propellant # 4 (with Sample N98 using PE) and Propellant # 5 (with Sample N110 using PEG 600).

The results of Table II indicate that the polyol reactant used for the polymer synthesis has a significant effect on the mechanical properties of the propellants processed with B-GAP. As reported in Refs. 5-7, a portion of the polyol included in the synthesis reaction of B-GAP is grafted on the polymer and depending on the type of polyol used, additional primary and/or secondary alcohols (originating from the polyol) are thus incorporated into the B-GAP molecular structure. This will have a noticeable effect on the curing reaction of B-GAP with the isocyanate and the resulting mechanical properties of the formulation.

The results of Table II show also a correlation between the hydroxyl functionality of the polymer and the strength of the propellant obtained with B-GAP. The values of σ_m measured for the propellant formulations seem to increase with the functionality of the B-GAP samples synthesized in DMSO using similar reaction parameters but with different polyols (or mixture of polyols). B-GAP samples N111 and N75 with a relatively high functionality (f = 3.4-3.7) gave propellants with the highest strength (σ_m = 0.51-0.58 MPa), whereas Samples N107 and N112 with f = 3.1-3.3 yielded propellants with a strength of 0.42-0.47 MPa. On the other hand, B-GAP sample GBP-092, N110, N116 and N98 with a relatively low functionality (f = 2.7-3.0) gave propellants having the lowest strength (σ_m = 0.35-0.39 MPa). Propellant # 10, which was processed with B-GAP sample N115 (synthesized without polyol), is an exception. Although Sample N115 had a relatively low functionality (f = 2.8), it gave nevertheless a Propellant # 10 with very interesting mechanical properties (σ_m = 0.45 MPa, ε_r = 28%, E = 3.0 MPa), which offer a good compromise between strength and elongation.

3.3 Effect of Polymer Blending

Branched GAP samples N75 (prepared with TMP) and N110 (prepared with PEG 600) were mixed respectively in a ratio 60-40 wt-% and then incorporated into Propellant # 11 in order to study the effect of polymer blending on the mechanical properties of the resulting formulation. As shown in Table II, the mechanical properties of Propellant # 11 (σ_m = 0.43 MPa, ε_r = 23%, E = 3.3 MPa) are similar to those of Propellant # 7 which was processed with Sample N112 synthesized by using a mixture of the same polyols (60% TMP / 40% PEG 600).

Consequently, a propellant obtained with a B-GAP sample prepared by including in the reaction a mixture of two different polyols will have approximately the same mechanical properties as a propellant processed with a blend of two polymers synthesized each by using singly one of the two polyol components. This is an

indication that polyols are grafted into the B-GAP molecular structure in the same proportion, whether they are incorporated singly or as a mixture of co-reactants in the synthesis.

4.0 CONCLUSIONS

Various B-GAP samples obtained by varying some reaction parameters were incorporated into rocket propellant formulations in order to study the effect of B-GAP synthesis parameters on the propellant mechanical properties. High NCO/OH ratios (1.3-1.5) were required to obtain propellants with optimum mechanical properties.

A rocket propellant processed with B-GAP synthesized in the system DMSO/CH_3OLi has a higher strength but a lower elongation compared with a propellant containing B-GAP prepared in DMA/NaOH.

The type of polyol reactant used in the B-GAP synthesis has a significant effect on the mechanical properties of the resulting propellant. The number of primary hydroxyl groups present in the polyol molecule seems to affect the maximum strength value, whereas the polyol chain length has a limited beneficial effect on the elongation. The maximum strength values of propellants processed with B-GAP seem to increase with the hydroxyl functionality of the polymers synthesized in DMSO with similar experimental conditions but using different polyol reactants.

A propellant processed with a B-GAP polymer synthesized without polyol gives very interesting mechanical properties which offer a good compromise between strength and elongation.

A propellant obtained with a B-GAP sample synthesized with a mixture of two different polyol reactants yields generally intermediate mechanical properties between those of the two propellants processed each with a GAP polymer prepared by using singly one of the two polyol components. These results tend to indicate that polyols are grafted into the B-GAP molecular structure in the same proportion whether they are included singly or as a mixture of co-reactants in the synthesis.

