

„High Energetic Composite Propellants based on AP and GAP/BAMO Copolymers”

K. Menke, P. B. Kempa, T. Keicher;

Fraunhofer-Institut für Chemische Technologie (ICT), Pfinztal, GE

A. Kawamoto, J. A. Saboia Holanda,

Aerospace Technical Center-CTA / Space Aeronautical Institute-IAE, Sao Jose dos Campos – SP /
Brazil**Abstract**

High energetic smoke reduced composite propellants have been formulated with bi- and trimodal AP fractions, up to 12% RDX, GAP/BAMO copolymers and BDNPF/A as a plasticizer. The propellants exhibit a convenient property profile with high thermodynamic performance, a specific impulse up to 2500 Ns/kg at 7 MPa, a smooth burning behaviour with burn rates from 16 to 30 mm/s at 10 MPa and pressure exponents of $n = 0,40$ with RDX and $n < 0,30$ without addition of nitramines. In comparison to AP/GAP propellants the new polymer has its main advantage in lowering the glass transition temperature and easier processing abilities. Further improvements are likely to achieve even a better overall performance than comparable AP/GAP formulations.

Introduction

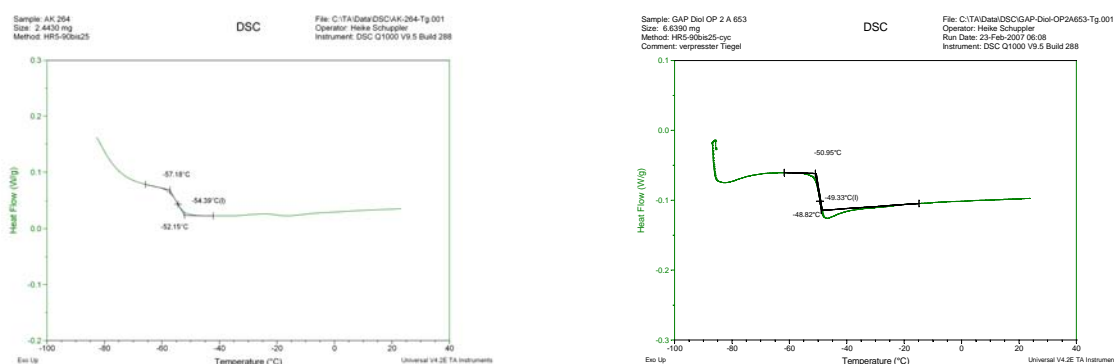
The energetic binder polymer GAP has been shown to be a useful binder for high energetic rocket propellants^{1,2)}. Together with energetic plasticizers like nitrate esters, aliphatic nitro- and nitramine compounds it offers high thermodynamic energy and performance with traditional oxidizers like ammonium perchlorate^{3,4,5)}, with nitramines^{6,7)} and even with low energetic oxidizers like ammonium nitrate⁸⁾. Due to its positive heat of formation, good oxygen balance and high density GAP opens the gate to high energetic composite propellants with much lower amount of solids and yet higher specific impulse and overall performance than traditional composite propellants based on AP/HTPB ingredients. With GAP it is possible to formulate composite rocket propellants with higher energy, higher density and higher burning rates in connection to a significant reduced content of hydrogen chloride and chlorine in the exhaust. Consequently it is pointing directly to high energetic chlorine reduced low pollution propellants for an eco friendly environment.

Due to its higher content of azido groups PolyBAMO offers an enhanced kinetic and thermodynamic advantage for propellant application in comparison to GAP^{4,5)}. Its disadvantage however is its high melting point, its higher glass transition point and poor processibility in comparison to GAP. To overcome these drawbacks GAP/BAMO copolymers might be the binders of choice for formulation of high energetic rocket propellants to keep the advantages of AP/GAP formulations and even improve their processibility, glass transition points and mechanical properties. It was the objective of this study to investigate the behaviour of GAP/BAMO copolymers in propellant formulation with AP, AP/RDX

and a convenient energetic plasticizer. BDNPF/A was chosen due to experiences with the AP/GAP propellants³⁾ and to keep a low mechanical and applicable sensitivity of the system.

