

Thermal and Mechanical damage of PBX's

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Abstract

At the TNO Prins Maurits Laboratory, considerable effort has been put into the research of energetic materials and their responses to several IM-stimuli like Cook-off, Bullet-fragment impact and Shaped charge impact. Besides the development of highly instrumented test set-ups, computer codes are under development, as a predicting tool.

The response of energetic materials to these stimuli, depends very strongly on the properties of these materials on the moment of event and/or during the event. To understand the mechanisms leading to the response but also knowing the parameters influencing the response, these materials have been submitted to laboratory scale experiments.

For impact modelling, the mechanical properties have been measured as a function of the strain rate (0.001 m/s-5m/s,) in the temperature range from -60 up to 60°C. Besides tensile-strength testing, relaxation testing for the same temperature range, has been performed. From these experiments the Poisson ratio and a time-temperature relation has been set-up to obtain mechanical properties of the explosive for intermediate and high strain rates impact, at ambient temperatures, in experiments like the gas gun and bullet/fragment impact experiments. Also optical microscopy as well as scanning electron microscopy has been used to examine the samples after impact. The information has been used to set-up and improve the erosion/damage models for the Autodyn calculations.

For thermal damage research, mechanical properties have been measured as a function of the temperature, up to 160°C, as well as degree of decomposition. These parameters are needed in the cook-off computer model to couple the thermal-chemical part of the code to the mechanical part. An overview of the research on the impact related as well as the thermal related research will be presented in this paper.

Introduction

After some terrible accidents in the late sixties, it became clear that less sensitive munition was needed to avoid these kinds of accidents in the future. The search for less-sensitive explosives started at that time and finally resulted in the World-wide Insensitive Munitions search of IM.

Although, in the beginning nobody believed that an explosive could be less-sensitive, the idea of the so-called Plastic Bounded eXplosive (PBX), disproved this thought. PBXs were and still are used in pressed as well as in cast-cured mixtures of explosives. Also, the use of bimodal mixtures instead of trimodal mixtures, removing the large crystal particles up to 1mm, lead to less sensitive explosives. However, insensitivity and large explosive power do not go hand in hand. The use of a plastic inert binder decreases the sensitivity to all kind of threats, but also decreases the performance of an explosive. However in some applications, like a shape charge, high energetic composition are needed for maximum performance.

Therefore, the search for new less sensitive explosive, like TATB, NTO, FOX7 and CL20, is still on his way, as well as the search for energetic binders to compensate for the decrease in energy in the explosive.

International, especially in many Nato countries, IM-policies have been set-up, giving a definition of IM and how IM should be implemented in the purchase of new munitions and platforms. Also the STANAG 4439 [5], with the AOP 39 [6] were set-up, with the prescription how to perform IM testing the interpretation of the test results. However, there is still no agreement on the test protocols, in the international IM policy. Some countries think that Threat Hazard Assessment for a munitions life time cycle is necessary to decide which type of IM testing is needed, while others think that all IM tests should be performed. Some, countries have introduced a so-called IM waiver system while other have different classes of IM munitions. Another item is statistics. Is doing one or two tests for all the different IM tests enough to assess the IM-ness of a munition item; because statistically seen, two tests is almost as good as no test. Should laboratory scale testing and simulation be a part of assessing the IM-ness of a munition item?

Already, several Nato countries have decided that all new munition purchased should be more or less “IM–developed” and more countries will follow in the future. IM testing on itself not only is expensive but also gives pollution to ground and air. Furthermore, the ultimate IM explosive, very energetic and extreme insensitive is still not developed an more research is needed to understand why an munition item is (in)sensitive and which parameters have a major influence on the response of the item to the IM testing.

For many years now, TNO-PML laboratory is doing research on IM, understanding the mechanisms that lead to the response and determining the parameters that have a major influence on the sensitivity. Tests have been developed to analyse the mechanisms and computer codes have been and are under development to simulate the processes. For all the mechanisms of the different IM tests, mechanical properties and damage play a major role in the process that lead to a detonation response (type I or II response) or a less violent reaction (type III, IV or V response). Also, others have already investigated the influence of damage on the sensitivity of composites [1, 2, 3, 4, 7].

The mechanical properties play a major role in the mechanisms of SDT, DDT and XDT, all leading to a detonation. The SDT (Shock-to-Detonation Transition) phenomenon is rather well understood, as the dependence of parameters like the critical diameter, shock Hugoniot, impact velocities and dimensions and properties of the Impact threat. Although it is understood that ignition sites play a major role in shock sensitivity, a quantitative relation between material properties, as porosity, amount of inclusions, damage etc., and shock sensitivity is still not well defined. In DDT and XDT process, material properties like mechanical properties and damage play an even bigger role (in such a way that it can lead to a detonation, on one side, or no reaction at the other side, with a range of intermediate reactions in between.)

