Quantification of thermal and mechanical damage in PBX's

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Abstract

TNO Defence Security and Safety, has conducted research in energetic material response to several Insensitive Munitions (IM) stimuli like cook-off, bullet-fragment impact and shaped charge impact. The response of energetic materials to these stimuli depends strongly on the properties of these materials at onset and during an event. In general, all types of laboratory-scale experiments are performed to understand the mechanisms and the parameters influencing the response of explosives and assessing the increase in sensitivity of the explosive. However, the *amount* of damage in relation to the sensitivity increase is unknown. Therefore, TNO introduced a new approach where a PBX is mechanically or thermally damaged and the extent of damage is quantified. In laboratory-scale tests, the increase of sensitivity (e.g. shock sensitivity) due to the damage is assessed and compared to the pristine material response.

In several test series thermal damage was induced by keeping a 9 grams PBX samples at elevated temperatures in the range from 160ºC to 175ºC for 5 hours, or damage was mechanically induced, by compression up to 10, 20, 30 and 60% strain at different compression rates. The damage was quantified by means of closed vessel testing assessing the increased burning area and the maximum pressure derivative, but also by determination of the area in between the load-unload cycle in the compression test. Pure shock initiation testing (flyer impact) and bullet fragment impact (SDT, DDT) tests of confined and unconfined samples were carried out to assess the sensitivity increase.

This way a quantitative relation between damage and sensitivity increase is established, leading to a better understanding of this phenomenon. In the future it is aimed to establish a macroscopical damage model for computer purposes and an improved initiation and growth model that takes damage in to account in shock initiation modelling.

1. Introduction

TNO Defence Security and Safety conducts research in IM, to understand the mechanisms that lead to the response of explosives and to determine the parameters that have a major influence on sensitivity. Several types of analyses and laboratory scale experiments are used to investigate the mechanisms playing a role and computer codes are under development to simulate these processes. For all the mechanisms of the different IM tests, mechanical properties and the amount of 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). Others have also investigated the influence of damage on the sensitivity of composites [1-5].

The material properties of an energetic material 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 relatively well understood. In DDT and XDT process, material properties like mechanical properties and damage that occurs play an even more important role (in such a way that it can lead to a range of reaction behaviour). Although it is understood that ignition sites play a major role in shock sensitivity of explosive materials, finding a quantitative relation between material properties, amount of porosity, inclusions, voids, damage, etc. compared to shock sensitivity changes is still difficult.

2. Background: Quantification of damage

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). Damage leads to a specific response of the explosive that at this time is not well understood. Damage is, among other things, a combination induced porosity, change of the binder properties, deformation of the binder, debonding of the binder from the crystals, crystal cracks, and phase change of the crystal material. All these effects need to be verified in an attempt to set-up a damage model. The amount of damage can also be determined by macroscopic measurement such as pressurisation rate in a closed combustion bomb (for thermal damage) or by the change of amount of work under a stress-strain curve (for mechanical damage).

In this approach the two PBX's are mechanically or thermally damaged and the extent of damage is characterised by the change in mechanical properties and by a change in pressure derivative in a closed vessel, changes in burning area etcetera. The sensitivity change is assessed by means of laboratory scale testing like flyer or fragment impact testing. In this way a relation between pristine and damaged materials and sensitivity is established. This should finally lead to a better understanding of damage and to a quantified damage model for implementation in computer codes. Figure 1 shows the assessment of the damage research.

In this program two different types of HMX-based PBX's have been used. An 80wt%, bimodal HMX mixture and an HTPB based binder system with a so-called chain-extender Trimethyl-hexaandiol (TMHD), with batch ID HU 43/44 (depending on the HMX batch) and an 85wt% bimodal HMX mixture with a 15wt% HPTB-based binder system (HU45).

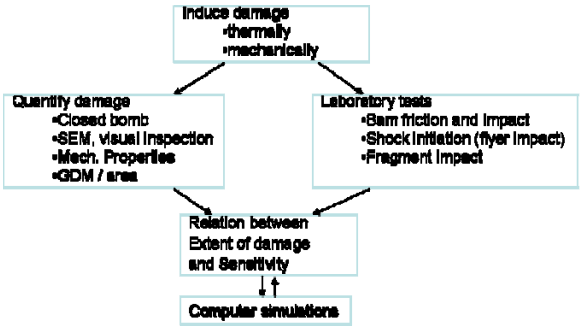

Figure 1. Assessment of damage research.