Raw Materials

The GAP/BAMO Copolymer used for this study was synthesized and characterized by A. Kawamoto et.al.. Its synthesis and properties are described in reference 9 (poster 71) of this conference. AK 264 was synthesized by copolymerization of Bis(bromomethyl)oxetane with epichlorhydrin followed by azidation. The resulting prepolymer contains roughly 30 to 40% PolyBAMO. It is endowed with an average molecular weight of $M_n = 1380$, OH equivalent weight of 1021, a significant low viscosity at room temperature and a glasstransition temperature of $T_g = -54,39\text{ }^\circ\text{C}$ which proves to be lower than that of GAP diole with $T_g = -49,33^\circ\text{C}$ as it was determined by DSC (figure 1 and 2).



Figures 1 and 2: Glass transition points of the GAP/BAMO copolymer AK 264 and GAP diole determined by DSC

Its heat of combustion was determined by burning a sample of AK 264 with pure oxygen in a microcalorimetric bomb to be $\Delta H_c = 20842,5\text{ kJ/kg}$. By elemental analysis its carbon, hydrogen contents were determined to be C: 36,20% and H: 5,21%. With the following reference values for heat of formation: $\Delta H_{\text{CO}_2} = -393,52\text{ kJ/mol}$ and $\Delta H_{\text{H}_2\text{O}} = -285,83\text{ kJ/mol}$ the heat of formation for AK 264 amounts to $\Delta H_F = 1595,3\text{ kJ/kg}$. According literature the values for GAP diole and for PolyBAMO are $\Delta H_F = 1171,5\text{ kJ/kg}$ and $\Delta H_F = 2209\text{ kJ/kg}$ respectively¹⁰⁾. $\Delta H_F = 1595,3\text{ kJ/kg}$ corresponds to a composition of 60% GAP diole and 40% PolyBAMO.

A commercial available GAP diole sample was used for comparison. The energetic prepolymer from charge OP2A 653 was purchased from SNPE. Its basic characteristics are $M_n = 2000\text{ g/mol}$ and $\text{OH}_{\text{EQ}} = 1470\text{ g/EQ}$. Curing of AK 264 and GAP diole was performed with the triisocyanate N 100 and DBTDL as curing catalyst. In formulation GAP-11 additional crosslinking of AK 264 was achieved with a GAP triole prepolymer from SNPE with $M_n = 900\text{ g/mol}$ and $\text{OH}_{\text{EQ}} = 413\text{ g/EQ}$.

BDNPF/A was applied as plasticizer and super fine iron oxide with $240\text{ m}^2/\text{g}$ surface area (SFIO from MACH I) was used as burnrate catalyst.

In order to achieve a high energetic and easy working propellant system AP and small amounts of RDX were set as oxidizer and energetic solids. AP fractions were based on a trimodal system with $192\text{ }\mu\text{m}$, $45\text{ }\mu\text{m}$ and $6,6\text{ }\mu\text{m}$ with 0,6% TCP, RDX only on one charge with $5\text{ }\mu\text{m}$ mps.

Propellant formulations

Formulation of rocket propellants with GAP/BAMO Copolymer was closely correlated to AP/GAP propellants to achieve a maximum of specific impulse correlated to an oxygen balance between $-5\% \leq O_2 \leq 0\%$, good processibility in the slurry casting process together with applicable chemical stability and sensitivity. Propellant formulations are outlined in the tables 1 and 2.

Tables 1 and 2: Ingredients and formulations of AP/GAP and AP/GAP/BAMO (green columns) rocket propellants with and without RDX

Ingredients	density	O ₂ bal.	GAP-02	GAP-03	GAP-04a	GAP-05b
	g/cm ³	%	ma%	ma%	ma%	ma%
AP 192 µm SNPE	1,95	34,04	40	40	42	42
AP 45 µm SNPE	1,95	34,04	20	20	21	21
AP 6,6 µm + 0,6%TCP	1,95	33,84	11	11		
RDX 5 µm Dyno	1,816	-21,61			11,5	11,5
GAP Diol OP2A 653	1,29	-118,26	12,75		10,99	
AK 264	1,29	-119,9		11,83		10,1
N100	1,14	-200,58	1,75	2,67	1,51	2,4
BDNPF/A	1,39	-57,64	14	14	12,5	12,5
Iron-III-oxide 240m ² /g	5,24	10,02	0,5	0,5	0,5	0,5
DBTDL	drops		5	5	5	5
Total			100	100	100	100
Theor. Density	g/cm³		1,724	1,721	1,737	1,734
O₂ bal. propellant	%		-2,46	-3,41	-4,22	-5,12
Total solids	ma%		71,5	71,5	75	75
R=NCO/OH value	EQ		1,05	1,20	1,05	1,26
Plasticizer in binder	%		49,12%	49,12%	50,00%	50,00%

