TITLE: EFFECTS OF ULTRASONIC BATH TREATMENT OF HMX CRYSTALS

AUTHOR(S): Cary B. Skidmore, DX-2

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# Effects of Ultrasonic Bath Treatment on HMX Crystals

by
Cary B. Skidmore
Group DX-2
High Explosives Science & Technology

## Abstract

Ultrasonic cleaning baths, common in many chemical laboratories, have been used to disperse particle agglomerates prior to automated particle size analysis and have been proposed for disassembly of consolidated powders of crystalline HMX (cyclotetramethylene-tetranitramine). This paper reports the effects of a Branson ultrasonic bath on coarse HMX crystals. Three experimental approaches are presented. The following observed effects are discussed: reduction of particle size, alteration of particle morphology, and fracture of individual crystals.

## Introduction

The beta polymorph of HMX (cyclotetramethylene-tetranitramine) is the principal constituent in many formulations of High Explosives. Powders that go into the formulations have been characterized. Most of the formulations are consolidated by pressing at elevated temperatures and pressures and it is of interest to characterize the HMX particles after pressing. That helps establish the initial state for further investigation into mechanically damaged explosives.

Ultrasonic cleaning baths have been proposed to facilitate removal of the binder so the HMX particles can be characterized. Such a treatment has also been proposed to disassemble powders which have been consolidated or pressed without binder. Prior to automated particle size analysis, agglomerated particles are typically dispersed in the liquid carrier by this method. Before adopting the proposed methods, it was important to establish whether there would be deleterious effects on the particles.

The first approach or experiment was an overtest. A sieve-cut sample of particles was processed them for 30 minutes and then examined. Only a small fraction of the original material was retained on the sieve. Next, an automatic particle size analyzer was employed to give a full size-distribution. Particle-size distributions (PSD) were run before and after incrementally longer residence times in the bath. The volume % peaks were progressively reduced and shifted toward smaller particles. The final experiment was to evaluate whether the effect was particle-particle collisions or the ultrasonic stimulus per se. Individual crystals were treated and fracture resulted.

## Experimental Details and Results

The ultrasonic cleaning baths used in these experiments were manufactured by Branson. The sieve-cut experiment used an earlier version (B-12) of the model 2210 which was used in the other experiments. The input frequency for each was 48 kHz at a power intensity of 1.8 watts per square inch. The model 2210 was operated in "degas" mode which is considered to give a 10% drop in power.[1]

The HMX used in the sieve-cut experiment was production lot HOL83-L030-050. The HMX used in the particle-size distribution experiment was production lot 92032. The individual crystals of HMX were recrystallized from acetone locally. The HMX was placed in a small flask with ethanol or water and then the flask was held in the bath during processing.

## *Sieve Cut*

The HMX particles used in this experiment passed through 177-micron, square openings in the screen but were retained on a screen with 125-micron, square openings. One gram of this material in ethanol was treated for 30 minutes. Before processing, the supernatant liquid was clear, afterwards it was clouded. Upon filtration, only 0.0369 grams were retained on the 125-micron screen. Representative pictures of the particles before and after processing are shown in Figure 1 and Figure 2.
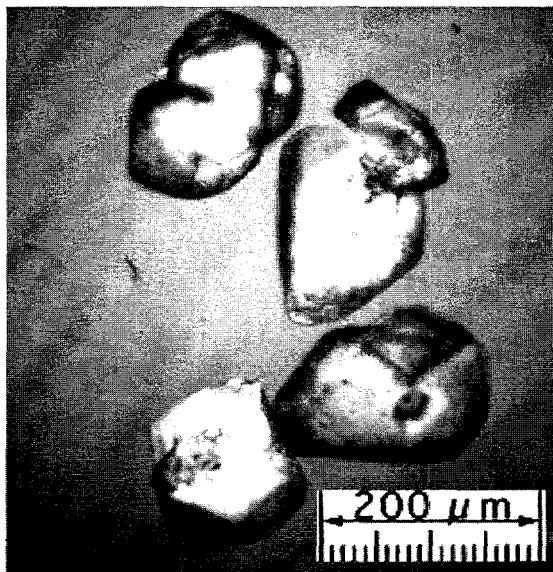