2.1 Burning Area Increase

Damage can be quantified in many different ways. As a measure of damage, the pressure derivative of a closed vessel test was used or the area under the curves of

a load-unload cycle in a compression test. However, having in mind that burning reactions are part of the mechanisms to the certain response, a parameter as the burning area increase seems to be a much better measure for induced damage. With the knowledge, experience and computer codes build in the gun propellant area; a method is developed to obtain an increased burning area as a measure of damage in explosives [6, 7]. Figure 2 shows an example of the burning area increase of a damaged sample in comparison with a pristine sample.

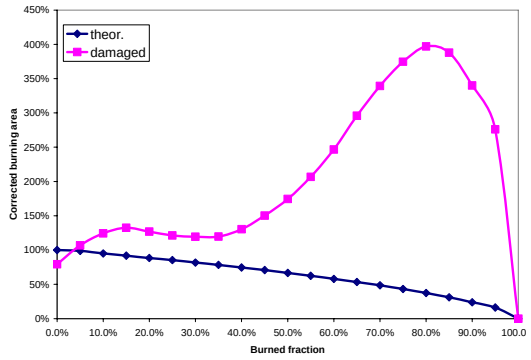

Figure 2. Burning area as a function of the burned fraction for a pristine and damaged sample.

The combustion rate, or gas production rate, of a propellant sample can be determined by closed vessel experiments. In the case of gun propellants the gas production rate can be expressed in terms of the dynamical vivacity, L [6]:

$$L = (dP/dt) / P / P_{max}$$

in which P is the pressure at any time t and P_{max} the maximum pressure determined for an experiment. L is mostly given as a function of P/P_{max}.
The combustion rate depends on the linear burning rate, r, and on the size of the surface area, which is determined by the geometry of the sample. The linear burning rate of propellants depends on the pressure and is generally described by Vieille's law:

$$r = \beta \times P^{\alpha}.$$

In the case of this study the surface area of the cylindrical pristine samples can be calculated as a function of the conversion, which is assumed to be proportional to P/P_{max}, which can be corrected for the pressure generated by the igniter material. From the dynamical vivacity and the surface area of these samples, the linear burning rate can be calculated. The pristine samples are assumed to be non-porous.
For damaged samples the dynamic vivacity is determined as a function of P/P_{max}. Using the equation of the linear burning rate as a function of P, which is obtained for the pristine sample, the burning surface area of the damaged samples can now be calculated as a function of P/P_{max} or burned fraction.
In the process of determining the burning properties the choice of closed vessel, is important to obtain the best results.
It should be noted that porous propellants may show a burning behaviour that differs from non-porous propellants. The apparent linear burning rate of propellants may be increased due to porosity above certain pressure levels. This leads to increased

vivacities above the pressure level at which the flame is forced into the pores of the propellant sample [7]. This may result in an increase of the pressure in the pores, depending on their size. This means that the calculation of the burning surface area of damaged samples using the described method may not always be fully correct. Nevertheless a strong increase of the vivacity above a certain pressure level surely indicates qualitatively the presence of porosity.

3. INDUCED DAMAGE

3.1. Mechanical damage

The work presented in this article builds up on the work presented in earlier paper, which concentrated on e.g. friability testing and damage, and included also numerical modelling of the event [5, 9]. From these results it became clear that compression testing is much more valuable than normal tensile strength testing, certainly when gas gun test results have to be compared with mechanical test results. For both tests the maximum pressure derivative of the closed bomb test after burning of the damaged samples can be compared. This is interesting, because much is know on the relation between a DDT reaction in a bullet impact experiment and the results of the friability experiment [10] (gas gun experiment followed by a closed vessel experiment).

In several test series mechanical damage was induced by compressing HU44 samples at various compression velocities up to 10%, 30% or 60% strain, thus creating various damage levels. The extent of the damage was obtained by means of flyer impact testing (shock sensitivity) and friability in combination with changes in burning area by closed vessel bomb testing.