Ingredients	density	O ₂ bal.	GAP-07	GAP-09	GAP-10	GAP-11
	g/cm ³	%	ma%	ma%	ma%	ma%
AP 192 µm SNPE	1,95	34,04	43,2	43,6	42,4	42,4
AP 45 µm SNPE	1,95	34,04	21,6	21,8	21,2	21,2
AP 6,6 µm + 0,6%TCP	1,95	33,84	11,8		11,6	11,6
RDX 5 µm Dyno	1,816	-21,61		11,9		
GAP Diol OP2A 653	1,29	-118,26		13,02	15,96	
GAP Triole	1,29	-112,86				1,23
AK 264	1,29	-119,9	18,46			12,12
N100	1,14	-200,58	4,34	1,78	2,19	2,85
BDNPF/A	1,39	-57,64		7,4	6,05	8,1
Iron-III-oxide 240m ² /g	5,24	10,02	0,6	0,5	0,6	0,5
DBTDL	drops		5	5	5	3
Total			100	100	100	100
Theor. Density	g/cm³		1,739	1,749	1,744	1,744
O₂ bal. propellant	%		-4,73	-3,49	-1,12	-0,67
Total solids	ma%		77,2	77,8	75,8	75,7
R=NCO/OH value	EQ		1,25	1,05	1,05	1,00
Plasticizer in binder	%		0,00%	33,33%	25,00%	33,33%

Table 1 gives the percentage of 4 composite propellants with AP and AP/RDX and 50% plasticizer in binder polymer, table 2 with plasticizer fractions from 0% to 33,3%. To achieve the desired O₂ balance and thus a high specific impulse formulations GAP-07 to GAP-11 have been equipped with higher solids loading than formulations GAP-02 to GAP-05b. According to precuring experiments AK 264 should be cured with a higher NCO concentration than GAP diole or additional crosslinking agents like GAP triole.

Thermodynamics

Thermodynamic performance calculations were carried out with the ICT code and data base developed by Volk and Bathelt¹¹⁾ and is now in charge of Paul Bernd Kempa. Standard values for specific impulse, volumetric specific impulse, combustion temperature T_c and molar output of gases n are given for a standard expansion ratio of 70:1 (1016 psi). The given data of energetic ingredients have been taken from ICT's database¹⁰⁾.

The values for the above listed formulations are outlined in tables 3 and 4. All three formulations with a binder based on AK 264 and GAP diole have a maximum of specific impulse between 2450 Ns/kg ≤ Isp ≤ 2500 Ns/kg which is reached with 70% - 77% energetic solids depending on the amount of plasticizer and the incorporation of nitramines like RDX and HMX. Incorporation of the oxygen rich energetic plasticizer BDNPF/A reduces the necessary amount of AP for the maximum of specific impulse. Addition of 10 – 15% RDX gives a 1% higher Isp compared to the formulations without nitramine. The application of a higher concentration of N100 drops the specific impulse a little bit compared to the corresponding AP/GAP formulations. Overall both AP/GAP and AP/GAP/BAMO propellants show equal thermodynamic performances. A better adaption of curing system and further optimization however might bring slight advantages to propellants with GAP/BAMO copolymers.

Traditional AP/HTPB propellants require 85% to 88% solids to achieve a similar thermodynamic performance than AP/GAP and GAP/BAMO propellants. A good castable AP/HTPB propellant with 86% solids and trimodal 85% AP fraction has an Isp = 2410 Ns/kg, density ρ = 1,69 g/cm³ and Isp*ρ = 4080 Ns/dm³, values which are about 3% lower than the thermodynamic performances of the AP/GAP and AP/GAP/BAMO propellants like GAP-10 and GAP-11 with 75% AP.

