

Fast Burning Rocket Propellants Based on Silicone Binders – New Aspects of an Old System

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Dedicated to Professor Hiltmar Schubert on the Occasion of his 75th Birthday

Summary

Fast burning rocket propellants based on ammonium perchlorate (AP) and polydimethylsiloxane (PDMS) binders have been formulated and basically characterized in aluminized and non aluminized versions. The thermodynamic performance of PDMS bound propellants is limited by the evaporation point of silicon dioxide (cristobalite at 2863 K), which is formed during the combustion. The maximum specific impulses calculated with an expansion ratio of 70:1 are $I_{sp} = 2340$ N s/kg (238 s) for AP/PDMS and $I_{sp} = 2430$ N s/kg (247 s) for AP/Al/PDMS formulations. With a tri-modal AP-fraction of 200 μm , 30 μm and 2 μm it was possible to achieve processable propellants with 83% and 84% solid loading. They give burning rates close to 60 mm/s at 10 MPa and pressure exponents between 0.5 and 0.6 in the range of 7–25 MPa. The thermodynamic specific impulse of these systems was calculated to $I_{sp} = 2319$ N s/kg (236 s) for AP/PDMS and $I_{sp} = 2403$ N s/kg (245 s) for AP/Al/PDMS formulations at 70:1 expansion ratio. Measurements of the glass transition point by DMA and TMA detected a time dependent partial crystallization of the used PDMS close to T_G in the temperature range from -44°C to -60°C . Basic tests reveal high thermal and chemical stability with flash points over 300°C and zero detonation sensitivity at 50 mm diameter. The property profile, so far determined, tends to fulfil the demands for applications as propellants with high burning rates and operating pressures together with a high thermal stability and a low vulnerability.

1 Introduction

The use of silicone resins as a part of the inhibition and thermal protection layers is well known in the frame of rocket motor technology^(1,2). Propellant formulations based on silicone binders are less known. They have been published in patents about 40 years ago. The first publication⁽³⁾ was made by Kuhn and Netzer in 1964. Gas generating propellants for fire extinguishers based on ammonium perchlorate, nitroguanidine and a silicone resin were found to be stable up to 140°C . In the same year Piehl claimed the burning rate enhancing effect of a mixture of 0.3% ferrocene and 0.8% silicone resin for a composite propellant based on AP and Butarez[®], a carboxyl-terminated polybutadiene resin⁽⁴⁾. Later on, the work of Doriath leads to SNPE's Butacene[®] – the burning rate enhancing prepolymer which

contains the ferrocene moiety chemically bound to the polybutadiene chain via an alkylsilane spacer⁽⁵⁾. Doriath also published in 1976 on composite propellants based on AP, Al and a silicone binder with a specific impulse of 233 s and burning rates up to 35 mm/s at 10 MPa⁽⁶⁾. In 1971, Davenas compared composite propellants with polyurethane, polybutadiene and silicone binders with respect to their stability against γ irradiation⁽⁷⁾. Silicone binders show only slight degradation. Another siloxane based rocket propellant with AP, specially treated magnesium powder and a silicone binder, has been developed and patented by Sanden about 20 years later⁽⁸⁾. The propellants may have been part of the series with partly neutralized acidic combustion products. Further on, the high temperature stability, its favorable oxygen balance and the possibility to achieve high burning rates enable formulations with silicone binders and alkali perchlorates or AP and alkalinitrates for airbag applications. Grebert et al. applied for a first patent⁽⁹⁾ for airbag applications in 1974⁽⁹⁾. It was followed by a series of patents and commercialized products which start about 20 years later^(10–12).

The goal which motivated us to start with formulations working on silicone bound rocket propellants concerns the development of propellants with ultrahigh burning rates, a comparatively low sensitivity and an applicability at high and low temperatures. Objectives for their application are rocket motors for highly accelerating missiles or end burning grains to enhance the impetus and penetration force of missiles and gun projectiles.

