PROCESS OPTIMIZATION TO IMPROVE PRODUCT QUALITY AND CONSISTENCY OF ENERGETIC MATERIALS AT HSAAP

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Briefing Outline

- Introduction and Program Overview
- Recrystallization of Crude HMX
  - Effect of process parameters on particle size distribution
  - Effect of process parameters on particle shape and crystal morphology
- Results and Path Forward
- Concluding Remarks
- Acknowledgements

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Introduction / Program Overview

- BAE Systems manufactured a wide range of energetic ingredients at HSAAP
  - Legacy: RDX/HMX (various classes); Comp B/C-4
  - Next Generation: NTO/TATB/DNAN/IMX family
- Robust manufacturing processes established
- Continuous Improvement effort between R&D, Operations and QA to further improve product quality & consistency via process parameters change
  - DOE / lab scale experiments
  - Extensive characterization
  - Production scale confirmation experiment
- HMX recrystallization targeted first
  - Other products will be studied

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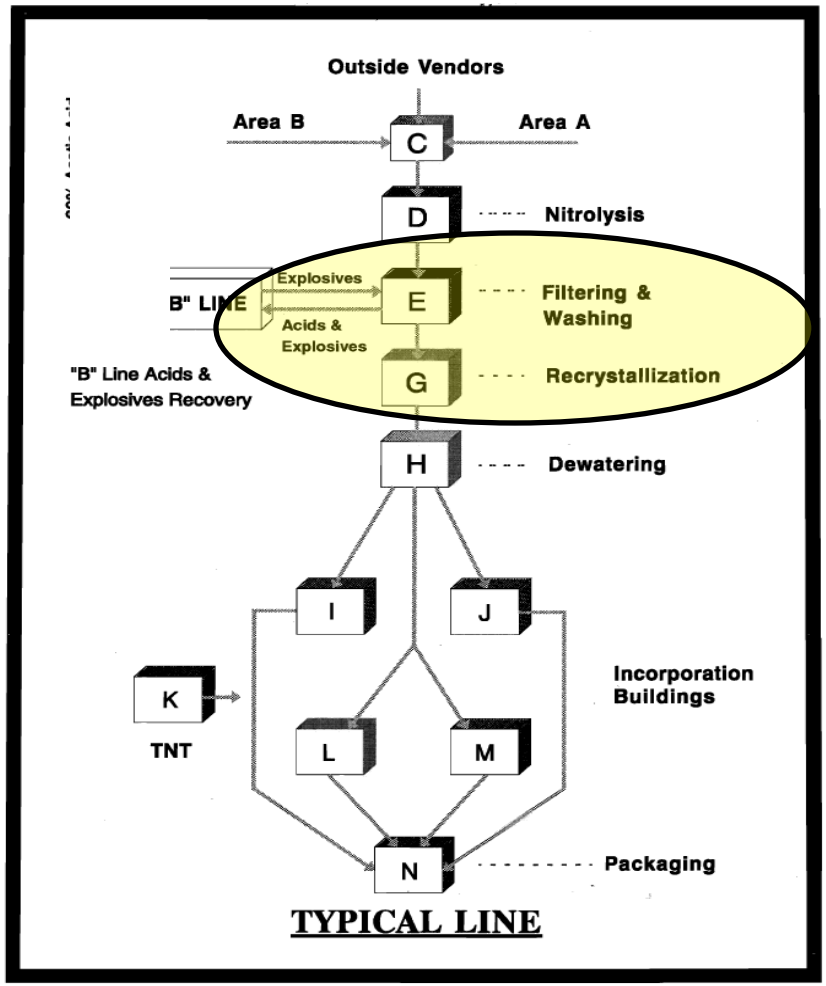
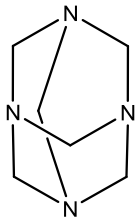
HMX Manufacturing Process at HSAAP



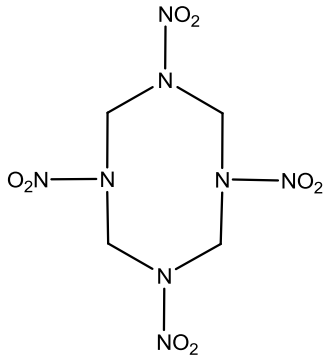


Hexamine

NITRATION

nitric acid

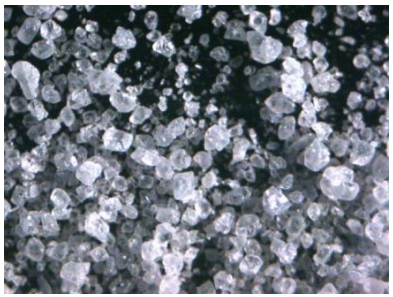
ammonium nitrate,
acetic anhydride,
acetic acid