Several years ago, research started in to the mechanical properties and damage, influencing the sensitivity of an explosive. After the preliminary studies to come to a research explosive like HU-43/44, the first results of the damage research are given. With the knowledge from the first results, a new approach has been set-up, in the attempt to find a quantitative relation between mechanical properties, thermal as well as mechanical damage and the sensitivity of

an explosive to IM-like threats. The first findings of this new approach will be presented as well.

Production of research PBXs

Several RDX and HMX-PBXs were produced for testing purposes, like shock initiation, cook-off and the influence on of crystal size and quality on the sensitivity of the explosive. First compositions have been made with a solid load of 85wt% RDX. The mechanical properties were varied by means of a chain extender and by changing the amount of plasticizer (IDP). However casting of these composites was almost impossible.

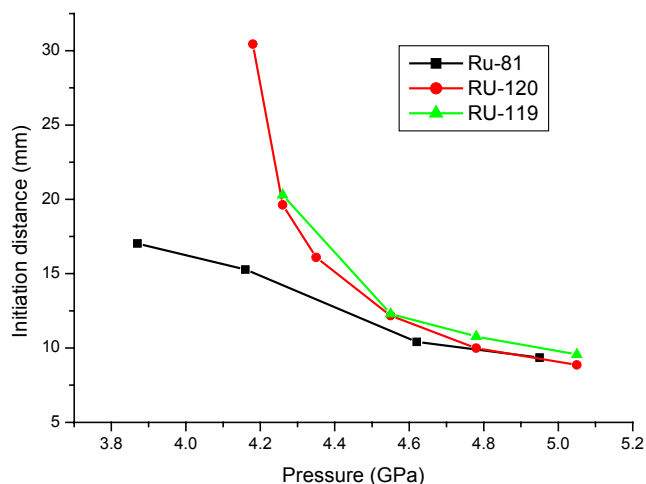


Figure 1: An overview of the Gap-test results. The compositions Ru-119 and Ru-120 with chain extender in comparison with the reference Ru81.

Then, another series of RDX-PBX was made with only 80%wt solid load and a binder system with chain extender and about 15 and 30% IDP. From this series, two compositions were selected that were castable, RU119 and RU120, and were tested in the friability test [9], on shock sensitivity and cook-off and compared with the reference RU 81 (85%wt RDX and no chain extender) and HU-28 (also 85%wt solid load but HMX). The newly developed PBXs were rather stiff compared to the reference. This seemed to have a positive influence for the results in the friability test [9]. The pressure derivative in the closed vessel test after impact of a sample on a steel wall of about 150 m/s, dropped to a low value of 5.8 MPa/ms for the RU120, to 2.2MPa/ms for the stiff RU119. However a comparison to an 80% solid load RDX-PBX was not possible. For the cook-off test, no real difference could be found, for the shock sensitivity in a flyer impact test a small change was shown [8].

Because it has been found empirically that in general HXM-PBX responds better in a cook-off test than RDX, HMX is used in the next series of compositions. This time the mechanical properties are varied systematically controlling the molar ratio of the chain extender (Trimethylhexanediol) and the prepolymer hydroxyterminated polybutadiene (HTPB). These ratios were respectively, 2.5, 1.25, 1.75 for the HU-39, HU-40 and HU-41. For the final composition HU-43 or HU-44 (different batch of HMX) a ratio of 1.5 was selected, based on the Youngs moduli and the mechanical damage observed in the friability test. The bimodal mixture of HU-43, has a main particle size of two composing HMX grades being about 16 and 350 micron and a density of about 1.57 gcm^{-3} .

Mechanical properties at high strain rates

Before laboratory-scale testing, the mechanical properties of HU-43 have been measured extensively by means of a Zwick 1445 and a Zwick 1852 draw-bench. With the Zwick 1445, the HU-43 samples have been elongated at a speed of 50 mm/min, at temperatures of -60 , -40 , -20 , 20 and 60 °C. With the Zwick 1852, the HU-43 samples have been elongated with speeds of 0.001, 0.01, 0.1, 1 and 5 m/s, and at temperatures of -40 and $+20$ °C.

In figures 2a and 2b one can see the stress-strain curves of HU-43 at different temperatures and strain rates respectively.