At the beginning the propellant concept was rather simple. A commercial, two-component polydimethylsiloxane (PDMS) resin (Wacker RTV 601[®]) with vinylsilane and SiH groups was used together with a mixture of AP-fractions containing ultrafine ammonium perchlorate (UFAP) with a mean particle size (mps) of 2 μm at a suitable extent. Aluminium with 5 μm mps and spherical particle shape was used as a metal fuel and iron-III-oxide with a surface area of 6 m^2/g as a burning rate catalyst.

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2 Propellant Formulation and Characterization

2.1 Thermodynamic Optimization

Thermodynamic calculations were carried out with the ICT-code and database developed by Volk and Bathelt^(13,14). The specific impulse and volumetric specific impulse were calculated for a constant pressure of 7 MPa and an expansion ratio of 70:1. Thermodynamic investigations of the propellant system were exercised within the following concentration limits:

- Total solids /. AP + Al : 75–90 mass-%
- Al : 0–20 mass-%
- Silicone resin : 25–10 mass-%

The results are presented in map like diagrams which are illustrated by Figures 1 and 2. Figure 1 shows the lines of constant specific impulse I_{SP} in N s/kg, Figure 2 shows the same for the volumetric specific impulse $I_{SPVOL} = I_{SP} \cdot \rho$ in N s/dm³. Without Al the maximum value of the specific and the volumetric specific impulse can be detected between 83 and 84 mass-% AP at $I_{SP} = 2340$ N s/kg and $I_{SPVOL} = 4000$ N s/dm³.

With about 7% Al and 79% AP the thermodynamic maximum of the specific impulse amounts to 2430 N s/kg and the maximum volumetric specific impulse to 4350 N s/dm³. Due to the higher density of the dimethylsiloxane polymer the maximum amount of solids for a castable formulation is limited to 83 mass-%. For this reason the complete thermodynamic profile was calculated only for processible formulations with 82% AP, 1% Fe₂O₃ and 17% dimethylsiloxane polymer as well as 77% AP, 6% Al, 1% Fe₂O₃ and 16% silicone resin which have been proved to be at the limit of processibility. The thermodynamic results for these two propellant systems are shown in Table 1 and Table 2.

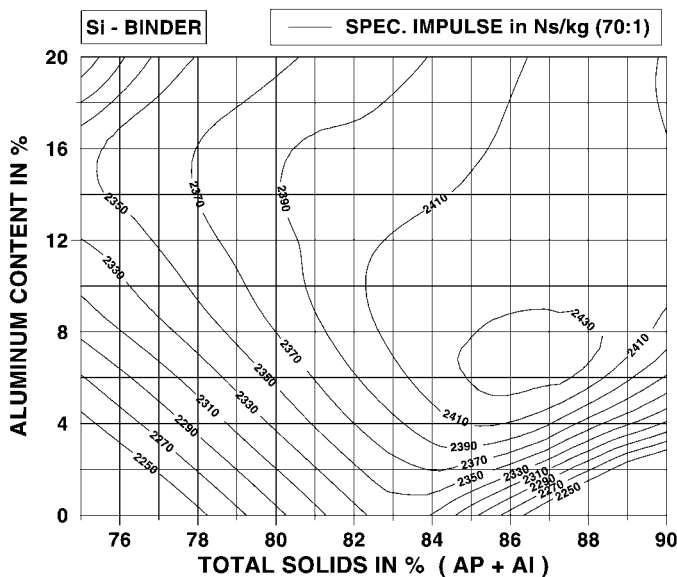


Figure 1. Thermodynamic specific impulse of the AP/Al/PDMS propellant system at 70:1 expansion ratio.

