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IMPROVED PROCESSABILITY OF CAST-CURE PBX'S BY MODIFICATION OF PARTICLE SHAPE

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Objectives

- Background
- Processing Issues and Goals
- Equipment and Analytical Technology Utilized
- Process Variable Investigations
- Production Process Focus
- Conclusions

Acknowledgement

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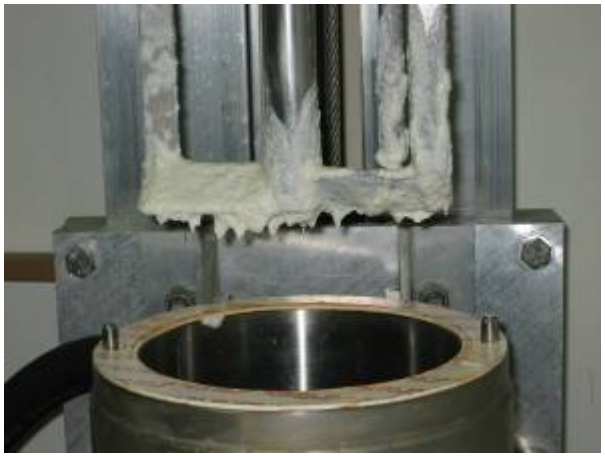
  - David Siggers
- Aerojet
  - Ron Taylor

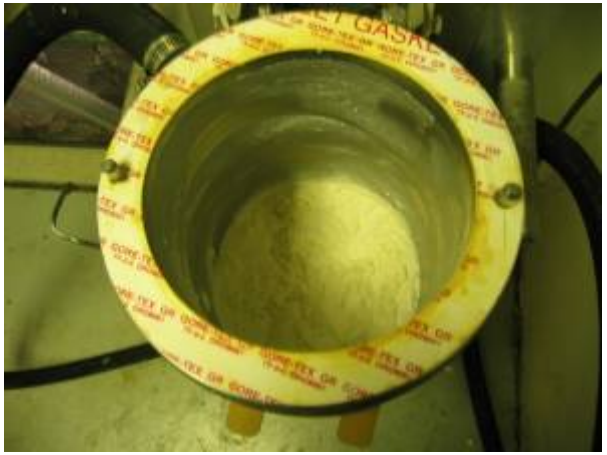
Background

- Customers experience processing issues with a cast-cure PBX (PBXN-110) using CXM-10 as the intermediate
  - Bimodal HMX classes premix supplied by OSI
  - Inconsistent processability of end product
    - Sometimes good, sometimes not so
  - End of mix viscosity variability greater than desired
    - Results in high viscosity
    - Reduces pot-life
- OSI R&D Department was tasked to resolve the processing issue
  - Investigate variability
    - Why does the process yield good and not-so good material?
  - Systematic investigation of HMX Class 3 manufacturing process

Processing Issues and Goals

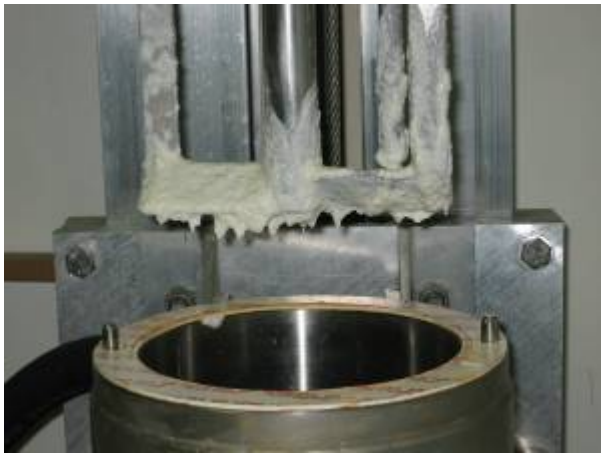
- End of Mix (EOM) Viscosity
  - High EOM Viscosity will affect processability during casting operation (shorter pot-life)
  - EOM viscosity usually depends on:
    - Solid Loadings (fixed)
    - Particle Coarse to Fine ratio (held constant at 3:1)
    - Particle Shape (main focus of this study)
    - Mix temperature and agitator speed (not studied)
    - Packing fraction (not studied)