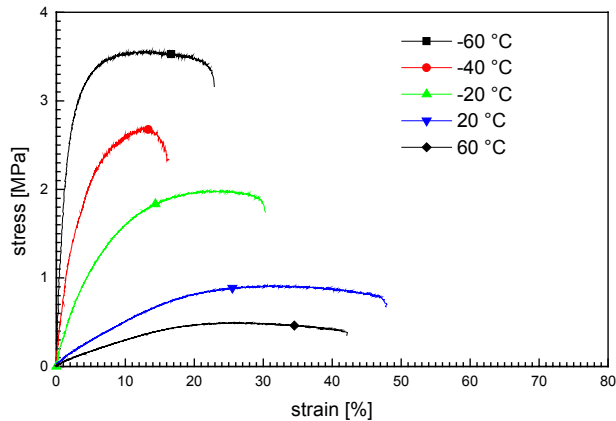


Figure 2a: Stress-strain curves of HU-43 at indicated temperatures, measured with the Zwick 1445 and sample elongation rate of 50 mm/min.

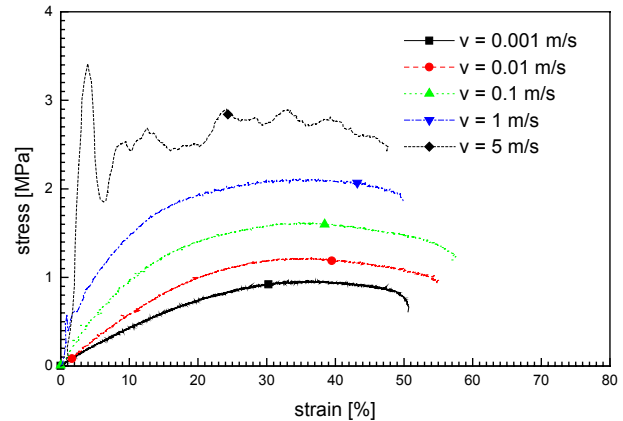


Figure 2b: Stress-strain curves of HU-43 at indicated sample elongation rates, measured with the Zwick 1852 at 20 °C.

The Young's modulus decreases from $2.1 \cdot 10^8$ Pa to $4.2 \cdot 10^6$ Pa with an increase in temperature from -60 °C to $+60$ °C. The maximum attainable deformation of HU-43 before rupture occurs strongly depends on temperature. By variation of deformation rate, no strong influence on maximum strain is observed. The data in figure 2b, corresponding to a sample elongation rate of 5 m/s show the limits of the rheometer, These data are not used in further analysis. At 20 °C the Young's modulus increases from 6.8 to $29.5 \cdot 10^6$ Pa, with an increase in sample elongation rate from 0.001 to 1 ms^{-1} . The strain rates corresponding to 0.001 and 1 ms^{-1} are 0.0125 and 12.5 s^{-1} .

Also relaxation testing has been performed on HU-43/44, in the range of -60 °C to $+60$ °C. The relaxation test results have been analysed and shifted to obtain the so-called temperature corrected master-curve to have a relation between the time and temperature. The temperature corrected master-curve for HU-43 is shown in figure 3a. In this graph, also Young's moduli results at different temperatures (see figure 2a and 2b) have been incorporated, using the shift factor of figure 3b. A good correlation between the relaxation results and the drawn-bench results is shown. With these result the material properties of HU-43/44 at very high deformation rate can be obtained. These data form the bases of hydrocode simulations of the gas gun experiment in which material is damaged by impact against a steel plate at 150 m/s (first part of the friability test) and of future simulations of bullet and fragment impact tests with this material.

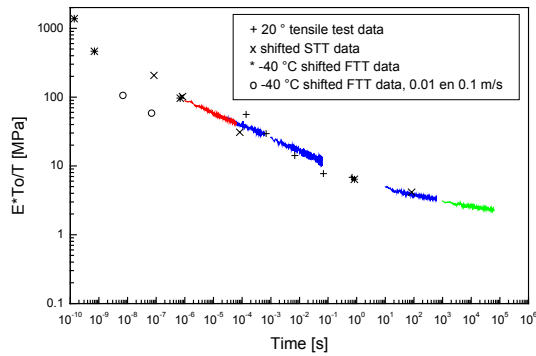


Figure 3a: The temperature shifted master curve of HU-43 together with the values of the tensile strength test

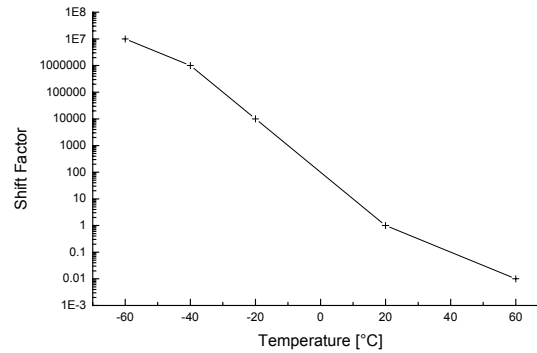


Figure 3b: The shift factors as a function of the temperature for HU-43

Mechanical damage after impact of the gas gun experiment

After mechanical characterisation, HU-43 samples of 9 grams and 18 mm diameter were damaged by means of the gas gun against a steel plate. Velocities in the range from 91 up to 154 m/s were used. The samples were inspected in three ways, by means of visual inspection, observation of the actual impact process with a high-speed film recording and analysis with scanning electron microscopy.