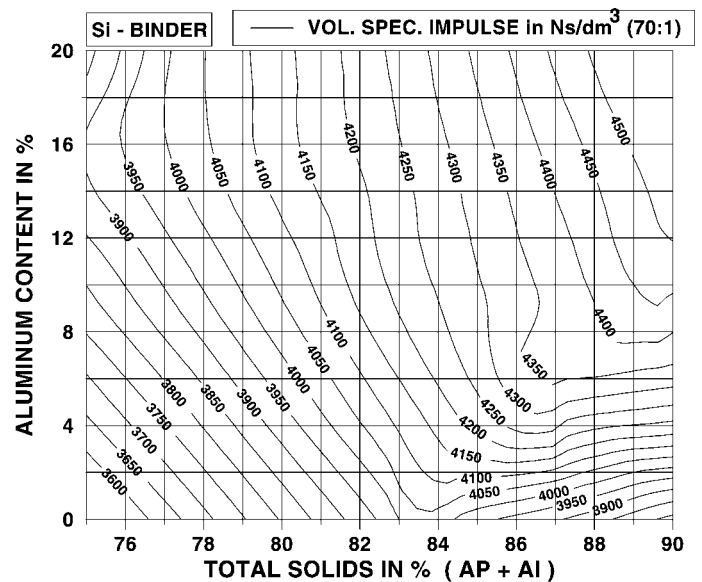


Figure 2. Thermodynamic volumetric specific impulse of the AP/Al/PDMS propellant system at 70:1 expansion ratio.

Table 1. Calculated thermodynamical properties of specified AP/Al/PDMS propellants.

BATCH – No:		37, 43	38, 42
SOLIDS	in mass-%	83	84
AP		82	77
Fe ₂ O ₃		1	1
Al		–	6
PDMS silicone resin		17	16
O ₂ -Balance	in %	–1.33	–6.64
T _c	in K	2863	2863
T _e	in K	1699	1996
c _p /c _v		1.19	1.21
c*	in m/s	1376	1359
I _{SP}	in N s/kg	2319	2403
I _{SPVOL}	in N s/dm ³	3968	4206
Density	in g/cm ³	1.71	1.75

In Table 1 the values for the specific impulse and the volumetric specific impulse are listed together with propellant density, oxygen balance, combustion and expansion temperature T_c and T_e , isentropic exponent $\kappa = c_p/c_v$ and characteristic velocity c^* .

A characteristic disadvantage of siloxane bound propellants is revealed by the combustion temperature T_c . It is limited by the vaporization of the silicon dioxide at 2863 K formed during combustion. The higher combustion energy of the formulations No. 38 and No. 42 with 6% Al is expressed by $T_e = 1986$ K, which is due to the melting point of SiO₂ cristobalite. Table 2 compiles the molar and mass percentages of the calculated main reaction products after expansion. Due to a more negative oxygen balance the metalized formulations produce higher molar percentages of H₂ and CO thus leading to a smaller molecular weight of the combustion gases.

About 15.35 mass-% and 25.98 mass-%, respectively, of the partly liquid and solid reaction products FeCl₂, SiO₂ and

Table 2. Calculated reaction products of specified AP/Al/PDMS propellants.

BATCH-NR:	37, 43		38, 42	
SOLIDS in mass-%	83		84	
AP	82		77	
Fe ₂ O ₃	1		1	
Al	–		6	
PDMS silicone resin	17		16	
	mol-%	mass-%	mol-%	mass-%
HCl	19.37	24.51	18.60	22.89
CO	1.10	1.07	5.47	5.17
H ₂	1.30	0.09	6.87	0.48
CO ₂	12.12	18.50	7.32	10.87
H ₂ O	49.06	30.67	41.83	25.43
N ₂	10.06	9.78	9.71	9.18
SiO ₂	6.61	13.77	5.80	12.96
Al ₂ O ₃	–	–	3.30	11.34
FeCl ₂	0.36	1.58	0.37	1.58

Al₂O₃ are produced, too. Together with 24.5% and 22.9% HCl the propellants obey to a CC AGARD signature classification⁽¹⁵⁾.

2.2 Processability

According to the thermodynamical optimization a high percentage of solids is necessary to reach a reasonable value of the specific impulse. A formulation with 83 mass-% AP and 17% polydimethylsiloxane contains about 77 vol-% solids, similar to a formulation with 88 mass-% AP and 12% HTPB. The castable formulations No. 37 and No. 43 with 82% AP and 1% Fe₂O₃ include about 76.8 vol-% solids, those of No. 38 and No. 42, which can be processed only with pressure, contain about 77.7 vol-%.