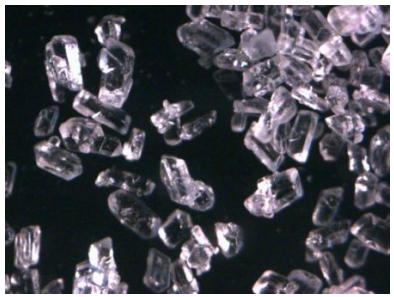
Crude HMX

RECRYSTALLIZATION
Acetone/Cyclohexanone
DEWATERING
GRINDING

HMX Class 1



HMX Class 3



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HMX Recrystallization with Acetone

- Exclusively used as the solvent to produce HMX Class 1 and HMX 80s
- Project Goals
  - 1. Particle Size Distribution Improvements
    - Determine which processing parameter(s) contributed to changes in the particle size distribution of HMX Class 1 and 80s
    - Once identified, focus on optimizing specific processing parameter(s) in the HMX Recrystallization to further improve consistency in particle size distribution
  - 2. Crystal morphology (Quality) Improvements
    - Adjust processing parameters to produce rounded HMX crystals for commercial applications (HMX mains).

Experiment Set Up

- All experiments were conducted initially in lab scale, with equipment simulating production scale
  - Two 13 Liter Glass Vessel with steam/water jacket; agitator; solvent recovery
  - HMX crude (dry) dissolved in acetone/water slurry in the dissolver (top)
  - Saturated HMX slurry gravity fed into recrystallization vessel (bottom)
  - Recrystallized HMX product filtered and oven dried
  - Recrystallized HMX product characterized by Malvern (PSD) and Digital Microscopy (Shape/Morphology)

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Project 1: HMX Class 1/80S PSD Improvement

- A Design of Experiment focusing on the major processing parameters affecting the HMX PSD was conducted:
  - Solids Concentration (amount of HMX crude in the slurry)
  - Solvent to Water Ratio
  - Agitation Rate in Recrystallization Vessel
  - Reaction Time at various stages during dissolution/recrystallization
  - Water presence in Recrystallization Vessel during the charge of HMX crude feed)
- A thirty-two run experiment was designed and conducted
- Particle size distribution of HMX crystals from each run was analyzed

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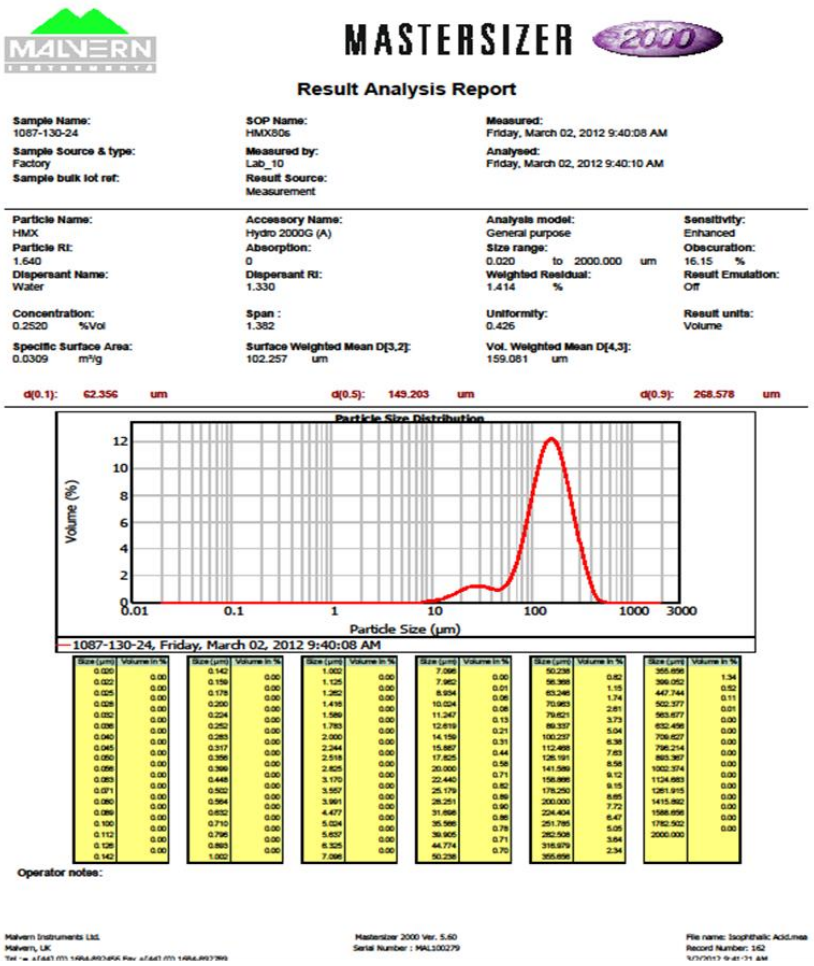
Design of Experiment (HMX Dissolution / Recrystallization)