VISUAL INSPECTION

Collecting the fragments of Hu-43 after impact on steel and looking at the damage visually has proven a very simple way to monitor the sensitivity of the explosive substance to deterioration under the effect of an impact. In this way the composition of HU-43 has been established by improving the resistance to mechanical deterioration, by systematic variation of the molar ration of the chain extender as mentioned before.

In figure 4, the damage of HU-43 by impact at different velocities is shown. At 91 ms^{-1} impact velocity one can see that the cylindrical charge, although still one piece, has become larger in diameter at the impact side. At 154 ms^{-1} quite a number of tiny fragments are formed at the impact side, and they appear to 'peel off' at the outer radius, leaving a cylindrical core in the centre. The mass lost in fragments (measured as the difference of initial mass, i.e. 9.0 ± 0.1 gram, minus the mass of the largest piece collected after the impact) equals 0.0, 0.1, 2.0 and 3.8 gram for impact velocities of 91, 110, 129 and 154 ms^{-1} , respectively.



Figure 4: Fragments of HU-43 collected after firing bare cylinders on a steel plate at indicated velocities.

HIGH SPEED FILM

With the 16 mm high speed film camera HYCAM, maximum 40000 frames per second, the impact of HU-43 on steel has been observed. An example is shown in figure 5, with 145 ms^{-1} impact velocity. Background light is used to create a shadow picture, and the optical axis of the camera is aligned parallel to the steel plate. In the picture below the left boundary of each frame, is therefore the steel plate and the explosive cylinder is coming from the right. In the latter frames one can also see the sabot. Extensive deformation of HU-43 is observed at impact, followed by fragmentation at the impact side, resulting in a fragment spray that blurs the location of the steel plate and eventually the location of cylindrical charge. Furthermore, a lot of energy must be dissipated in the intense fragmentation, and it takes a relatively long time before the remaining HU-43 core rebounds.

At lower impact velocities the deformation process (elastic and plastic) can be observed very well, as well as the time between impact and rebound.



Figure 5: Impact of 9 gram, 18 mm diameter HU-43 cylinder on steel, at an impact velocity of 145 ms^{-1} .

SCANNING ELECTRON MICROSCOPY

Pristine and damaged HU-43 samples have been examined with Scanning Electron Microscopy (SEM). In figures 6a and 6b the electron micrographs are given for samples damaged at 92 and 145 ms^{-1} respectively. Even though the sample of figure 6a has not fragmented in the impact experiment, for reference see figure 4, one can see that the HMX crystals show a lot of damage, and are broken up into smaller crystals. The same phenomenon is observed at the rear side of the sample in figure 6b.

In figures 7, the binder corresponding to the sample with a 92 ms^{-1} impact velocity, is examined. The magnification is larger compared to figures 6. Near the rear side no difference with the pristine sample is observed. However, in half the length of the sample measured from the impact side, elongation of the binder, and debonding of HMX from the binder, is

observed. This corresponds to that part of the sample that is deformed plastically, see figure 4. The location of extensive deformation can be seen even more clearly in the high-speed film recordings of figure 5.

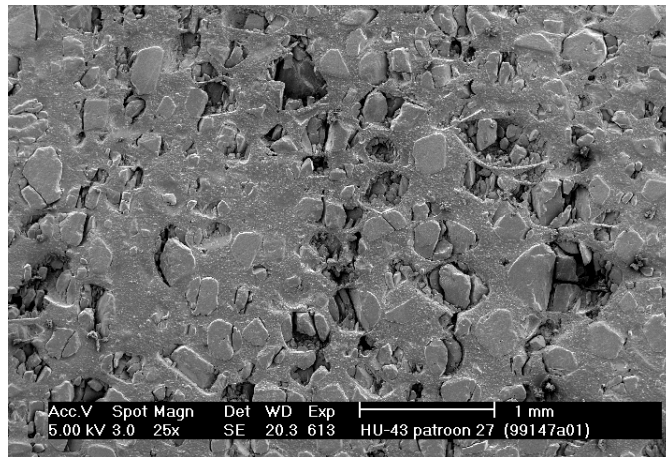


Figure 6a: SEM of HU-43 sample near the impact side, impact velocity is 92 ms^{-1} .