For this reason, it was very necessary to optimize the particle size distribution and the content of AP fractions used for the formulations. Three AP-fractions were applied for the tested propellant formulations. AP-fractions with 200 μm mps and with 30 μm mps were purchased from Japan Carlit. The materials consisted of fine crystallized and rounded particles with a narrow size distribution. The fractions turned out to be convenient for formulations with high solid loadings. To achieve high burning rates they were combined with 2 μm mps UFAP. The stuff was fabricated in Fraunhofer ICT by grinding the 200 μm AP in an air jet mill.

The dimethylsiloxane resin RTV 601 used for the binder fraction was purchased from Wacker. It was blended in the ratio of 4:6 with the short chain dimethylsiloxane plasticizer AK 100®. The viscosity measurements were performed with a Brookfield viscosimeter using T-spindle No. 9 at 30 °C processing and casting temperature.

Because an appropriate fraction of UFAP would be necessary for the designated purpose, we decided to have a look on the influence of processing viscosity on the variation of fraction content between AP 30 μm and AP 2 μm covering 60 parts of the total amount of perchlorate for different solid loadings from 76 to 84 mass-%. The remain-

ing 40 parts were filled with AP 200 μm mps. The result of this optimization is shown in Figure 3. The processing viscosity of the propellant batches passes a minimum between a content of 35% and 40% AP 2 μm from 60 parts of the AP 30 μm /AP 2 μm mixture. An optimized distribution of the particle size thus would contain about 17%–20% AP 2 μm from 83% solid loading. The applied concentration, however, was set a little bit higher to increase the burning rates.

2.3 Glass Transition Temperatures

Silicone resins are known for low glass transition temperatures and freezing points⁽¹⁶⁾. For this reason propellants with silicone binders are expected to have good low temperature properties, which always are of interest for the application profile. The test technique used for measuring the glass transition temperature was the dynamic mechanical analysis (DMA) and thermomechanical analysis (TMA). For DMA measurements, a typical sample configuration was a rectangular bar of 12 × 5 × 50 mm³. Measurements were carried out at a fixed frequency of 1 Hz (6.28 rad/s). The glass transition temperature was measured at different cooling and heating rates.

DMA: ramp up	rate of cooling:	12 °C/min, following
	rate of heating:	measuring of T_G at 1 °C/min
ramp down	rate of cooling:	measuring of T_G at 1 °C/min
TMA: ramp down	rate of cooling:	5 °C/min
	rate of heating:	5 °C/min

Table 3 shows the results of the glass transition temperature of the different samples and measurement options. The glass transition temperature T_G is defined by NATO STANAG 4540 as the maximum of the loss modulus G'' . Because this peak cannot be detected in all samples the

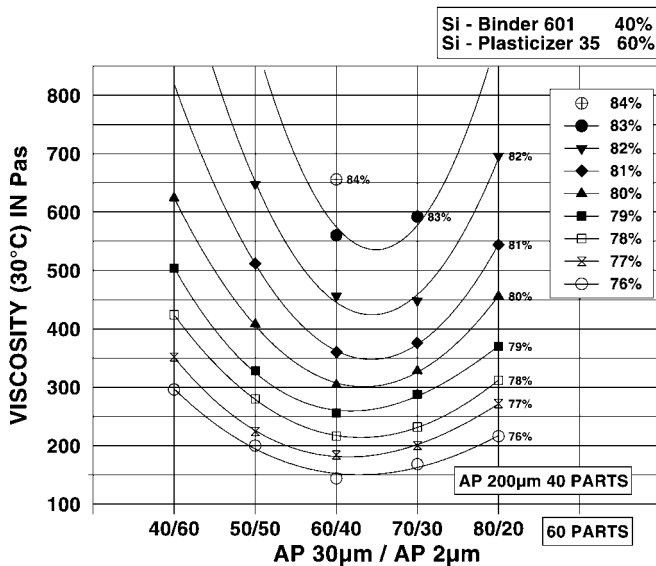


Figure 3. Casting viscosity of AP/PDMS propellant batches containing varying fractions of AP with 200 µm, 30 µm and 2 µm mps and increasing solid loadings.

maximum value of $\tan \delta$ has been determined, too, and has been listed in Table 3.