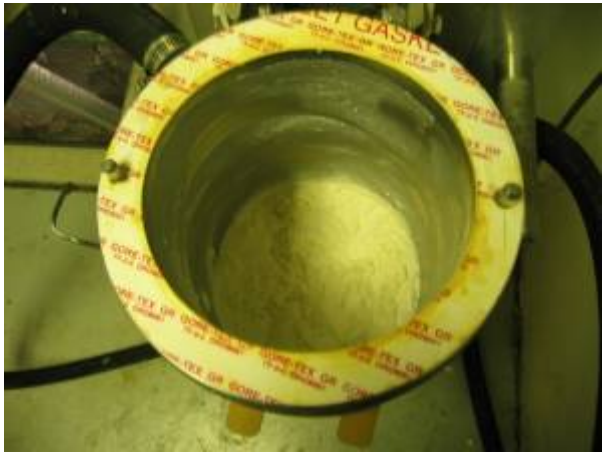




Processing Issues and Goals

- Our Goal – To evaluate the effect of particle shape on EOM viscosity
  - Determine key variables in current manufacturing process
    - How do they affect HMX Class 3 properties?
  - Assess the effect of various particle shapes on EOM viscosity
  - Test begins with small lab scale batches (2 lbs), to scale-up to production quantities (>1000 lbs)
  - Maintain particle size distribution requirement from the MIL-Spec.
  - Not change the manufacturing method for HMX Class 3 !
    - Qualification implications for process-change





Equipment – PBX Processing

- Low-shear anchor blade mixer
  - Capable of mixing and casting 10 lbs under vacuum at temperatures from ambient to 100°C
- OSI R&D evaluates intermediates (premix) for
  - PBXN-107, PBXN-109, PBXN-110, ROWANEX 1100, etc.

Equipment – EOM Viscosity Measurement

- Brookfield DV-II+ Viscometer
- Temperature controlled from ambient to 95°C
- Computer control and data acquisition for real-time viscosity measurement
- 500mL sample, T-C spindle, 1rpm, 60°C

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Equipment – Particle Size and Shape Analysis

- Malvern Laser Diffraction Particle Size Analyzer
  - Particle size distributions from 0.01µm to 3mm
- Motic Advanced Optical Microscope
  - Digital photomicrographs
  - Image enhancement and multiple measurement capabilities
- Wet Grist Analysis (not pictured)
  - For assurance of specification-grade material

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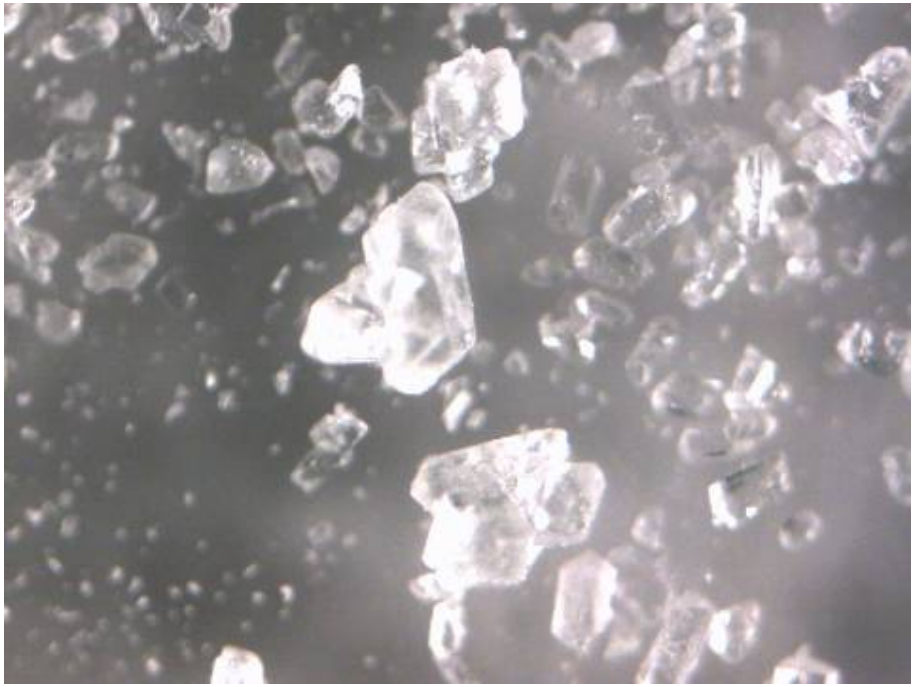
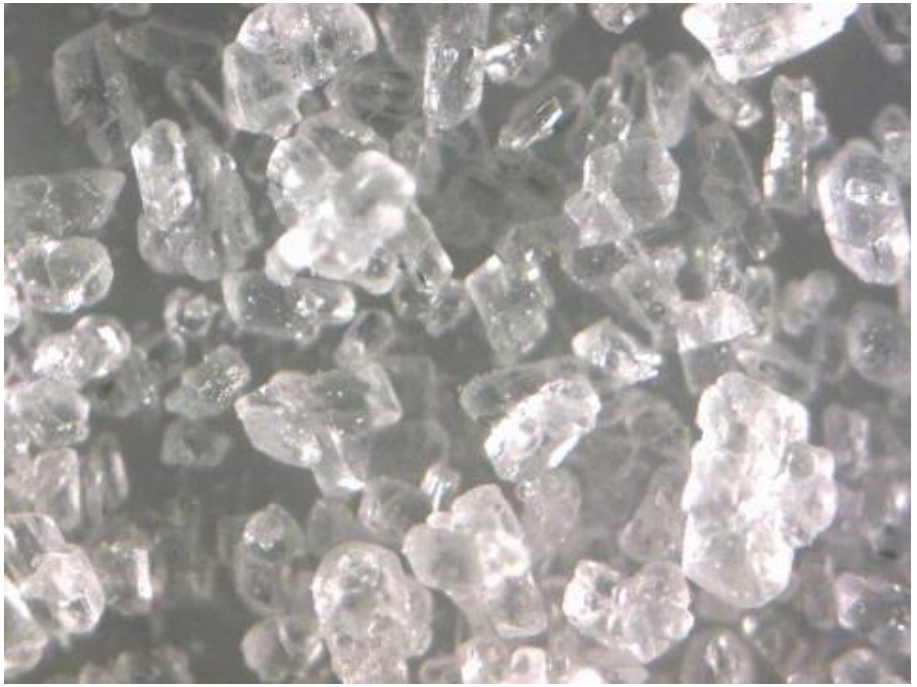
Initial Particle Shape and Processing Issues