Table 3 and 4: Results of thermodynamic performance calculations from AP/GAP and GAP/BAMO propellant formulations

Thermodynamics	Exp.70:1	GAP-02	GAP-03	GAP-04a	GAP-05b
Prepolymer		GAP	GAP/BAMO	GAP	GAP/BAMO
RDX	%	--	--	11,5	11,5
BDNPF/A of binder	%	49,12	49,12	50	50
Spec. Impulse	Ns/kg	2482	2478	2503	2499
Spec. Impulse	s	253,1	252,6	255,1	254,7
Volum. Spec. Impulse	Ns/dm ³	4280	4266	4348	4334
Char. Velocity c*	m/s	1496	1498	1512	1514
Tc Combustion	K	3068	3067,5	3112	3108,8

Thermodynamics	Exp.70:1	GAP-07	GAP-09	GAP-10	GAP-11
Prepolymer		GAP/BAMO	GAP	GAP	GAP/BAMO
RDX	%	--	11,9	--	--
BDNPF/A of binder	%	0,00	33,33	25,00	33,33
Spec. Impulse	Ns/kg	2469	2504	2479	2478
Spec. Impulse	s	251,7	255,2	252,7	252,6
Volum. Spec. Impulse	Ns/dm ³	4293	4380	4325	4323
Char. Velocity c*	m/s	1503	1511	1490	1487
Tc Combustion	K	3059	3110,5	3055,5	3050

Processibility, Mechanical Properties and Glass Transition Temperatures

The tables 5 and 6 show the experimental values for the end of mixing viscosities, surface hardness, mechanical properties and glass transition temperatures of the propellants GAP-02 till GAP-11.

Contrary to AP/HTPB propellants with 85% energetic solids AP/GAP/BAMO formulations are much easier castable and processable. With about 50% BDNPF/A plasticizer the propellants GAP 03 and 05b with AK 264 present excellent casting viscosity, even propellant GAP 07 with 76,6% energetic solids and without plasticizer reflects good processibility. The higher softness of the GAP/BAMO propellants compared to those with pure GAP diol may have its reason from plasticizing parts within the lab product AK 264. Further optimization of kind and ratio of curing agent and the amount of plasticizer will be necessary if the polymer binder will be produced in a larger scale. Similar results are obtained for the mechanical properties of AP/GAP and AP/GAP/BAMO propellants with lower or 0% of plasticizer. Further optimization and the addition of a suitable bonding agent appears to be necessary for both types of propellant formulations. The AP/GAP propellants should be equipped with a higher tensile strength, a better elongation and a reduced modulus. AP/GAP/BAMO propellants must be equipped with a higher tensile strength and modulus.

The glass transition points have been determined by DSC and TMA. Both values are outlined in the tables 5 and 6. For both GAP and GAP/BAMO polymers the glass transition points decrease with increasing amount of plasticizer. For 50% BDNPF/A in binder fraction T_g drops below -50 °C for AP/GAP/BAMO propellants like GAP-03 and GAP-05b. Compared to AP/GAP propellants like GAP 02 and GAP 04a with T_g = -48 °C the propellants with AK 264 (T_g = -50°C) show a clear advantage. The same result is observed for GAP-11 in comparison to GAP-09 with 33,33% plasticizer. As it is outlined above the prepolymer GAP/BAMO AK 264 with -54,3°C already shows a lower glass transition point than GAP diol - here it is GAP OP 2A 653 from SNPE with -49,3°C.

Chemical Stability and Sensitivity

For both AP/GAP and AP/GAP/BAMO propellants the values of dutch test, vacuum stability and deflagration point are clearly within the required limits (dutch test: 0,14 – 0,22 % mass loss, vacuum stability: 0,27 – 0,36 ml/g gas evolution, ignition temperature 200-206 °C). They represent the results of short term tests for the chemical stability. Limits for the dutch test are 2% mass loss within 8 – 72 h

heating at 105°C, limits for vacuum stability are 1,2 ml/g gas evolution within 40 h heating at 100°C. Both terms have been fulfilled within a good margin.