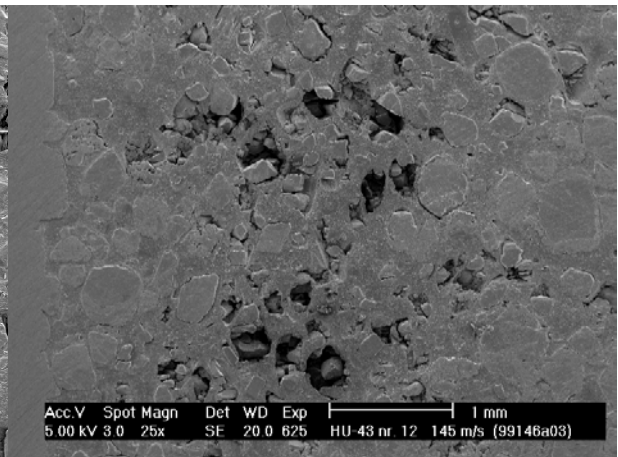


Figure 6b: SEM of HU-43 sample near the side opposite to impact, impact velocity is 145 ms^{-1} .

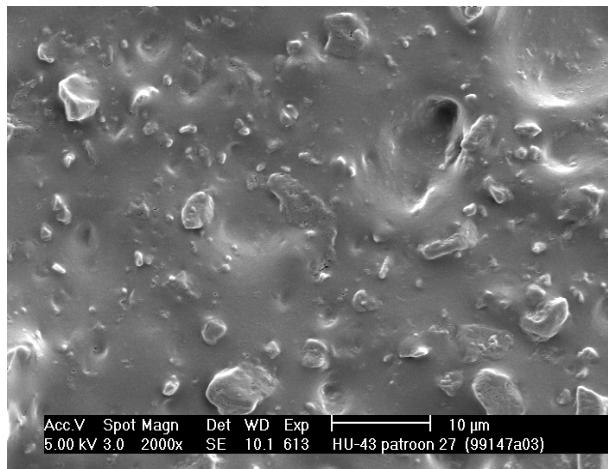


Figure 7a: SEM of HU-43 sample near the rear side, impact velocity is 92 ms^{-1} .

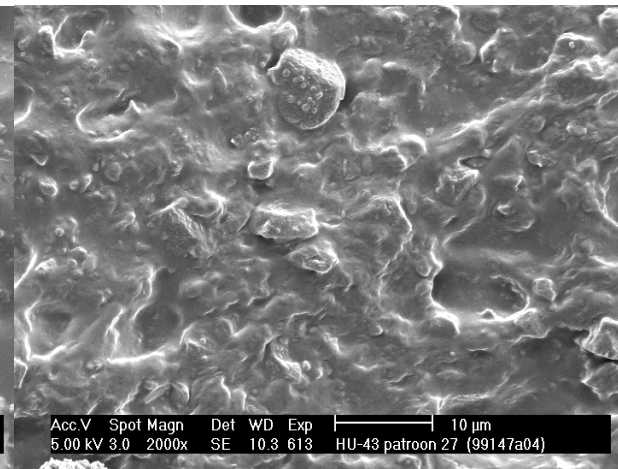


Figure 7b: SEM of HU-43 sample near the impact side, impact velocity is 92 ms^{-1} .

SHOCK INITIATION

The shock initiation thresholds of pristine and mechanical damaged HU-43 have been investigated with flyer impact. For the determination of the initiation threshold a series of about 8 samples is needed by means of a gas gun experiment. The damaged samples have been prepared by impact at about 150 ms^{-1} . The initial mass of those 8 samples is 9.00 ± 0.08 gram, the impact velocity of the samples is in the range 146.0 to 151.9 ms^{-1} , and the mass of the remaining core is in the range 4.70 to 5.58 gram.

The rear side of the damaged sample is impacted by a $125 \mu\text{m}$ thick Kapton foil. The thickness of the flyer is relatively small, the mean particle sizes of HU-43 are 16 and $350 \mu\text{m}$. It means that if a difference in shock initiation threshold with this flyer thickness is observed, the difference in shock initiation in a gap test will probably be even more distinct.

In order to observe the shock-to-detonation transition, or the failure of initiation, which will depend on the flyer impact velocity, a so-called fibre optic probe is inserted in the sample [10].