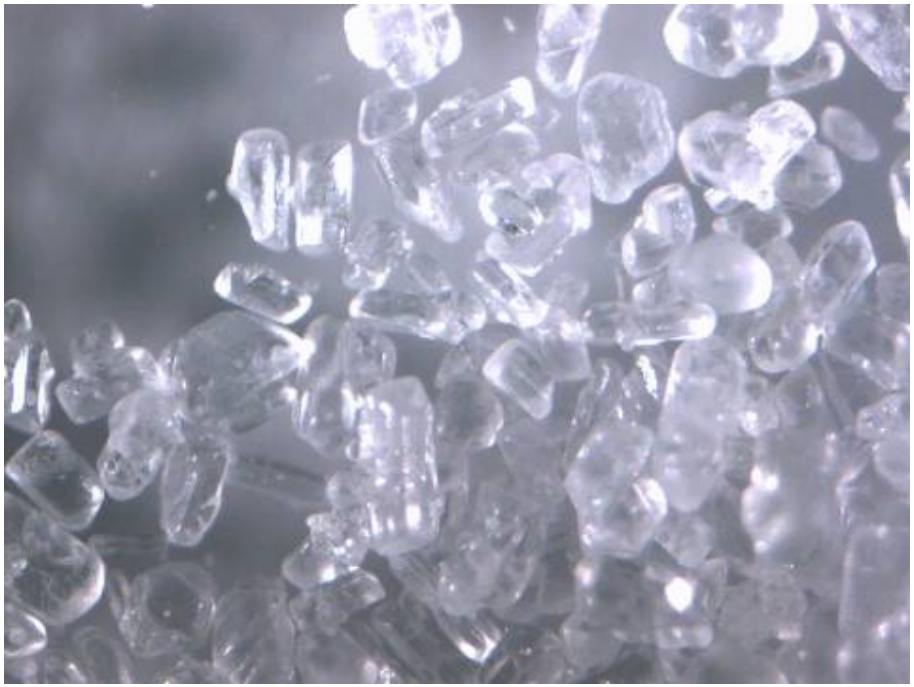




- Identified a batch of HMX Class 3 with poor processing characteristics
  - Very irregular and sharp-edged crystals
  - EOM Viscosity of PBX-N110 premix unrecordable (>1,000,000 cP*)
- OSI used this material as our baseline reference