Tables 5 and 6: Experimental values of casting viscosity, mechanical properties and glass transition points for AP/GAP and AP/GAP/BAMO propellants

Processibility		GAP-02	GAP-03	GAP-04a	GAP-05b
BDNPF/A plasticizer	Ma%	49,1	49,1	50	50
viscosity before curing	Pas	72	48	96	72
viscosity EOM	Pas	64	44	76	56
Surface Hardness	Shore A	60	20	53	20-25
Mechanical properties 20°C/50 mm/min					
max. tensile strength	N/mm ²	0,41	0,26	0,36	0,21
elongation at max t. s.	%	7,8	24,4	13,1	37,4
elongation at break	%	10,3	26,8	18,3	44,4
E-modulus	N/mm ²	7,42	1,61	5,44	0,9
Tg (DSC)	°C	-48,3	-50,4		-50,5
Tg (TMA)	°C	-49,85	-50,7		-51

Processibility		GAP-07	GAP-09	GAP-10	GAP-11
BDNPF/A Plasticizer	Ma%	0,0	33,33	25	33,33
viscosity before curing	Pas	408	224	186	120
viscosity EOM	Pas	248	152	160	104
Surface Hardness	Shore A	65	70	78	51,4
Mechanical properties 20°C/50 mm/min					
max. tensile strength	N/mm ²	0,68	0,56	0,73	0,44
elongation at max t. s.	%	12,8	7,6	4,8	8,6
elongation at break	%	15,4	12,2	6,6	10,7
E-modulus	N/mm ²	8,17	14,57	23,39	7,09
Tg (DSC)	°C	-40,9	-44,4	-43,3	-46,5
Tg (TMA)	°C	-40,7	-43,8		

Sensitivity values have been determined by impact (5 Nm) and friction sensitivity (32 – 48 N) as it was outlined in ref. 9 for the monomers and prepolymers. The determined reaction limits of BAM hammer and friction apparatus are within a convenient scope if they are compared to those of AP/HTPB rocket propellants which were determined by the same operator.

Burning Behaviour

The burning behaviour was determined by Crawford measurements. Burning rates were determined on coated propellant strands with 5 mm x 5 mm cross section and 50 mm measuring distance.

The burning rate was expressed and fitted according to Vieilles law:

$$r = Ap^n \quad \text{or} \quad \ln r = n \ln p + \ln A$$

where p is the pressure, n is the pressure exponent and A is the burn rate constant (for propellants with $n \leq 1$ and explosives $n \leq 1$). Practicable values for rocket propellants are $n \leq 0,60$, more convenient

ones for TMP propellants are $n \leq 0,50$. The values for AP/HTPB composited propellants are usually in the range between $0,30 \leq n \leq 0,50$.

The burning behaviour of the reference AP/HTPB propellant with 85% AP in the same trimodal fraction as it was used for the AP/GAP and AP/GAP/BAMO propellants formulated with 0,7 % iron-III-oxide with 6 m²/g is presented in figure 3.

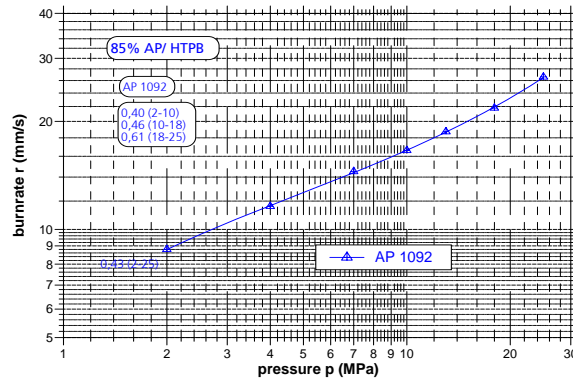
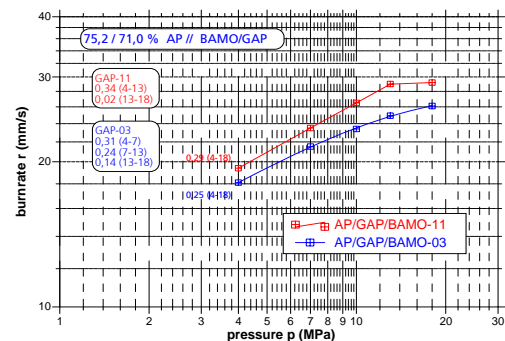
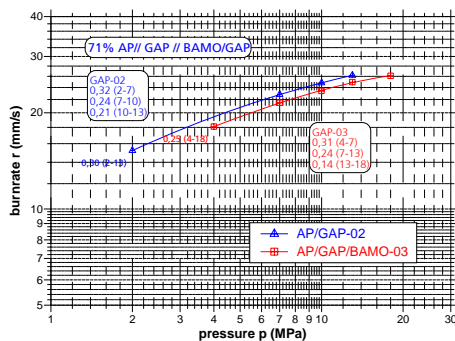