Run Order	PROCESS PARAMETERS						
	A	B	C	D	E	F	G
1	Low	Low	High	Low	High	High	High
2	High	Low	Low	Low	High	Low	High
3	Low	High	Low	High	High	Low	High
4	Low	Low	High	Low	High	High	High
5	High	High	High	High	High	High	High
6	High	High	High	Low	High	Low	Low
7	Low	Low	Low	High	Low	High	High
8	Low	High	Low	Low	High	High	Low
9	Low	High	High	High	Low	High	Low
10	High	High	Low	High	Low	Low	Low
11	High	High	Low	Low	Low	High	High
12	High	Low	High	Low	Low	High	Low
13	Low	High	High	Low	Low	High	High
14	High	Low	High	High	Low	Low	High
15	Low	Low	High	High	High	Low	Low
16	Low	Low	Low	Low	Low	Low	Low
17	Low	Low	High	High	High	Low	Low
18	Low	Low	Low	Low	Low	Low	Low
19	Low	Low	Low	High	Low	Low	High
20	High	High	High	High	High	High	High
21	Low	High	High	High	Low	High	Low
22	High	High	High	Low	High	Low	Low
23	High	High	Low	High	Low	Low	Low
24	Low	High	Low	High	High	Low	High
25	High	Low	Low	High	High	High	Low
26	Low	High	High	Low	Low	Low	High
27	Low	High	Low	Low	High	High	Low
28	High	Low	Low	Low	High	Low	High
29	High	High	Low	Low	Low	High	High
30	High	Low	High	High	Low	Low	High
31	High	Low	Low	High	High	High	Low
32	High	Low	High	Low	Low	High	Low



HMX Particle Size Distribution by Malvern Particle Size Analyzer

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Design of Experiment Statistical Results

- The DOE was deemed a success
- After analyzing all the PSD results, statistically significant models were generated which determined that **two processing parameters** impacted the HMX PSD more than others
- Due to the complexity of the DOE, large amount of noise was detected in the measurement
  - Gave directional trends rather than accurately predicted values
- Additional lab scale experiments were conducted to validate the models further (additional runs focusing in the two parameters)
- In 5 out of 6 experiments, the PSD of the final product met the predicted ranges
- Valuable findings in the effort to improve PSD consistency of HMX crystals

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Project 1 Path Forward

- A second DOE is recommended to further optimize the two variables
- Incorporate minor changes in other parameters without affecting PSD results
- Upon satisfactory result, scale up to full size production with optimized processing parameters
- Conduct similar study for RDX Recrystallization with Cyclohexanone

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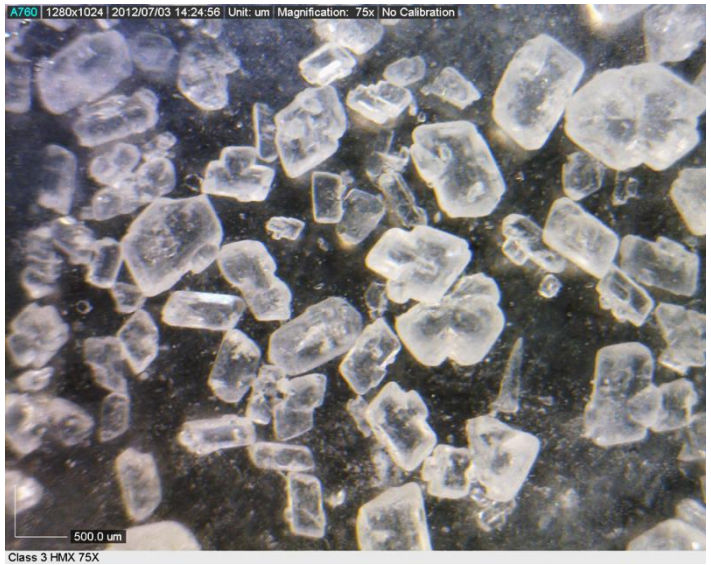
Project 2: HMX Crystal Morphology (Quality) Improvements

