Production and Characterization of Insensitive Explosives

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1. Introduction

The use of insensitive energetic materials for propellants and ammunitions is a matter of particular interest. By using insensitive explosives, the sensitivity against shock waves can be reduced drastically. Insensitive behaviour of the basic raw material is as well important for a save processing of the explosives.

The focus of this study is to process different explosives with insensitive behaviour. Therefore e.g. commercially available 1,1-diamino-2,2-dinitroethylene (FOX 7) and cyclo-tetramethylenetetranitramine (HMX) were recrystallized from solution.

2. 1,1-diamino-2,2- dinitroethylene

The properties of commercially available 1,1-diamino-2,2-dinitroethylene (FOX-7) were investigated before using it for experiments. SEM, light microscope and AFM images have been taken [Fig. 1, Fig. 2].

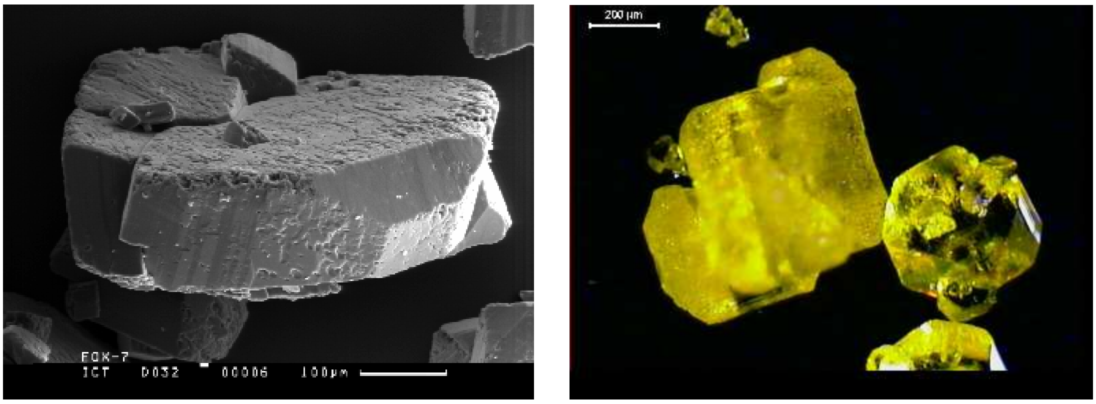

Fig. 1: left side: SEM- exposure of FOX-7, right side: light microscope exposure of FOX-7

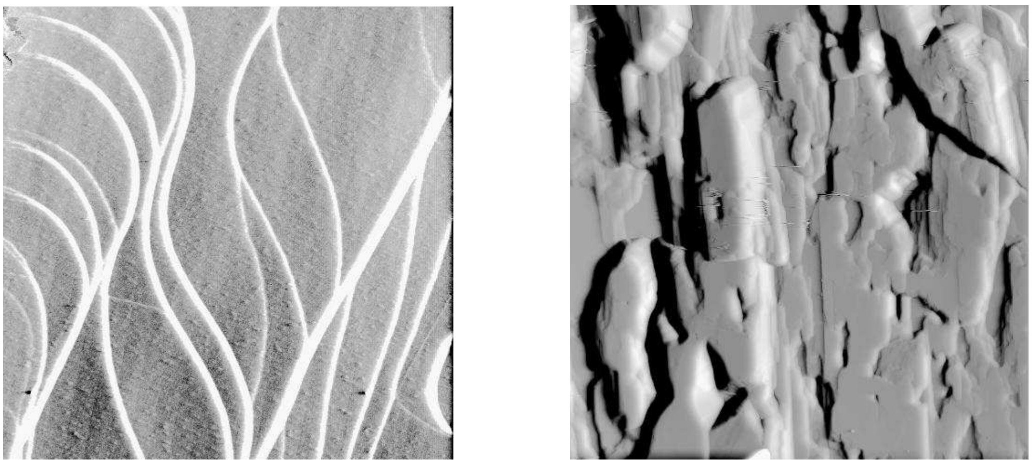

Fig. 2: AFM-exposures of different faces of a FOX-7 crystal

Friction and impact sensitivity tests of commercially available FOX-7 have been carried out according to BAM. A comparison of FOX-7 and RDX is shown in Tab.1. GAP-tests have also been done at ICT. Therefore, pressed explosive charges with three different binders have been tested. The results have been compared to those of RDX [KRET04] (Fig. 4).
The structure of the unit cell of FOX-7 is shown in Fig. 3. It was constructed with Materials Studio (Accelrys) by using the data collected by Bemm and Östmark [BEMM98]. In the inside of a layer, the molecules are linked together by strong hydrogen bonds. Among each other, the layers are connected by relatively weak Van-der-Waals interactions. The insensitive behavior of FOX-7 can be explained by its structure. Stresses inside the crystal are reduced by the movement of two layers. In this manner the crystal is getting more insensitive against mechanical strain.