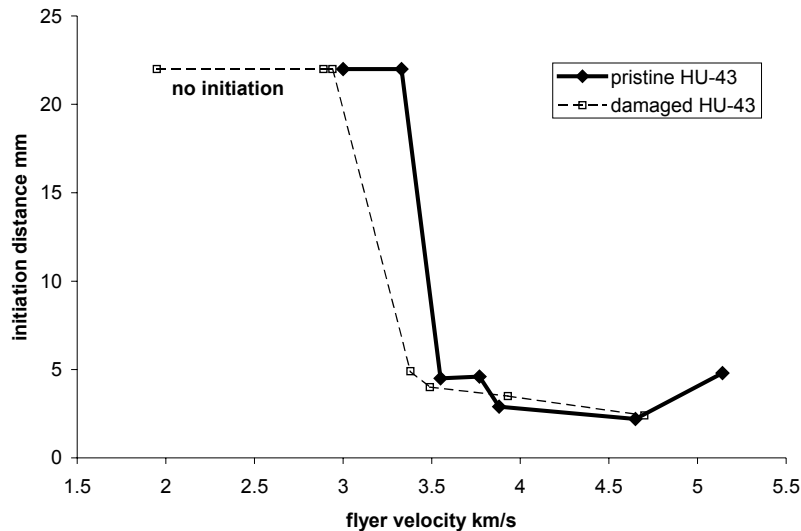


Figure 8: Initiation distance vs. flyer impact velocity of pristine and damaged HU-43.

In figure 8, the initiation distance of pristine and damaged HU-43 is given as a function of the flyer impact velocity. The initiation threshold corresponding to pristine and damaged HU-43, and impact of a 125 μm Kapton flyer is between 3.33 and 3.55 kms^{-1} , and between 2.94 and 3.38 kms^{-1} . The influence of shock sensitivity of damage by low velocity impact is therefore significant.

Quantification of damage, a new approach

The amount of induced damage of an energetic material, due to a kind of threat like bullet impact, and the response of the material after impact, strongly depends on the mechanical properties of the material, before as well as during the impact. Damage can be induced by means of a thermal threat (cook-off), a mechanical threat (bullet impact) or a combination of both (hot fragment impact). How much damage results in the specific response of the explosive is still unknown, to describe what damage really is, is even harder. Of course damage is a combination induced porosity, change of the binder properties, deformation of the binder, debonding of the binder from the crystals, crystal cracks, but also phase changes of the crystal like in HMX at higher temperatures. All these items need to be implemented in an attempt to set-up a damage model.

However, the amount of damage can also be described by means macroscopic parameters as pressure derivative in a close vessel test (for thermal damage) or the change of amount of work under a stress-strain curve for mechanical damage. TNO-PML has set-up a test plan to relate the amount of damage, thermal as well as mechanical damage, to the response in an IM like threat, like a shock initiation test or fragment impact test. Furthermore, the thermal damaged will also be related to the mechanical properties of the material compared to the pristine material.

For the thermal damage, the material is set at elevated temperatures of up to about and 175°C for several hours and tested afterwards in a Flyer impact test (SDT), a fragment impact test. The mechanical properties have been measured by means of a Gas dilatometry test [4]. To quantify the amount of damage, closed vessel testing will be performed.

To induce a quantified amount of mechanical damage, a Load-Unload-load cycle experiment is performed on a explosive in a draw-bench. Due to the foregoing load, the material has lost a part of its reinforcement (reduced stiffness). Therefore the material will follow a slightly changed curve. The area in between of the two consequent curves defines the amount of damage in the material. (see figure 9). After damage shock initiation testing will be performed, closed vessel testing to related the mechanical damage to the closed vessel parameters and hopefully fragment impact testing.