To induce a *quantified* amount of mechanical damage, a Load-Unload cycle experiment, is performed in a tensile tester. HU45 samples were mechanically damaged by compression up to 10% and 20% strain, and were compressed by either a single cycle or a double cycle. Due to the foregoing load, the visco-elastic/plastic material has lost a part of its reinforcement (reduced stiffness) and the material exhibits hysteresis behaviour. The area in between the two consequent loading curves defines the amount of damage in the material. After mechanical damage, closed bomb testing is performed, to relate the mechanical damage to the closed vessel parameters.

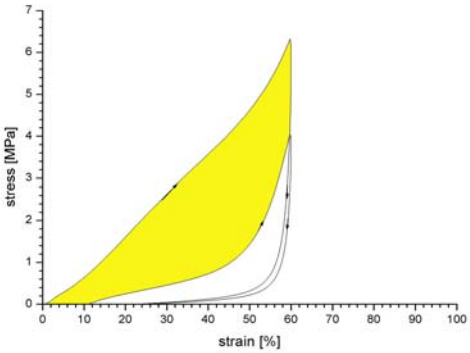

Figure 3. Area between two compression cycles, indicating the extend of damage.

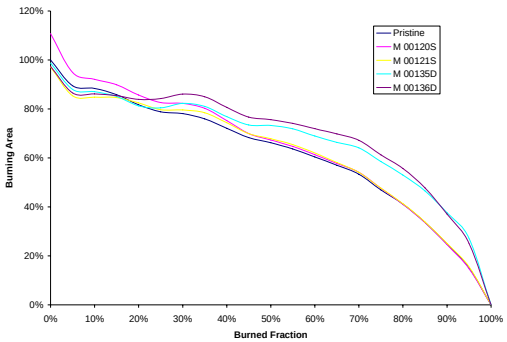

Figure 4. The calculated burning areas of the pristine, single and double cycle damaged HU45 at 20% compression.

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Figure 3 shows the result of a cycled compression test of HU44 sample up to a 60% of the length at a rate of 50 mm/minute. Due to the first cycle, the induced damage is of such a magnitude that the sample lost most of its strength at comparable strain levels. The calculated toughness, energy per unit of volume is the very large area in between the two lines (darkened area) and has an average value of 1.14 MJ/m^3. Figure 4 shows the calculated burning areas of the pristine, single and double cycle damaged HU45 samples at 20% compression. The average values for the energy per unit of volume were 0.05 MJ/m^3 for the 10 % compression and 0.285 MJ/m^3 for the 20% compressed samples (both at 500mm/min).
In fast compression testing or bullet/fragment impact tests, cracking of the explosive crystal occurs. In a load-unload cycle, damage by the polymeric chain failure, occurs at rather low strain levels. At higher compression levels, debonding starts and even crystals cracking start to occur at a certain level. Probably in the second load-unload cycle in the compression test, more crystals are cracked after resettling.

3.1.1 Gas gun experiment

The standard friability test as described in the UN manual is used to determine the effect of impact on an EIDS candidate [10]. The test is split into two phases. In the first phase, a test sample of 9.0 grams has to be projected against a steel plate at a velocity of about 150 m/s. In the second phase, this impacted sample is tested in a manometric bomb (a closed vessel) to determine the average maximum pressure rise rate, $(dp/dt)_{max}$, of three tests, which is not allowed to be more than 15.2 ± 0.2 MPa/ms. In case this value exceeded, a DDT reaction in a bullet impact test is more likely to happen.

So, for comparison reasons, a series of gas gun experiments was performed with velocities in the range of 56-85 m/s. In this range, the sample does not break apart; damage is induced inside the sample. Also for these samples, the extent of damage was obtained by means of the closed vessel parameters.

Figure 5 shows the pressure derivative of the HU44 samples as a function of the gas gun velocity. In the velocity range, the maximum derivative was in the range from 28.5- 72.4MPa/ms. For the compression test at 500 mm/min the maximum pressure derivatives for 10 and 30 % compression were around 30 MPa/ms and for 60 % compression in the range from 36-55 MPa/ms. For the friability test at a velocity of 79.9m/s and the compression test at 60% at 500 mm/min, the burning area have been determined. The results are shown in figure 6. It is very obvious that these are not similar, except for the end part in the burning process.