**Figure 1.  HMX Crystals Before Treatment in Ultrasonic Bath**
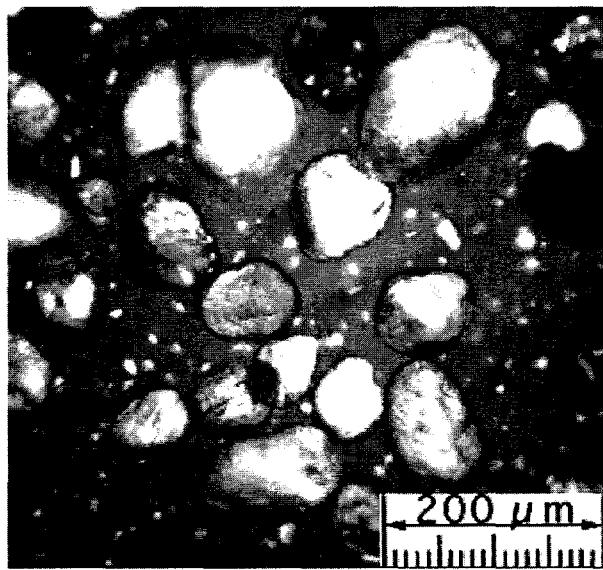


**Figure 2.  HMX Crystals After Treatment in Ultrasonic Bath for 30 Minutes**

## *Particle Size Distribution*

Particle-size distributions (PSD) were measured using water as the carrier fluid in a Coulter LS 230 particle size analyzer. PSDs were determined for the following residence times: 0, 1, 5, 15, and 30 minutes. Volume percentage as a function of particle size for each run is shown in Figure 3. For all but the one-minute run, a second PSD is given to show repeatability of the analysis.
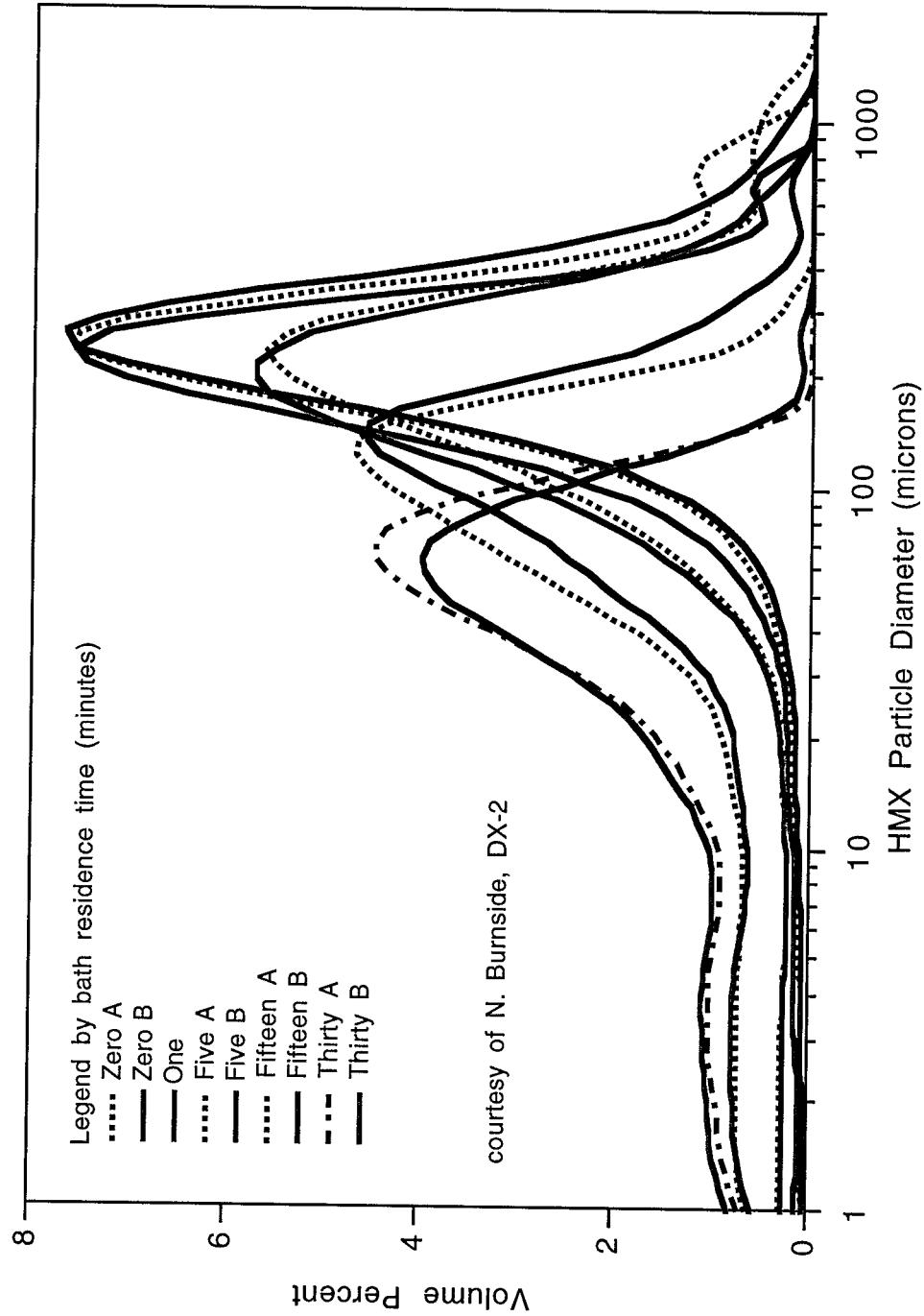
**Figure 3. Particle Size Distribution Analysis**