Concerning the propellant system Si 42, the glass transition temperature is -44.4°C and for Si 43, the glass transition temperature amounts to -46.0°C . Figure 4 shows the result of the DMA measurement of the binder.

As an interesting result of the DMA and TMA measurements, the mechanical properties close to the glass transition temperature depend on whether they are determined by heating or cooling the samples. This may result from the time depending process of the partial crystallization, thus the determination of the glass transition temperature differs between heating and cooling. To show that this is not a problem of inhomogeneous temperatures in the sample the following investigation was made. Sample 1 was cooled down at $1^\circ\text{C}/\text{min}$ until $T_G + 10^\circ\text{C}$ was reached. After this treatment, the temperature was kept constant at -65.5°C . After 10 minutes, the modulus G^* increases up to $+14\%$. The time (and temperature) depending process of partial crystallization occurs slowly after more than one hour, the modulus increases, as can be seen in Figure 5, from $6.0 \cdot 10^5 \text{ Pa}$ till $5.3 \cdot 10^7 \text{ Pa}$.

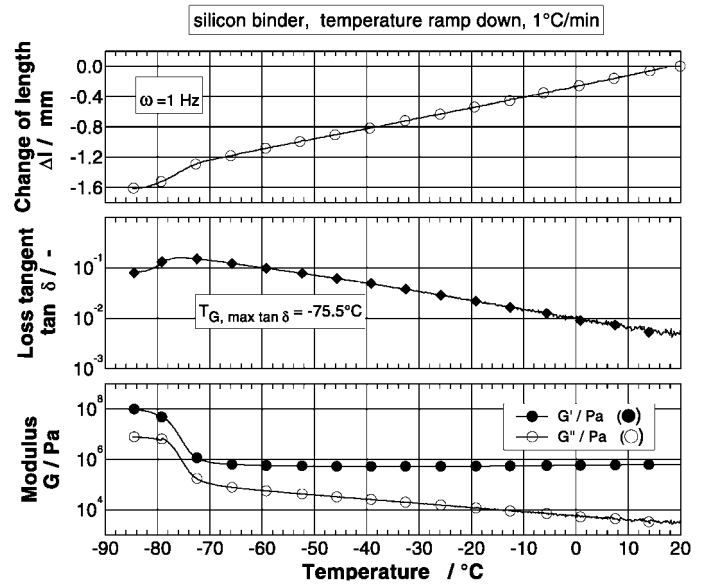


Figure 4. Plots of change of sample length, $\tan \delta$, storage modulus G' and loss modulus G'' from PDMS polymer RTV 601®, measured by DMA.

At first sight, the comparatively high glass transition temperatures and the partial crystallization at low temperatures have not been expected. It is, however, well known that freezing and crystallization of polymer chains depend on size, stereoregularity and variety of alkyl substituents⁽¹⁷⁾. The polydimethylsiloxane used for these propellant formulations is expected to undergo freezing and partial crystallization easier than siloxane polymers with alkyl substituents containing two and more carbon atoms.

2.4 Burning Behavior

The burning behavior was determined by Crawford measurements. Burning rates were determined on coated propellant strands with $5 \text{ mm} \times 5 \text{ mm}$ cross section and 50 mm measuring distance. In Table 4 two formulations based on AP/PDMS and two formulations based on AP/Al/PDMS are shown with their measured burning rates at 2, 4, 7, 10, 13, 18 and 25 MPa and a medium pressure exponent. The burning behavior of one aluminized formulation and one without Al is graphically presented in Figure 6. In both cases the achieved burning rates are close

Table 3. Glass transition temperatures T_G ($^\circ\text{C}$) of PDMS, PDMS with plasticizer and propellant samples determined by DMA and TMA.