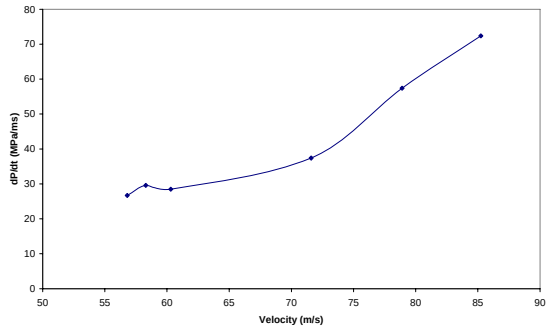

Figure 5. Pressure derivative as a function of the sample velocity in the Friability test.

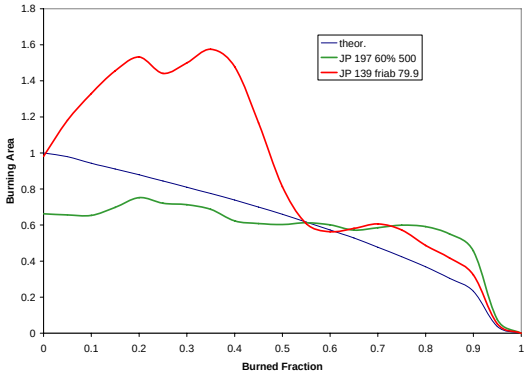

Figure 6. Comparison of the burning area of due to damage in the a gas gun experiment at 79.9m/s and a compression test at 500 mm/min up to 60% compression.

3.2 Thermal damage

The HU45 samples were heated up to a certain prescribed temperature and maintained for 5 hours. Several levels of damage were obtained by the variation of the prescribed temperatures, 165ºC, 170ºC and 175ºC. Like the mechanically damaged samples, the extent of damage was determined by means of the pressure derivative and burning area from closed vessel test results. The sensitivity of the samples were tested by flyer impact test and closed vessel bomb tests. The sensitivity of HU45 after thermal damage was also assessed by fragment impact testing.

Mass loses were measured of of 0-0.05 g at 165ºC, 0.1 g at 170ºC and 0.25 g at 175ºC. The thermally damaged samples were tested in a closed vessel to obtain the burning area of the damaged samples. From figure 7 it is very clear that due to the thermal damage the burning area increased dramatically in comparison to the pristine sample.

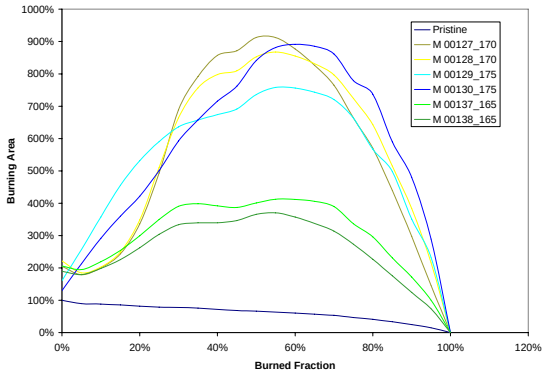

Figure 7. Burning area increase due to thermal damage of HMX-PBX samples.

At 165°C the maximum burning area is about 3 to 4 times larger than the initial pristine burning area. Also from earlier test series it seems that above temperature of about 170 °C, the calculated burning area seems to drop again due to probably the consumption of energetic material (HMX). This has to be confirmed in another test series.

4. SENSITIVITY TESTING

4.1 Mechanical damage

The shock initiation thresholds of pristine and mechanical damaged HU44 have been determined by flyer impact. The initiation threshold is determined in a series of 8 samples.