** EOM viscosity considered adequate less than ≈500K cP*

Process Variable Investigation Trial #1



- Some smoothing and polishing observed
- EOM Viscosity of PBXN-110 premix : 480K – 630K cP
  - More than 50% improvement compared to reference HMX Class 3 material

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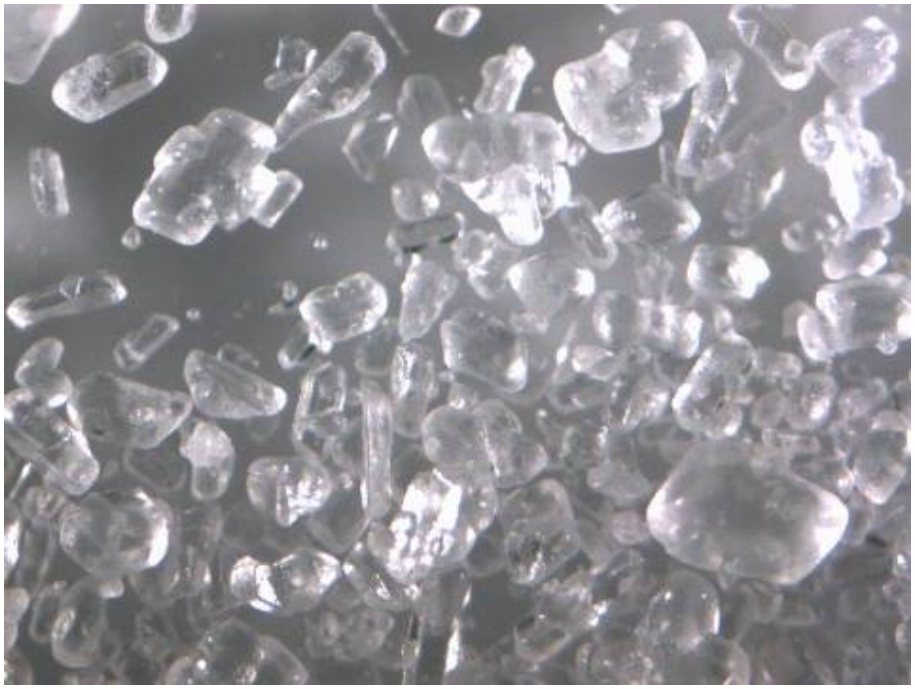
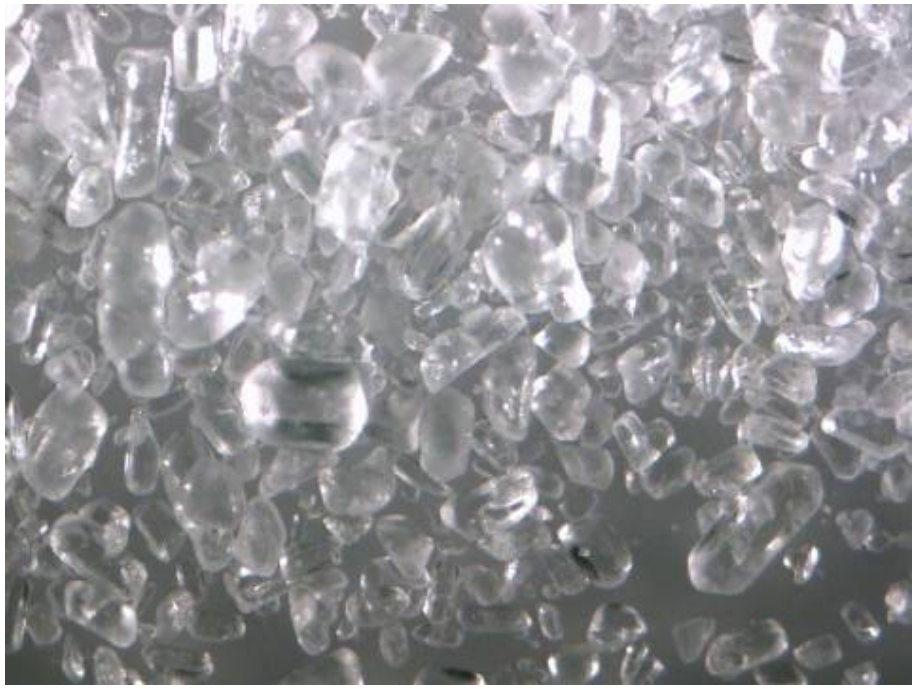
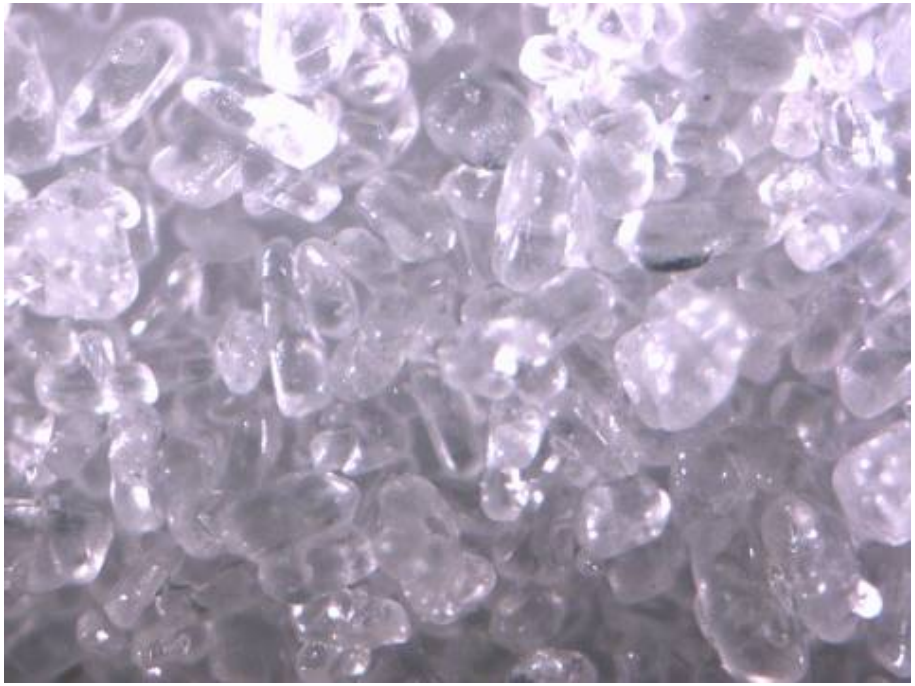
Process Variable Investigation Trial #2