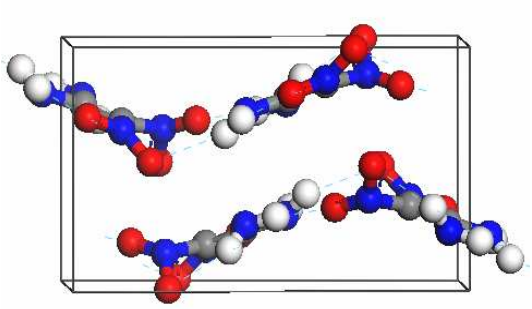

Fig. 3: Unit cell of FOX-7

	FOX-7	RDX [MEYE91]
Impact [Nm]	25	7,4
Friction [N]	240	120

Tab. 1: Comparison of FOX-7 and RDX

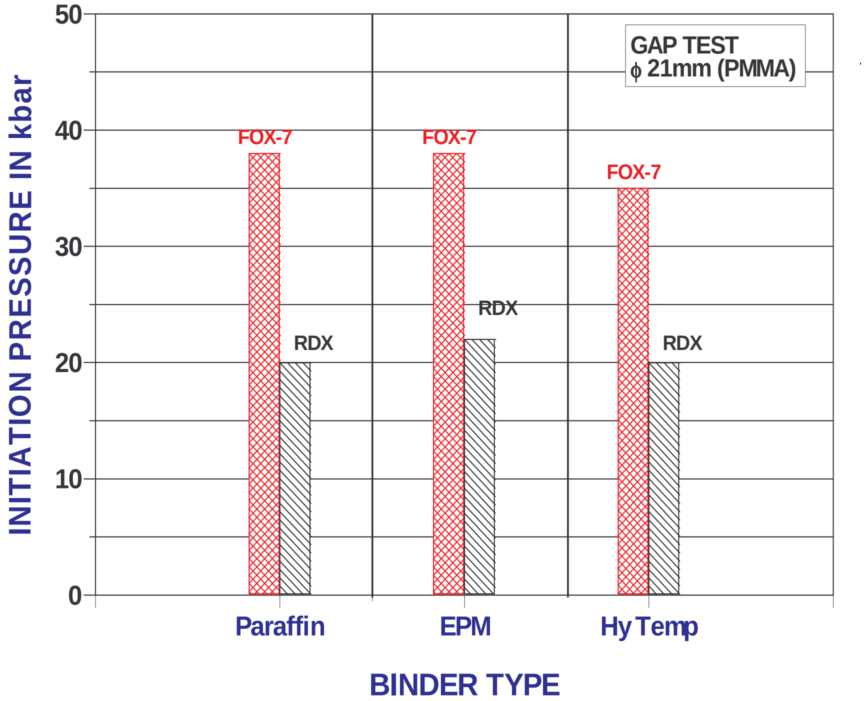

Fig. 4: GAP test of FOX-7 and RDX with three different binders [KRET04]

To be able to conduct crystallization experiments, solubility curves of FOX-7 have been determined. As solvents, n-methyl-pyrrolidone (NMP), dimethylformamid (DMF)

and mixtures of NMP/water in different ratios have been served. In Fig. 5, the solubility curves for nMP and nMP/water-mixtures (30/70 weight%, 50/50 weight-%, 70/30 weight-%) are shown.
The experimental conditions of the experiments are listed in Tab. 2.