5.0 ACKNOWLEDGEMENTS

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TABLE I

Properties of B-GAP Samples

Sample*	Synthesis Parameters		$\left(\frac{\text{Polyol}}{\text{PECH}}\right)$	M_w	M_n	f	Viscosity (25°C) cP
	Solvent	Polyol					
N76	DMA	TMP	0.25	13,250	1925	3.5	50,000
N75	DMSO	TMP	0.25	9500	1290	3.7	42,000
GBP-092	"	glycerol	1.0	7830	1490	2.7	32,000
N98	"	PE	0.25	8950	1413	3.0	38,000
N110	"	PEG (MW 600)	0.25	11,600	1959	2.8	41,000
N116	"	HT	0.25	9360	1548	2.9	40,000
N112	"	TMP(60%)/PEG 600(40%)	0.25	10,300	1530	3.3	39,000
N111	"	PE(60%)/PEG 600(40%)	0.25	9870	1572	3.4	39,000
N107	"	PE(60%)/HT(40%)	0.25	9250	1478	3.1	37,000
N115	"	NONE	0	12,100	1660	2.8	47,000

* All B-GAP samples were prepared at 120°C

The reaction time was 16 h for all the samples except GBP-092 (obtained after 30 h)

All the samples (except N76) were synthesized with a wt ratio (CH_3OLi/PECH = 0.030)

Sample N76 was prepared with a wt ratio (NaOH/PECH = 0.045)

TABLE II

Mechanical Properties of Processed Propellants

Propellant	+ B-GAP		Polyol*	NCO/OH	Mechanical Properties		
	Sample	f			σ_m (MPa)	ε_r (%)	E (MPa)
1	N76	3.5	TMP	1.3	0.39	32	2.3
2	N75	3.7	TMP	1.4	0.58	18	6.2
3	GBP-092	2.7	glycerol	1.5	0.39	23	3.7
4	N98	3.0	PE	1.3	0.39	21	3.6
5	N110	2.8	PEG (MW 600)	1.3	0.36	32	2.3
6	N116	2.9	HT	1.5	0.35	36	1.8
7	N112	3.3	TMP(60%)/PEG 600(40%)	1.5	0.47	19	4.4
8	N111	3.4	PE(60%)/PEG 600(40%)	1.5	0.51	17	5.1
9	N107	3.1	PE(60%)/HT(40%)	1.3	0.42	23	2.9
10	N115	2.8	NONE	1.5	0.45	28	3.0
11	N75(60%)/N110(40%)	-	TMP/PEG 600	1.3	0.43	23	3.3

+ All B-GAP samples were synthesized in DMSO (except sample N76 obtained in DMA)

Propellant # 11 was processed with a blend of two polymers: sample N75 (prepared with TMP) and sample N110 (prepared with PEG-600)