- Some smoothing and polishing observed
- EOM Viscosity of PBXN-110 premix : 390K – 600K cP
  - Slight improvement compared to lab trial #1

Process Variable Investigation Trial #3



- More rounded and polished particles
- EOM Viscosity of PBXN-110 premix : 390K – 500K cP
  - Very similar to lab trial #2

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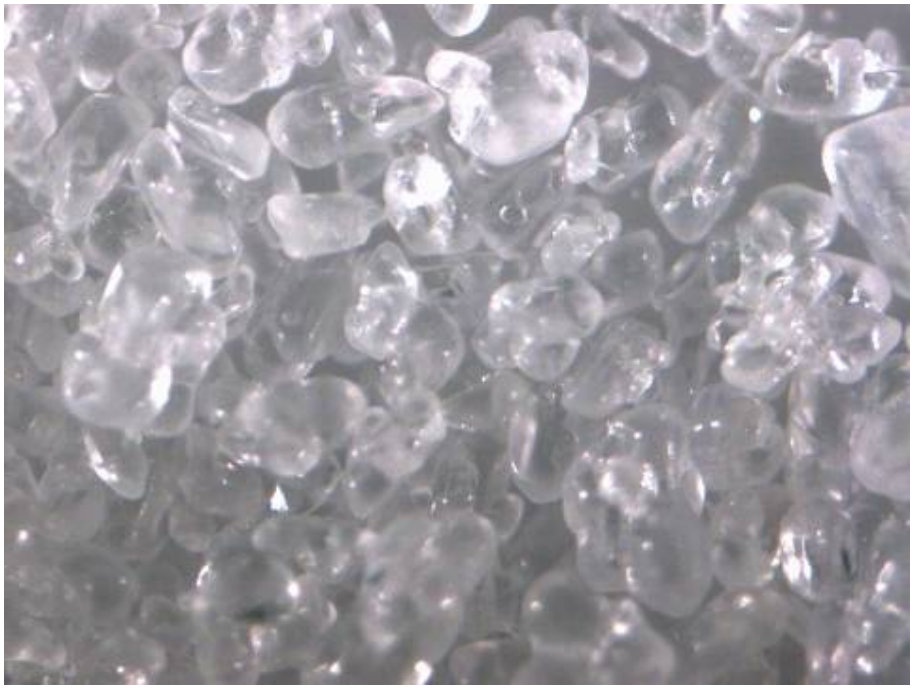
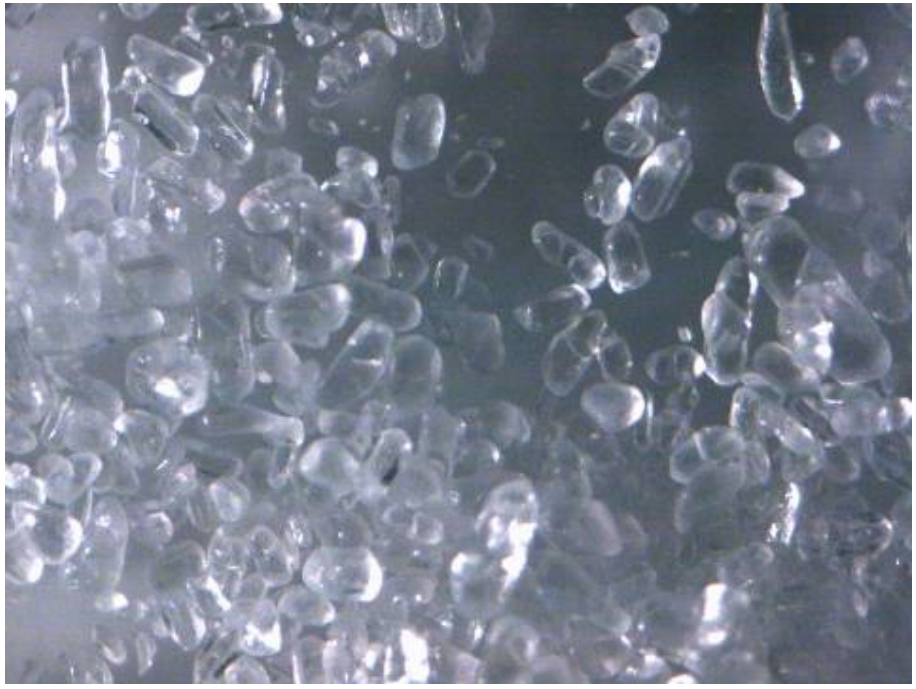
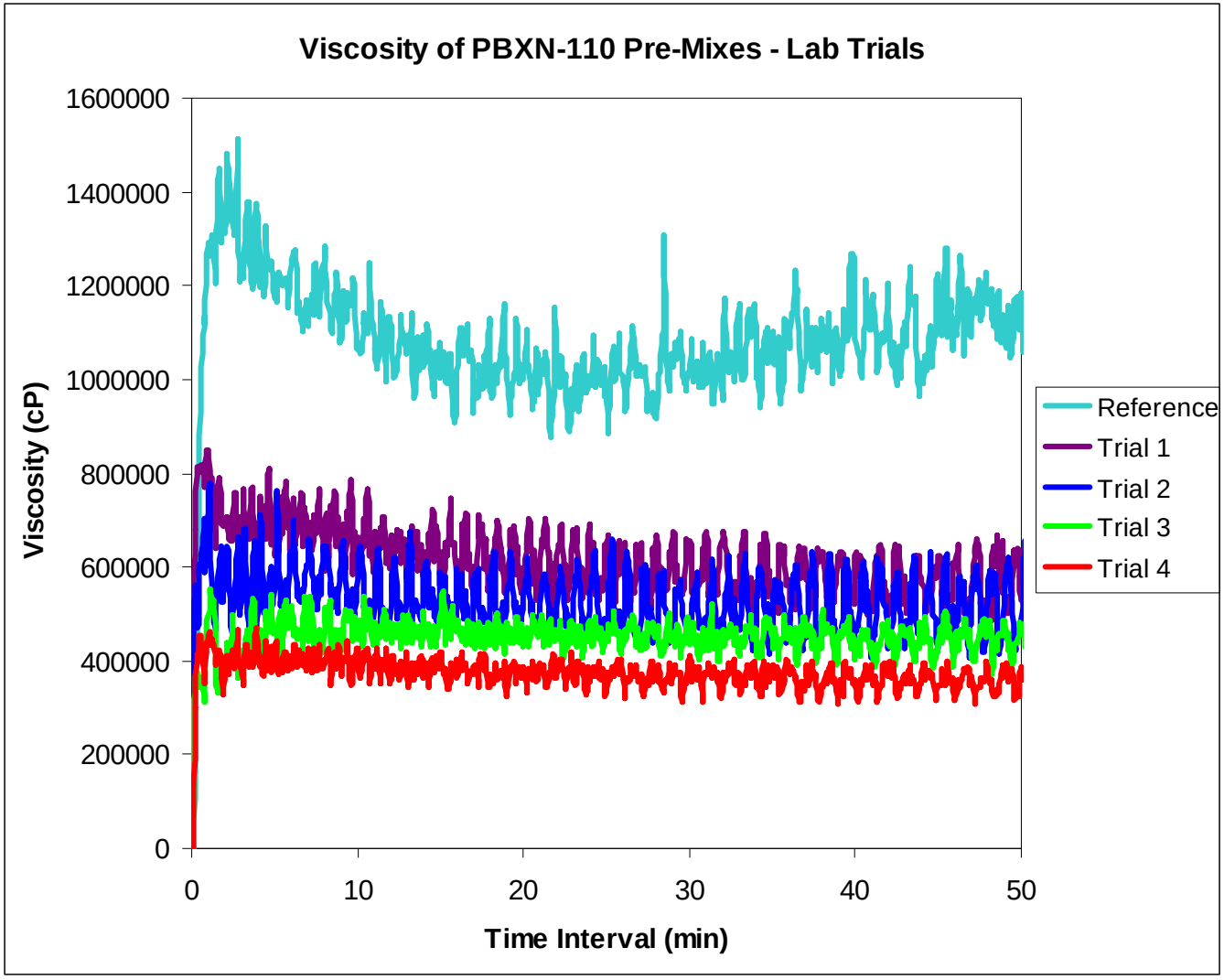
Process Variable Investigation Trial #4