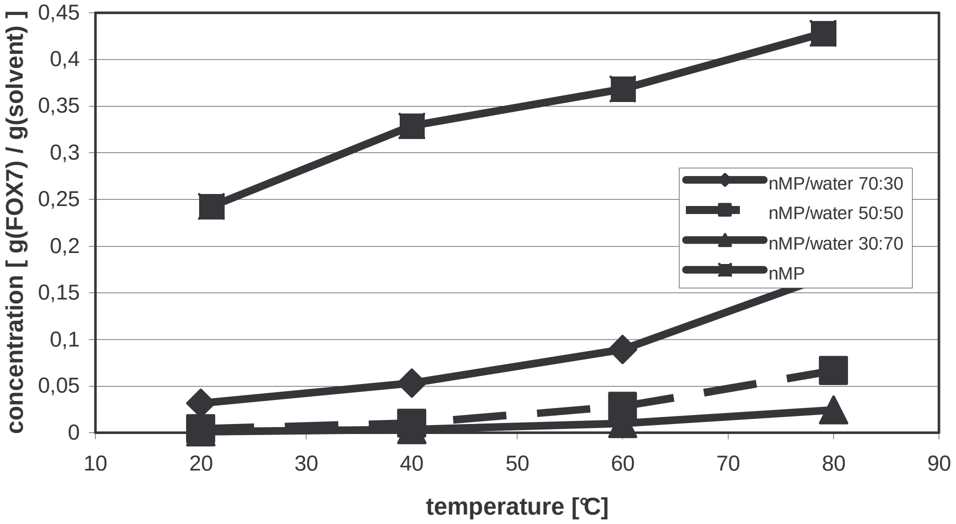

Fig. 5: Solubility curves of FOX-7 / NMP and NMP/water-mixtures

Tab. 2: Experimental conditions

Experimental setup	Jacked vessel, 300ml
Cooling rate	10 K/h
Cooling time	4 h
Saturation temperature	60℃
Stirrer / stirrer speed	Blade stirrer / 200 min^{-1}

The recrystallization was carried out with the aim to study influences on crystallization like temperature run, solvent and antisolvent. Considering the example FOX-7 grown from NMP/water (70/30 weight-%) it can be seen that the addition of antisolvent leads to higher rates of yield and a narrow particle size distribution (Fig. 6, Tab. 3).

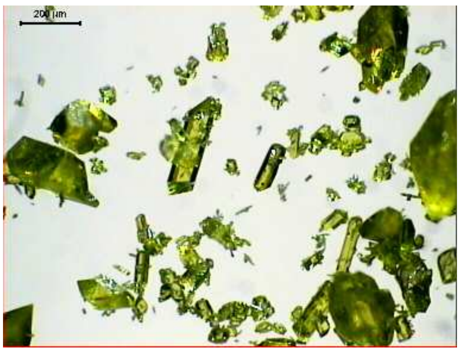

Fig. 6 left: FOX-7 grown from NMP/water (70/30 weight-%), right: FOX-7 grown from NMP/water (70/30 weight-%) and antisolvent

Tab. 3: Properties of the recrystallized FOX-7

FOX-7	NMP/water	NMP/water + antisolvent
Mean particle size [µm]	213	273
Span (90/10)	1,445	0,506
Rate of yield	50%	80%
Impact sensitivity [Nm]	25	35
Friction sensitivity [N]	240	192

3. Cyclo-tetramethylenetetranitramine

For nitramines such as cyclotrimethylenetrinitramine (RDX) and HMX in former work is shown [KROE98], that inner defects act as centres for Hot Spots. Therefore on the one hand we recrystallized and on the other hand we comminuted HMX to obtain inclusion free, high density material.

In a first step the solubility of HMX in propylencarbonate (PC) was determined. We found out following dependence of the solubility from temperature:

$$X\left[\frac{gHMX}{gPC}\right] = 0{,}0018\frac{T\left[{}^{\circ}C\right]}{\left[{}^{\circ}C\right]} - 0{,}0156 \qquad (1)$$

The crystallization was carried out in solution crystallization batch process, with a batch size of 3 l. The material was soluted into propylencarbonate at a temperature T_S with a supersaturation of one. Afterwards the solution was cooled down with a constant cooling rate b. Afterwards the material was washed out with acetone. Fig. 7 and Fig. 8 show the crystals obtained by this process.