* Type of polyol used in B-GAP synthesis

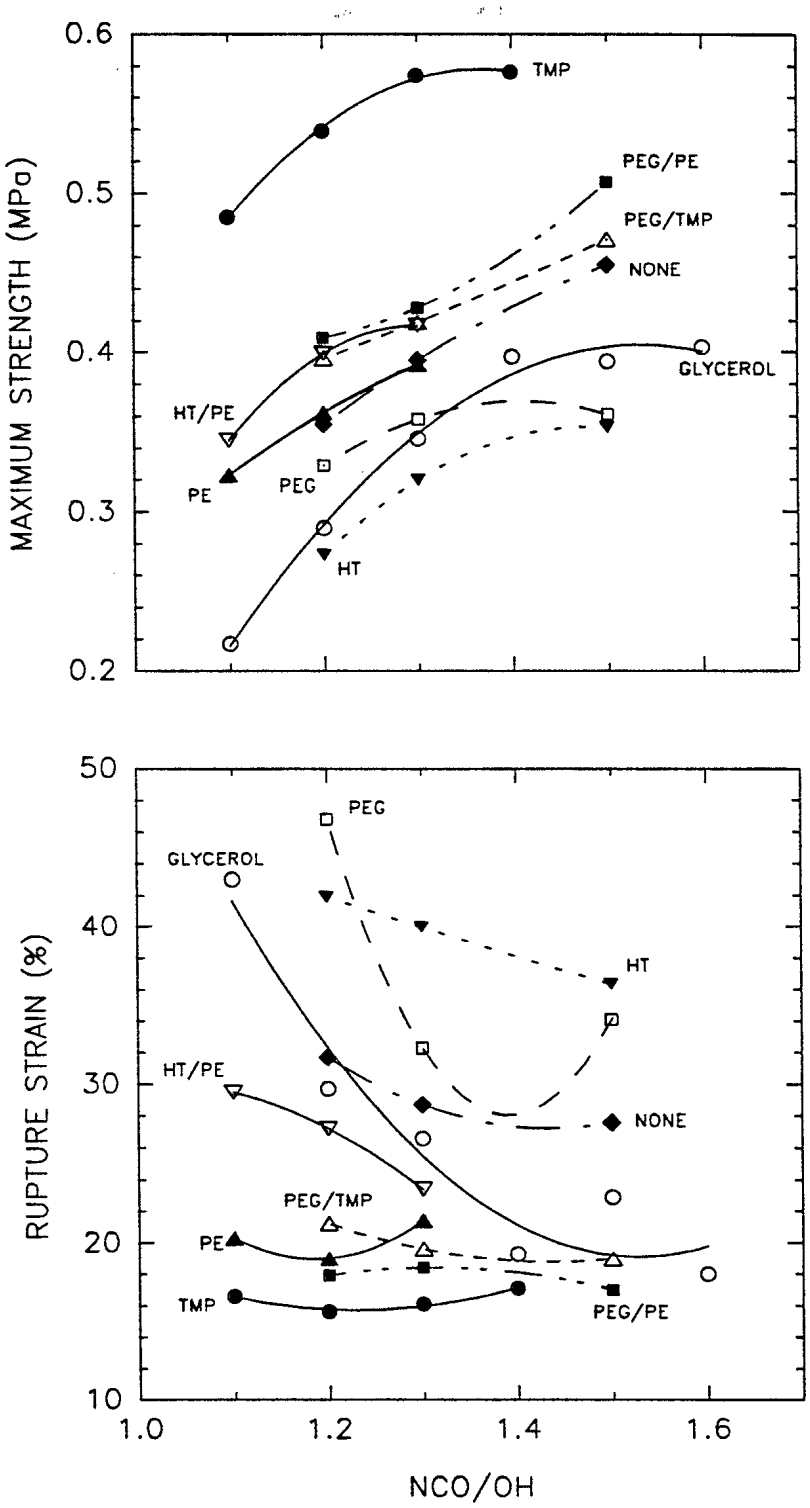

FIGURE 1 - Effect of NCO/OH ratio on mechanical properties of propellants

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An experimental minimum-smoke low-vulnerability rocket propellant composition was developed at DREV. The formulation contains phase stabilized ammonium nitrate (PSAN) as the oxidizer and an energetic binder based on branched glycidyl azide polymer (B-GAP). Different B-GAP polymers, obtained by varying some synthesis parameters, were incorporated into the formulation in order to study the effect on the mechanical properties of the resulting propellants. The reaction parameters investigated were the solvent and cleaving agent as well as the polyol used in the synthesis of B-GAP. The study highlighted the fact that some experimental parameters selected for the polymer synthesis have a strong influence on the mechanical properties of the propellant processed with B-GAP.

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Glycidyl azide polymer
GAP
Branched structure
Synthesis parameters
Cleaving agent
Polyol
Molecular weight
Equivalent weight
Hydroxyl functionality
Viscosity
Mechanical Properties
Rocket Propellants
Phase Stabilized Ammonium Nitrate
Energetic binder
Reaction Solvent
Polymer blending
Propellant formulation
low smoke
low vulnerability
rupture strain
maximum stress
modulus
elongation

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