- HMX Class 3 recrystallized in Cyclohexanone
- HMX Class 3 used in various commercial products
- Crystal Morphology not ideal (twinning / rough edges)
- Customer requested a different (improved) morphology without changes in particle size characteristics
  - Improvement in product sensitivity and flow properties
  - Higher Bulk Density with rounded crystals
- Evaluate recrystallization with an <u>alternate solvent</u>
  - Larger HMX crystals to mimic the Class 3 PSD profile

Typical HMX Class 3 Crystals



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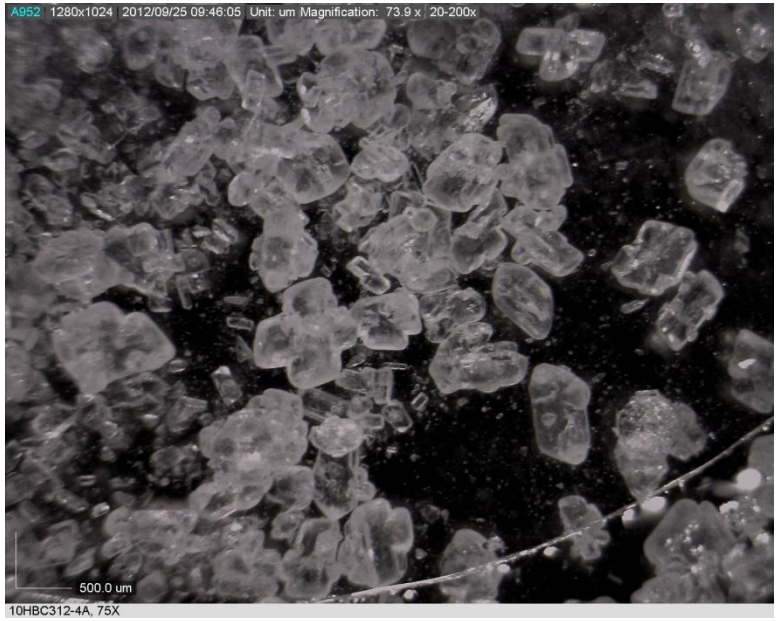
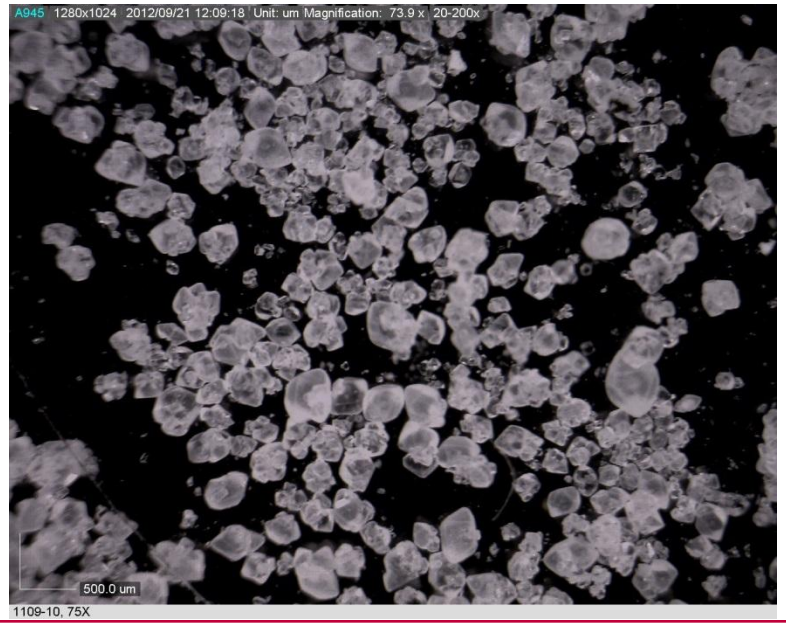
Project 2: HMX Crystal Morphology (Quality) Improvements

- Conduct lab scale experiments to adjust processing parameters from Project 1
  - Different solubility levels
  - Distillation rate
- Significant improvement in crystal shape (more rounded, less twinning)