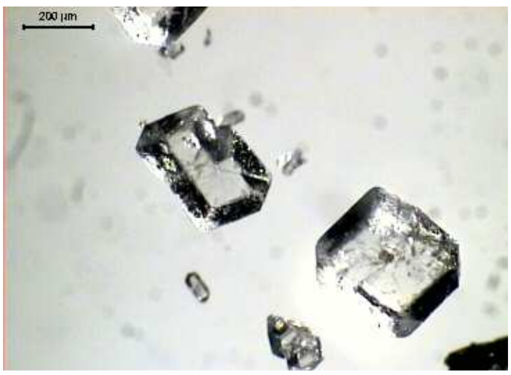

Fig. 7: Microscopic pictures of recrystallized HMX

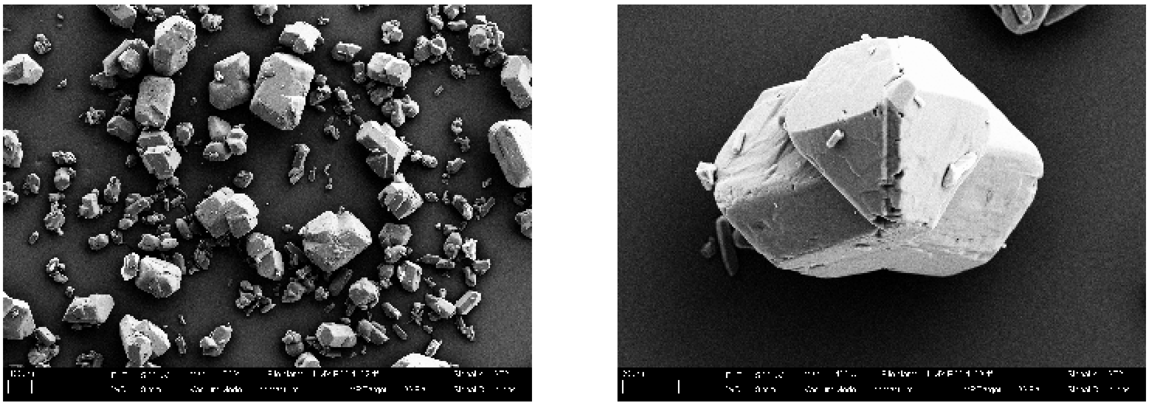

Fig. 8: SEM-pictures of recrystallized HMX

The obtained material has a low number of inclusions and the single crystals are not agglomerated. On the pictures almost none secondary crystals can be found.

Tab. 4 summarizes the key parameters of the crystals, such as mean particle size, particle size distribution coefficient, specific surface area and density.

Tab. 4

Recystallized HMX	
Mean particle size	150 µm
Particle size distribution	0,78
Specific surface area	0,040 m²/g
Density	1,91 g / cm³

Another HMX product was produced by comminution. We used an annular gab mill with a gab size of 5 mm to obtain submicron HMX. The idea was to break up the inclusions in the feed material by milling and to design a very tight distributed fine particulate material.

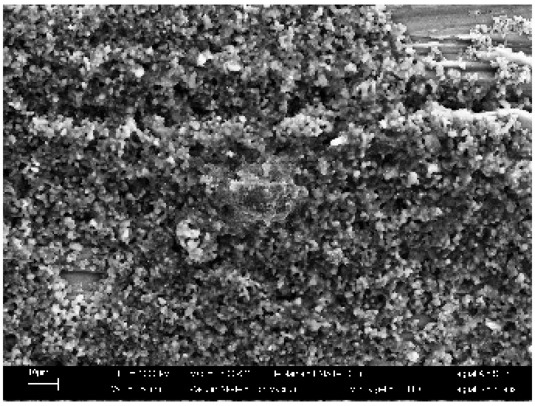

Fig. 9:
Comminuted HMX material

Fig. 9 shows the comminuted HMX fraction. It seems to have a particle size around 1µm, but due to the different refractive indices of the different crystal faces, we weren't able to measure it accurately by laser diffraction method. The particle size distribution is very narrow. The particles are rounded without splits which should result in lower sensitivity.

Comminuted HMX	
Mean particle size	Approx. 1 µm
Specific surface area	4,85 g/m²
Density	1,92 g/cm³
Impact sensitivity	7,5 Nm
Friction sensitivity	96 N

The impact sensitivity of the grinded material is 50 % lower than from the original material. In the friction sensitivity there is no change. The high density points at break up of inclusions.

These investigations will be improved with standard GAP tests.

References

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