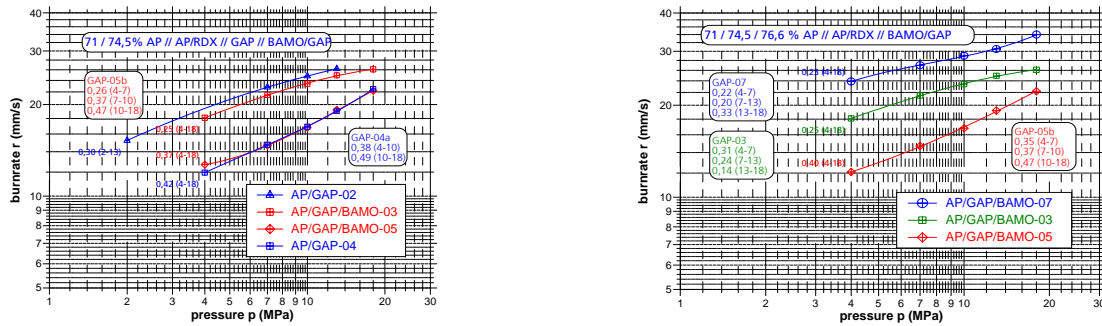
Figure 3: Burn rate versus pressure diagram for a conventional AP/HTPB propellant with 85% AP. The burning behaviour of the propellant formulations with AK 264 comparatively with GAP, accordingly for the formulations from the tables 1 and 2, are graphically presented in Figures 4, 5, 6, and 7.



Figures 4 and 5: Burning behaviour of AP/GAP and AP/GAP/BAMO propellants with the same (GAP-02 and GAP-03 with 50% BDNPF/A) and different amounts of plasticizer. (GAP-03 with 50% and GAP-11 with 33,33% BDNPF/A)

AP/GAP and AP/GAP/BAMO propellants GAP-02 and GAP-03 with the same amount of plasticizer show nearly equal burnrates and pressure exponents. The burnrate enhancing effect of the azido polymers is reflected in burnrates $r = 23 - 24$ mm/s at 10 Mpa compared to $r = 16-17$ mm/s for the AP/HTPB propellant with the same trimodal AP fraction being used for the GAP and GAP/BAMO propellants. Further increase of burnrates up to 29 and 31,5 mm/s is observed with decreasing amount of BDNPF/A plasticizer in the propellants GAP-07, GAP-10 and GAP-11. All AP/GAP and AP/GAP/BAMO propellants exhibit a promising burn rate behaviour with decreasing pressure exponents in the high pressure region. For AP/GAP/BAMO propellants a slight advantage is observed in this case. For GAP-11 and nearly for GAP-03 the decrease of pressure exponents even reaches a

plateau burning level. Similar findings have been described in reference 4 for propellants based on the AP/PolyBAMO system.



Figures 6 and 7: Difference in burning behaviour between the AP/GAP/BAMO and AP/GAP formulations (GAP-02, 03 and 07) in comparison to the AP/RDX/GAP and AP/RDX/GAP/BAMO (GAP-04a and 05b) formulations.

Propellants with 11 - 12% RDX 5 μ m like GAP 04a, 05b and 09 clearly differ in burn rates with 16 – 19 mm/s at 10 Mpa compared to propellants with AP 6 μ m like GAP 02, 03, 07 and 10 with 23 to 31 mm/s at 10 Mpa. The burnrates and pressure exponents of AP/RDX/GAP/BAMO propellant GAP-05b are almost equal to those of the AP/RDX/GAP propellant GAP-04a. The AP 6 μ m formulations however do have an advantage in higher burnrates and lower pressure exponents in comparison to those with RDX 5 μ m. Nevertheless all propellant formulations exhibit quite convenient burning behaviour which may be brought to good use in rocket motor applications.