The damaged sample is impacted by a 125 μm thick Kapton foil. To access the shock-to-detonation transition, or the failure of initiation, a fibre optic probe is inserted in the sample [8]. The HU44 samples were mechanically damaged by compression. The samples with the highest damage level were the ones compressed up to 60% of their original length by compression velocities of 50 and 500 mm/min. These samples did not recover after a week and remained in their "damaged shape". In the flyer impact test this resulted in a difference in measurement for the detonation velocities. The sample at a compression rate of 500 mm/min showed a variation in detonation velocity from 7.6 km/s at the end of the sample down to 6.8 km/s in the middle of the sample. For the sample at 50 mm/min this was 7.7 km/s and 7.1 km/s, respectively. This is due to the fact that the density in the middle of the sample is less compared to the top and bottom. The samples compressed up to 10% and 30% show no big difference from pristine HU44. Figure 8 shows the detonation velocity as a function of distance. In the graph on the right, showing the 60% strain sample, one can observe a clear dip in detonation velocity corresponding with the centre of the sample. Table 1 shows the detonation velocity (D) and initiation threshold velocity (V_F) of the compressed samples. This shows that within the range of error taken at these tests no significant change in sensitivity is measured.

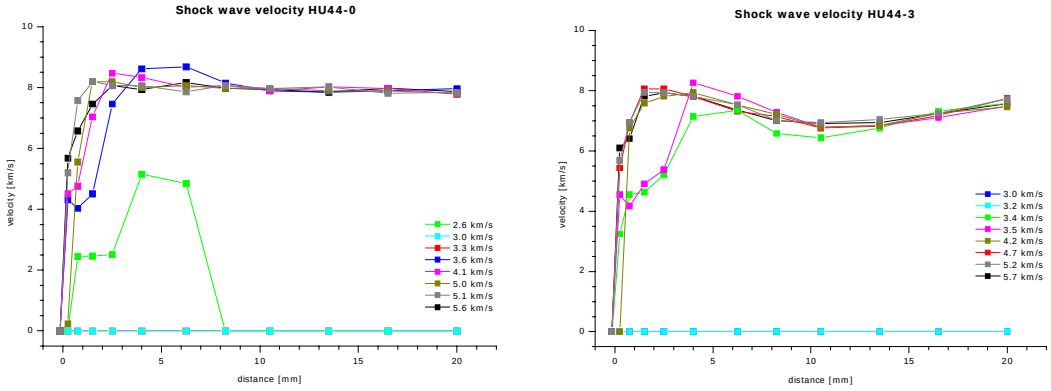

Figure 8. Left : MAP HU44 pristine and right : HU44 compression 500 mm/min, 60% strain.

Table 1. Comparison of detonation velocity and critical flyer impact velocity of HU44 (pristine and mechanically damaged).

Material	D (km/s)	V_F (km/s)
HU44 pristine	7.9	3.5 ± 0.2
HU44 10%, 50mm/min	7.9	3.2 ± 0.3
HU44 30%, 500mm/min	7.9	3.5 ± 0.2
HU44 60%, 50mm/min	7.1 – 7.7	3.6 ± 0.2
HU44 60%, 500mm/min	6.8 – 7.6	3.3 ± 0.2

4.2. Thermal damage

Thermally damaged samples of HU45 were also tested by flyer impact to determine their shock initiation thresholds. Table 2 shows the difference in detonation velocity (D) and initiation threshold velocity (V_F). It shows clearly that the aged HU45 is more sensitive and can be shock initiated around 2 km/s. It also shows the decrease in detonation velocity after being thermally damaged.

Table 2. Comparison of detonation velocity and critical flyer impact velocity of HU45 (pristine and thermally damaged).

Material	D (km/s)	V_F (km/s)
HU45 0ºC	8.3	3.1
HU45 165ºC	7.8	2.6
HU45 170ºC	6.8	2.7
HU45 175ºC	7.0	1.9

Also samples of HU45 for bullet/fragment impact were thermally damaged. One sample was confined at a temperature of approximately 160ºC for 5 hours and then fired at. Another was thermally damaged at 165ºC for 5 hours, and then cooled before it was fired at.

Fragment impact testing was performed on pristine as well as on thermally damaged material. The test was performed with a cylindrical tungsten fragment of 70 g and a diameter of 22.1 mm. The test vessel was made of 34CrNiMo6V steel with a static burst pressure of ~130 MPa and the cap thickness of 5 mm. The go-no-go threshold of the pristine material was between a fragment velocity of 500-532 m/s.

At 500 m/s no reaction occurred and the fragment was even stopped by the explosive material. At 532 m/s a prompt shock was observed.