Typical HMX Class 3 Crystals



Lab Scale Improved HMX Crystals



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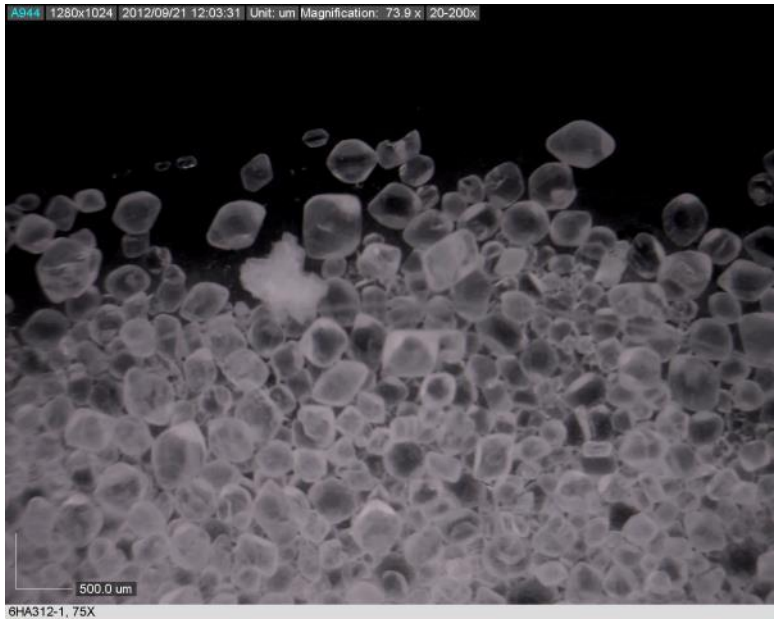
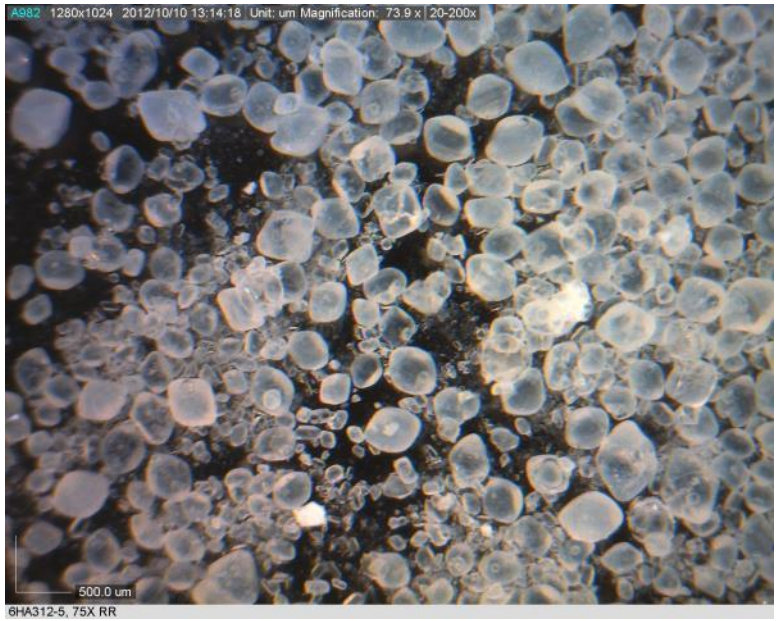
Project 2: HMX Crystal Morphology (Quality) Improvements

- Process improvements evaluated in large scale production
  - 8 batches manufactured
  - Significant improvement in crystal morphology (more rounded)
  - Reduction in Friction Sensitivity (BAM) observed
    - HMX Class 3 (50% ~ 167 N); Rounded HMX Class 3 (50% ~ 204 N)