Sample	Specification	Ramp up		Ramp down		Ramp up	Ramp down	Thermal expansion 1/K
		$T_{G, \max \tan \delta}$	$T_{G, \max G''}$	$T_{G, \max \tan \delta}$	$T_{G, \max G''}$	$T_{G, \text{TMA}}$	$T_{G, \text{TMA}}$	
1	RTV 601	-56.7	-	-75.5	-	-	-	-
2	40% RTV 601 60% AK 100	-38.7	-41.0	-	-	-	-	-
3	Si 42	-39.1	-44.4	-61.2	-65.4	-41.7	-76.1	$11.6 \cdot 10^{-5}$
4	Si 43	-40.0	-46.0	-60.1	-	-45.2	-67.7	$12.0 \cdot 10^{-5}$

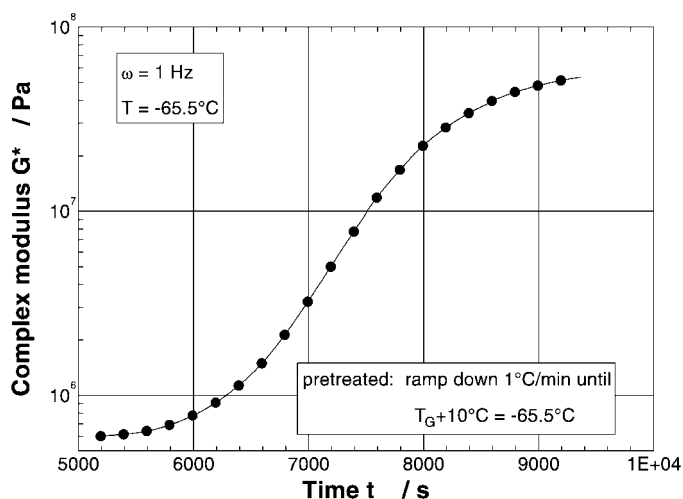


Figure 5. Increase of the complex modulus G^* with time by keeping constant the sample temperature at $T_G + 10^\circ\text{C} = -65.5^\circ\text{C}$ referred to partial crystallization.

to 60 mm/s at 10 MPa. The pressure exponents tend to move between 0.6 and 0.5 for aluminized formulations. For AP/PDMS propellants they are high at low pressures and decrease significantly at high pressures.

2.5 Chemical Stability and Sensitivity

Chemical stability and sensitivity were determined by basic tests. Typical values are listed in Table 5. In the Dutch test the weight loss of a 2 g propellant sample was determined by heating the sample at 105°C for 8–72 h. The vacuum stability was measured by the gas evolution of a 2.5 g sample heated for 40 h at 100°C under a pressure < 1 hPa. The flash point was determined in glass tubes at a heating rate of $20^\circ\text{C}/\text{min}$.

For the determination of the impact and friction sensitivity BAM methods have been used. The given values represent the energy of mechanical impact where at least 1 of 5 samples reacted.

The Gap test was performed on cylindrical tubes of 50 mm diameter and 100 mm length using a PMMA-Gap material and pressed booster charges of RDX/Wax (95:5).

Within these basic tests the propellants demonstrate superior chemical and thermal stability. They show higher flash points than AP/HTPB and AP/PU composite propellants in all cases⁽¹⁸⁾.

The detonation sensitivity appears to be low, but it was not proved if the critical diameter could be higher than 50 mm as for typical composite propellants. The impact and friction sensitivity are similar to the fast burning AP/HTPB propellants.

3 Discussion and Conclusion

The fast burning propellant systems based on AP/PDMS and AP/Al/PDMS which are presented here have been formulated and characterized on a basic level up to now. Their thermodynamic performance, the specific impulse, is limited by the formation of silicon dioxide which keeps the combustion temperature constant at its evaporation point of 2863 K. Therefore, the specific impulse even of the aluminized formulations cannot exceed those of AP/HTPB propellants. The values amount to $I_{SP} = 2320$ N s/kg (236 s) for AP/PDMS and to $I_{SP} = 2403$ N s/kg (245 s) for AP/Al/PDMS formulations at an expansion ratio of 70:1. To achieve a good performance it is necessary to work with high solid loadings equal to 83 and 84 mass-%. With a trimodal AP fraction based on AP 200 μm mps, AP 30 μm mps and UFAP with 2 μm mps it is possible to formulate castable propellants with 83% solids based on AP/PDMS and similar ones with 84% solids based on AP/Al/PDMS which ought to

Table 4. Particle size distributions, viscosities EOM, specific impulses, burning rates and pressure exponents at ambient temperature for AP/PDMS and AP/Al/PDMS formulations.