Legend by bath residence time (minutes)

........ Zero A
———— Zero B
———— One
········ Five A
———— Five B
········ Fifteen A
········ Fifteen B
———— Thirty A
—·—·— Thirty A
———— Thirty B

courtesy of N. Burnside, DX-2

HMX Particle Diameter (microns)

Volume Percent

## *Individual Crystals*

Two individual crystals were chosen for this experiment. These are shown in Figure 4. One appeared to be a single crystal with a major crack and the other appeared to be two crystals grown together. Both samples were approximately 40 times larger than typical production crystals. Large crystals were chosen for easy retrieval and examination. After one minute of processing time, there was no observable change in the large single crystal but there were some bits of crystalline debris floating on top of the water. The dual crystal broke into two crystals after approximately 10 seconds. No change in features of the individual pieces was observed. This is shown in Figure 5. Some bits of crystalline debris were also floating on the top of the water from this run.

The water, with bits of debris from each sample, was discarded and replaced with fresh water. The first individual crystal was placed alone in a flask. Each large piece of the dual crystal was placed in another flask. This offered one sample with individual crystal effects and one sample with a higher probability of particle-particle collisions. Observations were made after four additional minutes of residence time. The first sample produced a clouded suspension of fragments. The second sample produced a less clouded suspension. The largest particles were retrieved and are shown in Figure 6. The fragments from the second sample (on the right) appear to be more faceted and less rounded, than those from the first sample.
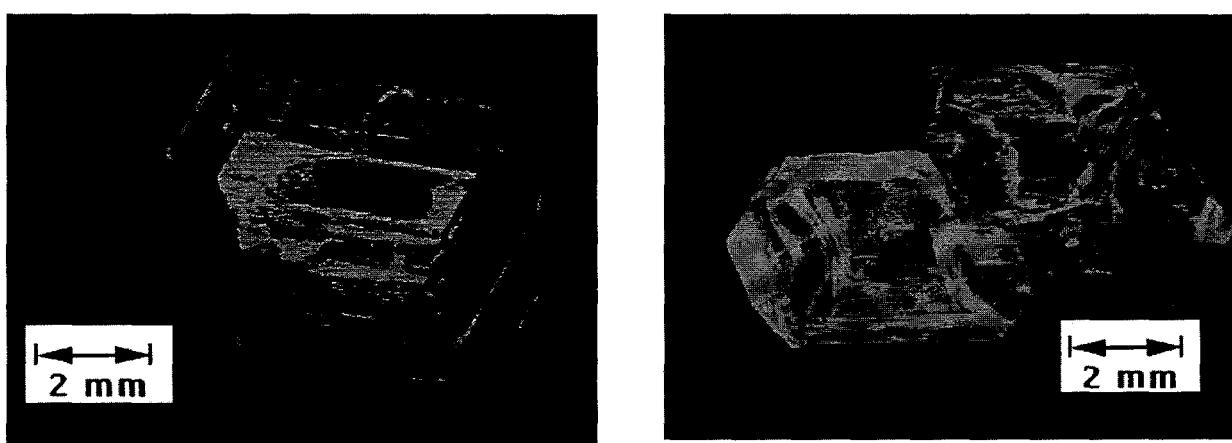