Conclusion

A copolymer with about 60% GAP and 40% BAMO sequences was investigated and has been proved to be a convenient binder polymer for the formulation of high energetic rocket propellants. In particularly optimized formulations of smoke reduced rocket propellants with trimodal AP or AP/RDX fractions they demonstrate superior properties. With varying amounts from 0 % to 50 % of the energetic plasticizer BDNPF/A and 70 to 77 % energetic solids the propellants exhibit a thermodynamic specific impulse of $2470 \leq I_{sp} \leq 2500$ Ns/kg and $4250 \leq I_{sp} \cdot \rho \leq 4370$ Ns/dm³ for the volumetric one at 70:1 expansion ratio. Being formulated with AP only the specific impulse stays between 2470 and 2480 Ns/kg, by incorporation with 11-12% RDX 2499 – 2505 Ns/kg have been achieved. The thermodynamic specific impulse of a comparable AP/HTPB propellant with 85 % AP in the same trimodal distribution only reaches 2410 Ns/kg and 4080 Ns/dm³ expansion ration 70:1, which is about 3 % lower than the corresponding AP/GAP or AP/GAP/BAMO propellants. The curing system and mechanical properties of the AP/GAP and AP/GAP/BAMO propellants still have to be optimized. The applied GAP/BAMO copolymer AK 264 demonstrates clear advantages by a lower end of mixing viscosity and by a reduced glass transition point than it could be achieved with GAP alone. In the investigated propellant formulations only GAP/BAMO copolymers reached a glass transition point $\leq -50^\circ\text{C}$.

The burn rates of the AP/RDX/GAP or AP/RDX/GAP/BAMO propellants fall between 16 – 19 mm/s at 10 Mpa and the pressure exponents between $n = 0,40 - 0,42$ from 4 – 18 Mpa, which is roughly the same which was achieved by the AP/HTPB propellant. The burnrates of the AP/GAP and AP/GAP/BAMO propellants differ significantly with the amount of plasticizer and fall between 23 – 31 mm/s at 10 Mpa with 23 mm/s for 50% and 31 mm/s for 0 – 25% BDNPF/A in the binder fraction and pressure exponents between $n = 0,23 - 0,30$ from 4-18 MPa. The increase of burnrates at lower pressures is accompanied by a decrease of pressure exponents at higher pressures. Especially the investigated AP/GAP/BAMO propellants are endowed with a beneficial reduction of pressure exponents down to plateau burning behaviour at pressures above 13 Mpa. The GAP/BAMO copolymers show even more than GAP alone a favourable burning behaviour and may be brought to good use in rocket motor applications.

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List of Abbreviations and Symbols

AK 264	Lab synthesized copolymer with about 40% BAMO and 60% GAP
AN	Ammonium nitrate
AP	Ammoniumperchlorate
BAMO	Bisazidomethyloxetan
BDNPF/A	1:1 mixture of Bisdinitropropylformal/-acetal
C*	Characteristic velocity (by thermodynamic calculation)
DBTDL	Dibutylzinndilaurate
DSC	Differential Scanning Calorimetry
GAP	Glycidylazidopolymer
GAP/BAMO	Copolymer from BAMO and GAP
HMX	Octogen
HTPB	Hydroxyterminated Polybutadiene
$I_{sp} = I_{SPEQ}$	Specific Impulse (Equilibrium Flow)
$I_{SPEQ} \cdot \rho$	Volumetric Specific Impulse (Equilibrium Flow)
MACH I	US Company
mps	median particle size
n	pressure exponent according to Vieille's law
N100	n-Hexylmethanediisocyanate - trimer
p. B.	part of the binder fraction
PolyBAMO	Bisazidomethyloxetan
r	Burning rate
ρ	density
RDX	Cyclotrimethylenetrinitramine
SFIO	Super fine iron oxide (MACH I)
Tc	Combustion Temperature
TCP	Tricalciumphosphate
TMP	Tactical missile propellants

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