Batch-Nr:		37	43	38	42
Solid loadings	mass-%	83%	83%	84%	84%
AP	2 μm	24.6	23.0	23.1	23.1
AP	30 μm	16.4	9.8	23.1	15.4
AP	200 μm	41.0	49.2	30.8	38.5
Al	5 μm	–	–	6.0	6.0
PDMS silicone resin		17.0	17.0	16.0	16.0
Fe ₂ O ₃	6 m ² /g	1.0	1.0	1.0	1.0
viscosity EOM	(Pa s)	544	440	> 800	> 800
$I_{SP, EQ}$	(N s/kg)	2322	2319	2403	2403
$I_{SP, VOL, EQ}$	(N s/dm ³)	3973	3968	4206	4206
Burning rates in mm/s	2 MPa	19.5 ± 0.1	20.9 ± 0.1	25.1 ± 1.5	23.2 ± 0.1
	4 MPa	33.9 ± 0.6	32.9 ± 0.4	36.9 ± 0.1	37.4 ± 1.1
	7 MPa	48.8 ± 2.7	47.8 ± 0.6	52.9 ± 3.9	49.5 ± 0.5
	10 MPa	62.9 ± 0.4	58.1 ± 0.6	59.5 ± 1.4	58.8 ± 0.4
	13 MPa	71.4 ± 1.1	72.5 ± 2.1	66.6 ± 4.8	70.4 ± 0
	18 MPa	86.9 ± 0.7	84.0 ± 0.7	80.0 ± 0.6	80.7 ± 1.3
	25 MPa	104.0 ± 0.1	100.0 ± 0	98.0 ± 1.8	100.0 ± 2.0
Pressure exponent		0.8 → 0.5	0.65	0.5	0.6 → 0.5

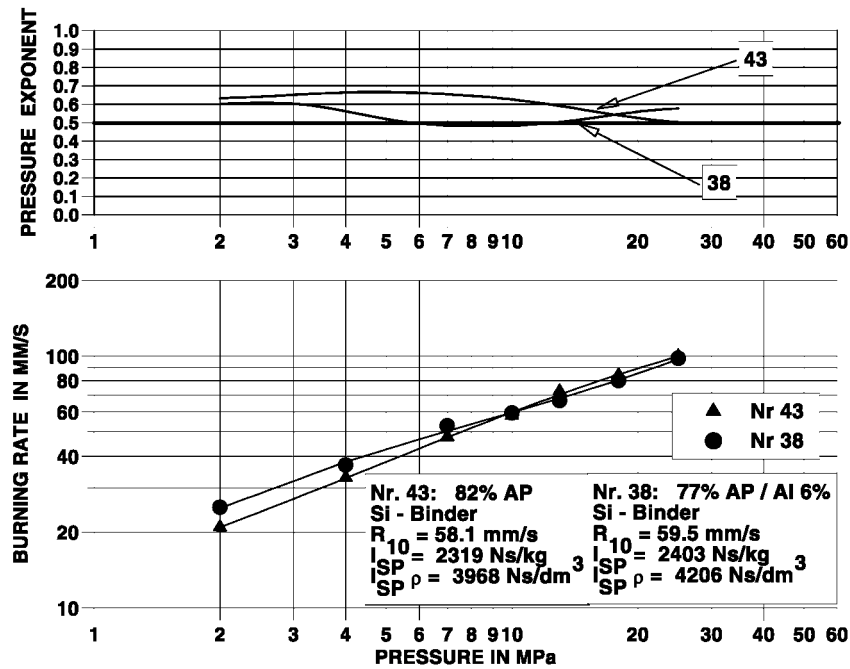


Figure 6. Plot of burning rates and pressure exponents from AP/PDMS and AP/Al/PDMS formulations.