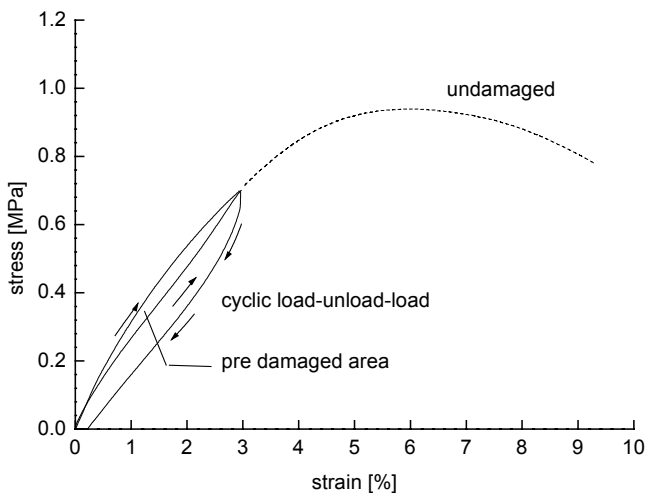
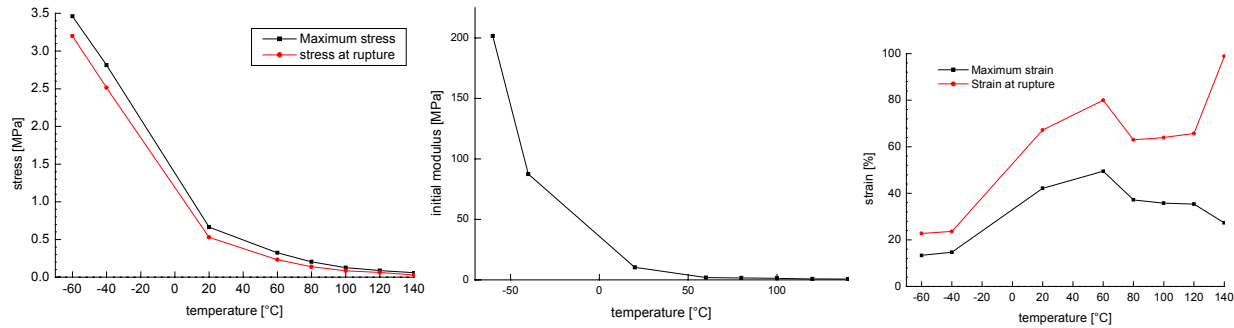


Figure 9: A load-unload load cycle on an HMX-PBX, giving a quantitative amount of mechanical damage to a sample.

First results of the new approach

CHANGE OF MECHANICAL PROPERTIES AT HIGH TEMPERATURES

It was shown already in figure 2a, that the strength of a composite like HU43 drops significant at elevated temperature. To see how the mechanical properties change as a function of the temperature, tensile strength tests have been performed up to a temperature of 140°C. For this series, the samples were held at a constant temperature for 1 hour before the tensile strength test was performed. The results are shown in the figures 10. From the figures it is clear that the mechanical properties drop significant and with the initial modulus almost dropping to zero at temperature at about 120°C. Also the strength drops to very low values. However the strain, first increases up to temperature of about 60°C. Above this temperature the strain at maximum strength and at rupture both decrease. However, above a temperature of 120°C the strain at the maximum strength still drops while the strain at rupture still increases.



Figures 10 a, b, c: Mechanical properties of HU44 after 1 hour at indicated elevated temperatures. In figure a, the maximum stress and stress at rupture is given in, b, the initial modulus and in figure c, the maximum strain and strain at rupture.

GAS DILATOMETRY TESTING (GDM)

Gas dilatometry testing, in combination with tensile strength testing, gives a good insight of the damage process of HTPB-binder systems and is also used for rocket propellants. As also described extensively in the reference [4], one can divide the damage process into 4 stages. First phase, the visco-elastic response phase, second phase, the polymeric chain failure, third phase, dewetting (dilatation) and in the last phase, macroscopic failure. A composite possesses a highly complex, time-temperature dependent behaviour. Compared to the polymeric binder, the solid load particles result in a reinforcement of the material (increased modulus). Failure of the composite, initiates early in the stress-strain history. Stress concentrations in the vicinity of filler particles, result in a local failure of the polymer chains (polymeric chain failure, slippage and polymeric reorientation). After sufficient micro-structural damage, vacuoles form and grow with continued straining. Depending on the nature of the polymer-filler bond, this vacuole can spread to the interface and unbond the particle (dewetting). In case of a very strong bond between the binder and the filler, vacuoles appear to stay entirely in the polymer phase. After further growth of the vacuoles, cracks form resulting in a failure of the composite. In fast compression testing or a bullet or fragment impact test also cracking of the explosive crystal occurs, as has been shown already in the photographs of figure 6 and 7. This has not been part of this study yet.

In a load-unload-load cycle, damage of phase 2, the polymeric chain failure, shows up at rather low strain levels (see figure 9). The forming of vacuoles starts at higher levels, as is shown in figure 11b. However, a direct partial rehealing of damage is possible in this kind of composites. A recovering time of about 2 weeks at 50°C, can even lead to an almost virgin composite.