**Figure 4. Individual, Large, HMX Crystals Before Ultrasonic Treatment**
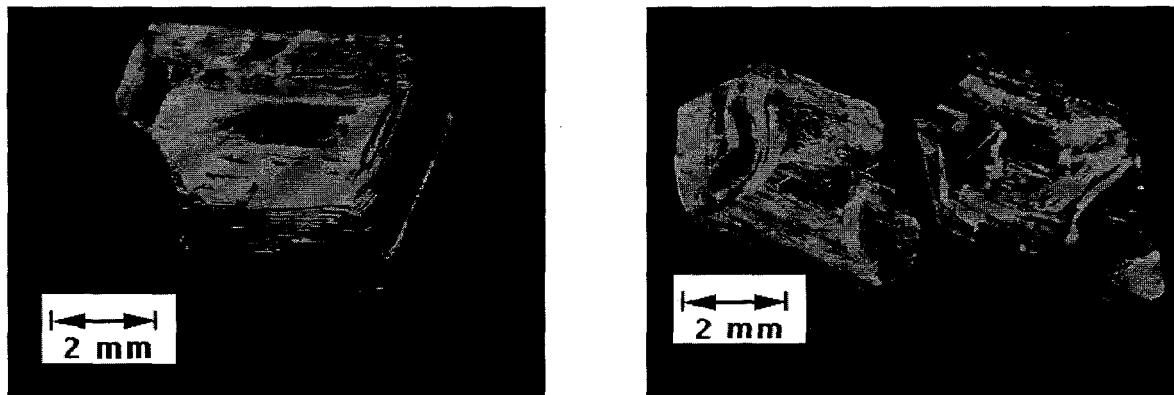


**Figure 5. HMX Crystals After One Minute of Ultrasonic Treatment**

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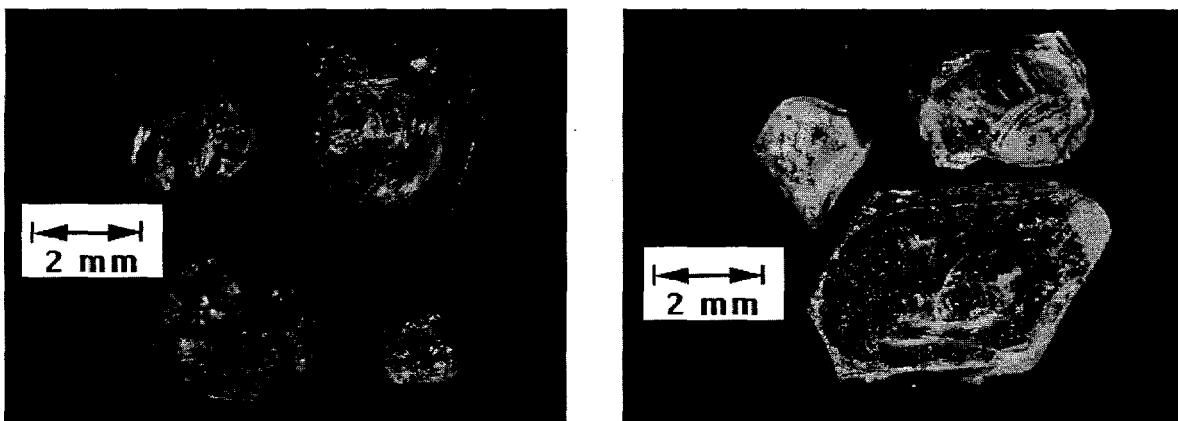
**Figure 6. HMX Crystals After Five Minutes of Ultrasonic Treatment**

## Discussion and Conclusion

Three different approaches were taken, each using a different lot of material, to investigate the effects of ultrasonic bath treatment on HMX crystals. Each showed a pronounced effect.

A sieve-cut sample (125-177 microns) of HMX crystals retained less than 4 weight percent of original material on a 125-micron screen after 30 minutes of processing. Comparing pictures of the crystals before and after processing shows that the crystals are smaller and more rounded after processing. Rather than looking like faceted, monoclinic crystals, they have the appearance of river rocks.

A shift was demonstrated in particle-size distribution as a function of processing time. The volume % of large crystals was reduced and the peak particle-size became smaller after one minute of processing time.

Individual, oversize crystals exhibited some degradation after one minute and extensive fracture after five minutes of processing time. It may be argued that these 40-times oversize crystals are more susceptible to fracture due to larger flaws and higher triaxial stress states. However, the general results are consistent with the other experiments. A comparison of the results from the second phase of the experiment with individual crystals (one crystal vs. two crystals in the flask) suggests that mechanical abrasion or erosion from particle-particle collisions are not the only factors involved in breaking up HMX crystals. Apparently, the ultrasonic stimulus has an effect in and of itself.

## Acknowledgments

[1] Private communication with product support of Branson Ultrasonics Corp., Danbury, CT on Sept. 18, 1996. Power decrease is attributed to periodic killing of the 100% amplitude modulated waveform for 3 milliseconds.