Table 5. Results of basic tests for chemical stability and sensitivity of AP/PDMS formulations

Batch-Nr:		37	43	38	42
Solid loadings	mass-%	83%	83%	84%	84%
AP		82	82	77	61
Al 5 μm		–	–	6.0	6.0
PDMS silicone binder		17.0	17.0	16.0	16.0
Dutch Test (105°C/8–72 h)	(%)	0	0	0	0
Flash point (20°/min)	(°C)	315	321	317	318
Vacuum stability (40 h/100°C)	(ml/g)	0.03	0.06	0.07	0.06
Friction sensitivity	(N)	20	28	20	20
Impact sensitivity	(N m)	7.5	5.0	5.0	7.5
Gap test 50 mm \varnothing	(mm PMMA)	–	0	–	0

be processable under pressure. These propellants exhibit burning rates close to 60 mm/s at 10 MPa and pressure exponents between 0.5 and 0.6.

With flash points over 300°C the chemical and thermal stability of these formulations appears to be very good, probably better than that of other composite propellants based on AP/HTPB or AP/PU systems. Impact, friction and detonation sensitivity are comparable with conventional AP/HPTB systems. Due to the high thermal stability and good elasticity the detonation sensitivity and vulnerability ought to be better, which, however, has to be confirmed in further tests.

At first sight, the fact was puzzling that the glass transition temperature determined by DMA or TMA turns out to lie between -44°C and -60°C and differs when T_G is measured on heating or cooling. Measuring the pure binder polymer reveals a time dependent process of modulus increase close to T_G which might be due to a time dependent partial crystallization of the polymer.

Although siloxane polymers are known for very low glass transition temperatures and freezing points, these effects will be influenced according to the size and variety of alkyl substituents. For polydimethylsiloxane a more or less homogeneous chain structure is covered with a continuous row of small methyl groups. This structure effects the high pyrolytical and chemical stability and is obviously responsible for the comparatively high freezing and glass transition temperature including the phenomenon of partial crystallization and the inhibited time dependent increase of the modulus close to T_G . This effect might easily be overcome when a siloxane polymer with alkyl substituents containing two and more carbon atoms will be used.

In conclusion, overall composite propellants based on ammonium perchlorate and silicone resins offer a special application potential within the class of existing rocket propellants.

As presented here, AP/PDMS systems can be formulated with very high burning rates together with an outstanding

chemical and thermal stability at high temperatures. Good elasticity down to very low temperatures is expected for siloxane polymers with larger alkyl substituents consisting of two and more carbon atoms. The prospective properties of perchlorate and nitrate PDMS formulations like

- Favorable oxygen balance
- Adjustability of high burning rates
- High chemical and thermal stability

make these systems be major candidates for airbag applications and commercialized products. Other potential benefits like elasticity at very low temperatures and low vulnerability, which tend to be subject for further investigations, may find additional applications in weapon technology.

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List of Abbreviations and Symbols

AGARD	Advisory Group for Aerospace Research and Development
Al	Aluminum
AP	Ammonium perchlorate
c^*	Characteristic velocity
c_p	Specific heat at constant pressure
c_v	Specific heat at constant volume
DMA	Dynamic Mechanical Analysis
G'	Storage modulus
G''	Loss modulus
G^*	Complex modulus ($G^* = \sqrt{G'^2 + G''^2}$)
HTPB	Hydroxyterminated Polybutadiene
I_{SP}	Specific impulse (equilibrium flow)
I_{SPVOL}	Volumetric specific impulse (equilibrium flow)
κ	Isentropic exponent (C_p/C_v)
PDMS	Polydimethylsiloxane
PMMA	Polymethylmethacrylate
PU	Polyurethane
ρ	Density
RDX	Cyclotrimethylenetrinitramine
$\tan \delta$	Loss tangent ($\tan \delta = G''/G'$)
T_c	Combustion temperature
T_e	Temperature after ideal expansion
T_G	Glass transition temperature
TMA	Thermomechanical analysis
UFAP	Ultrafine Ammonium Perchlorate (2 μm mps)

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