To perform a fragment impact test on a thermally damaged material, the test vehicle was heated using an electric heater over the total length of the vessel. The maximum temperature of a test vehicle of this size is about 165 °C, resulting in an estimated temperature inside the vehicle of 156°C and a few degrees higher. A higher temperature results in a runaway of the explosive. Just before testing, the material was heated for 5 hours at the prescribed temperature. At a velocity of 472 m/s, no reaction occurred and partly decomposed and thermally damaged material was found all over the test bunker. At 511 m/s a detonation was observed. This value falls in-between the go-no-go threshold (500-532 m/s) of the pristine material so a difference in sensitivity is not yet apparent at this temperature. Also a fragment impact experiment was done on a damaged and non-confined sample. This sample was stored at 165 ºC for 5 hours and then cooled and given a week to reheal. The go-no-go threshold of the damaged material was between a fragment velocity of 544-672 m/s. Figure 9 shows the different go-no-go thresholds for fragment impact testing on thermally damaged HU45.

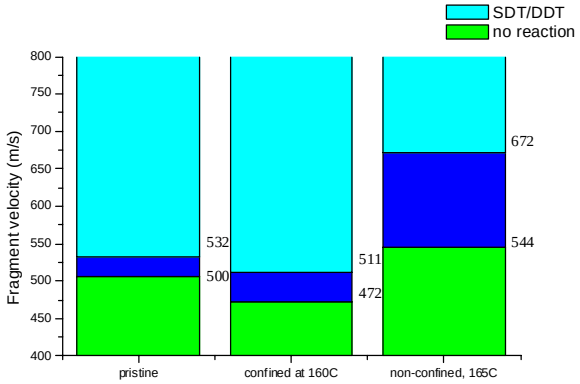

Figure 9. The impact sensitivity of HU 45 in pristine state, heated and confined state, heated-then-cooled and non-confined state. Impact with the Ø22 mm tungsten fragment.

DISCUSION AND CONCLUSIONS

This paper is build up on earlier papers on damage research [5, 9]. A start is made to relate the amount of damage to the change in (shock) sensitivity. The amount of damage is quantified by determining the amount of energy in a double load-unload compression cycle, but also by means of the pressure derivative and burning area increase in a closed vessel.

The aged material is much easier to initiate than the pristine material. It seems that the increase in sensitivity, due to thermal damage, starts between 160 and 170°C, which is near cook-off temperature. The oxidation of the binder and decomposition of HMX leads to a porous and brittle material, which is definitely the case above 170°C. A full relation to the changes in sensitivity and/or burning area is not yet made, but will be under attention in the future. Fragment impact testing on thermally damaged HU45 in confined and non-confined geometries. The difference in sensitivity in the fragment impact tests is not yet understood.

The mechanical damage on the HU44 at 60% compression strain and at high compression velocities is severe. By flyer impact testing the change in density in the centre of the sample resulted in a dip in detonation velocity. The change in sensitivity was not significant. The damage level by means of pressure derivative of the 60% compression strain samples is comparable with gas gun velocities of approximately 80 m/s.

In the compression tests with HU45 a comparison has been made between the energy of two compression cycles and the burning area increase. More data needs to be gathered to obtain a better relation. It is obvious that a double cycle induces more damage than a single cycle; this has to be taken into consideration in the future.

Although a lot of data was gathered in these experiments, a lot more is needed to fully establish a quantitative relation between the damage and sensitivity increase.

During the research we learned that a proper closed vessel is essential in determining the different parameters used for quantification of damage. Also by inducing thermal damage, a tailored oven is needed and individual measurement of the sample temperature is preferred.

The burning area calculated from closed vessel experiments seems to be a good en sensitive method to determine quantitatively the amount of damage in a sample.

In the future the attention will also be more on the development of a macroscopical damage model for computer purposes. At this moment the increased burning area parameter is implemented in TNO's pore collapse, ignition and growth model such that damage and increased porosity is supported in the shock initiation modelling.

5. ACKNOWLEDGEMENTS

The work done by Mrs. EJ Albrecht, Mr. AG Boluijt, Mr. W Duvalois, Mr. P van 't Hof, Mr. FAMH Jacobs, Mr. O Leurs, Mr. JC Makkus and Mr. FP Weterings is greatly appreciated.

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