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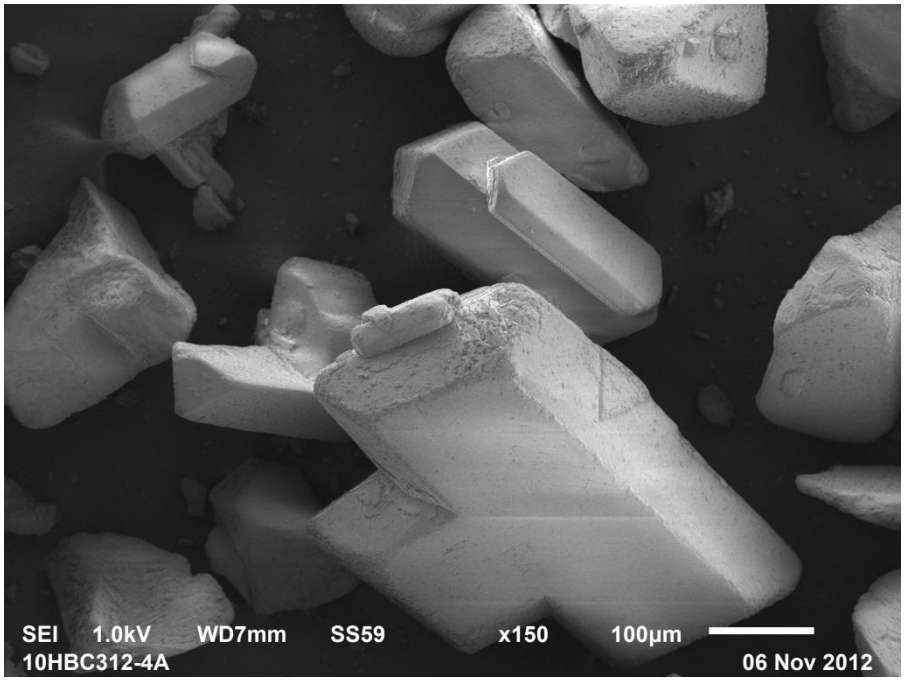
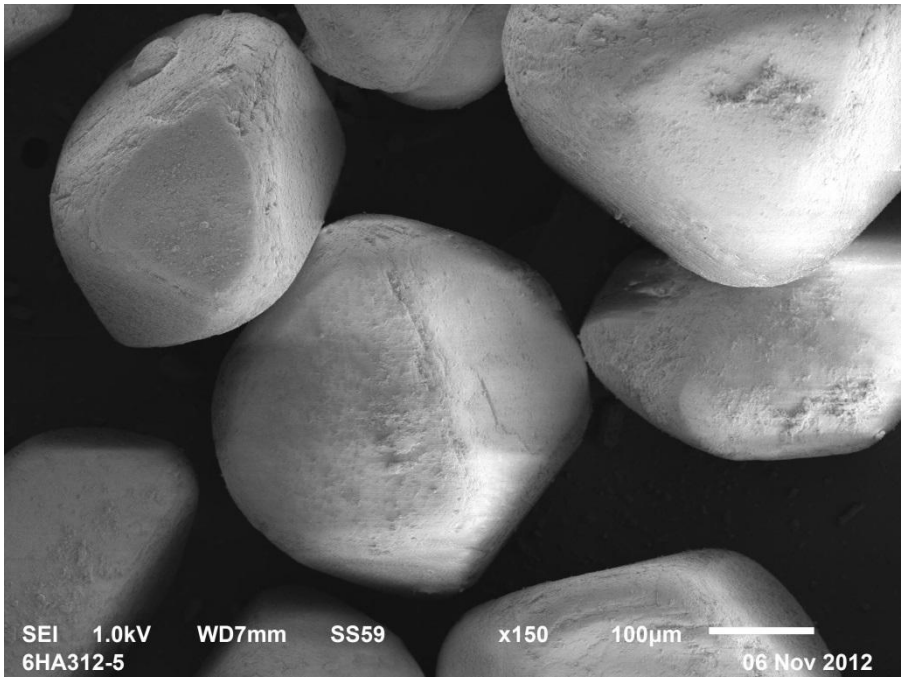
Project 2: HMX Crystal Morphology (Quality) Improvements



Typical HMX Class 3 Crystal



Rounded HMX Class 3 Crystal

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Project 2: Path Forward

- Further optimize the improved production scale HMX Class 3 process
- Gather end-user feedback upon evaluation of the new products
  - Processability of PBXN-110 from CXM-10
    - Lower end-of-mix viscosity due to improved crystal morphology
  - Impact/Friction Sensitivity improvement in PBXN-110

  - Coated HMX products from the Oil and Gas industries (HMX Main)
    - Flow properties improvement (benefit high volume loading in feed hopper)
    - Lower friction sensitivity

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Concluding Remarks

- Currently lots of emphasis focused on the next generation insensitive ingredients
- However, BAE Systems are committed to improving the legacy ingredient products
- Good collaboration between R&D and Operations to evaluate product improvement through well-thought DOE
- Significant improvement in crystal quality in HMX Class 3 achieved with recrystallization using an alternate solvent
- Potential improvement in HMX product consistencies after DOE runs
- Further process improvement projects planned for other products (e.g. RDX & NTO)

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Acknowledgements

- Process Operators and Engineers for technical advise and support
- Operation Analysts for analytical support
- OSI senior management for financial support
- Customers (Oil and Gas Industries) for end-users' evaluation feedback