To analyse the influence of an elevated temperature on the damage process, samples of HU44 were held at a temperature of 120 and 160°C and tested afterwards in the GDM. The results were compared with the pristine material. The results of the GDM test are shown in figure 11 a and 11 b. From figure 11a, one can see that the initial modulus changes slightly compared to the pristine material, resulting in a softer material and up to a strain of about 10%, no real differences are shown between the 120 and 160°C samples. The maximum strength has changed just a little. Looking at figure 11b, no real change in the dilatation behaviour can be observed between the pristine and damaged sample at 120°C. However, for the sample damaged at 160°, the curve almost immediately starts rising, indicating that damage in form of vacuoles were formed during the heating process, probably an increase of the porosity.

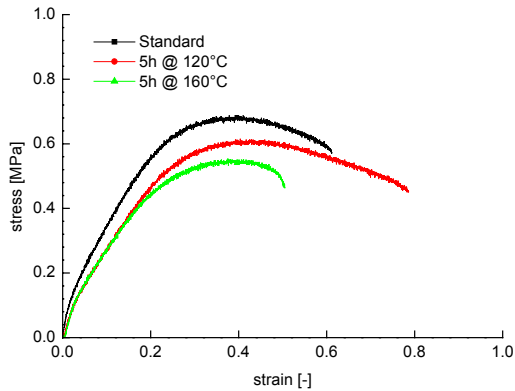


Figure 11 a: The Strain-stress curves in a GDM of samples held at a temperature of 120 and 160°C, for 5 hours, in comparison with pristine material.

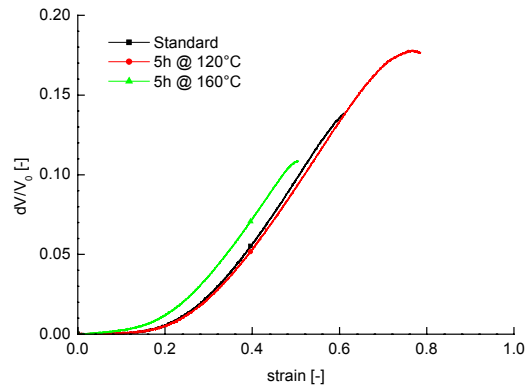


Figure 11 a: Dilatation behaviour of samples held at 120 and 160°C, for 5 hours, in comparison with pristine material.

THERMALLY DAMAGED SAMPLES

For the preparation of the future, closed vessel, Flyer impact and GDM testing, sample were thermally damaged at several elevated temperature. The Bam Friction and Fallhammer test [9] of a sample heated at 170°C already indicated that the material is already more sensitive than the pristine material. In figure 12a, the sample are shown and compared to the pristine material. It is clear that an oxidation process has been taken place, shown by the darkening of the samples. The higher the temperature the darker the sample became afterwards. Compared to the pristine material the samples that were held at 165°C, for 5 hours, lost about 0-0.05 grams in weight, of 170°C around 0.1 grams and at 175°C even 0.25 grams. Also the form of the cylinders changed due to the heating. The GDM samples also became longer compared to the pristine material. In the magnification of the sample at 175°C (figure 12 b), cracks are shown indicating a high level of damage has occurred already during heating. This kind of damage will probably have a major influence on the sensitivity of the explosive, which will be revealed the experiments performed in the next test series.



Figure 12 a: Thermally damaged samples of temperatures of respectively 165, 170 and 175°C (for 5 hours) compared to pristine material



Figure 12 b: Magnification of one of the samples showing cracks after thermal heating.

Discussion and Conclusions

The research of mechanical properties of HMX and RDX PBXs, in relation with thermal as well as mechanical damage of the last few years has been summarised in this paper. Already from the first test series with RDX-PBX, it could be concluded that the change of mechanical properties had a major influence on the sensitivity of the explosive, in particular in the friability and the Gap test. From these results, a new reference explosive, Hu-43/44 was developed for research purposes and mechanically characterised. Mechanically damaged HU43 samples were submitted to visual as well as SEM inspection and also to shock initiation testing. Several types of damage were found like, deforming of the binder and reorientation (Polymeric chain failure), vacuole forming and debonding of the binder from the crystal and crystal fracture. Also mechanical damage had a somewhat influence on the shock sensitivity. In a new programme, a new approach has been started, in an attempt to quantify the amount of damage and find a relation between amount of damage and the sensitivity of an explosive. The first results indicate that especially for mechanical damage, rehealing occurs in this type of binder-systems. For thermal damage probably oxidation processes start at temperature of about 150°C, resulting in a more irreversible type of damage. Already at 120°C for 5 hours, a slight change of mechanical properties was found but not in a real negative way. At a temperature of 160°C, for 5 hours, the dilatation test results indicated an increase of vacuoles. The future experiments in this programme, like closed vessel, GDM, Shock initiation and fragment impact testing, will reveal in what way this has increased the sensitivity of the explosive. The same will be done for the mechanically damaged material.

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