- Significantly rounded particle shape observed
- EOM Viscosity of PBXN-110 premix : 300K – 370K cP
  - Further improvement compared to previous lab trials

Comparison of EOM Viscosities – Lab Trials



- The EOM viscosity varies significantly depending upon shape of particle produced in standard manufacturing process
- All lab trials produce viscosity profiles more stable than the reference material (less fluctuation)
- Particle size distribution of the material meets MIL-SPEC requirements

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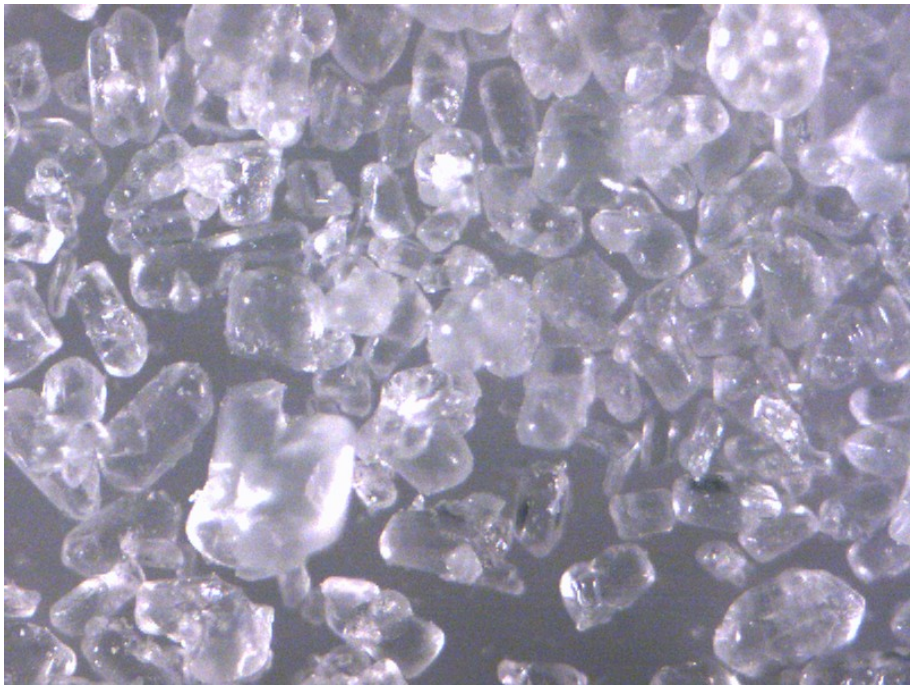
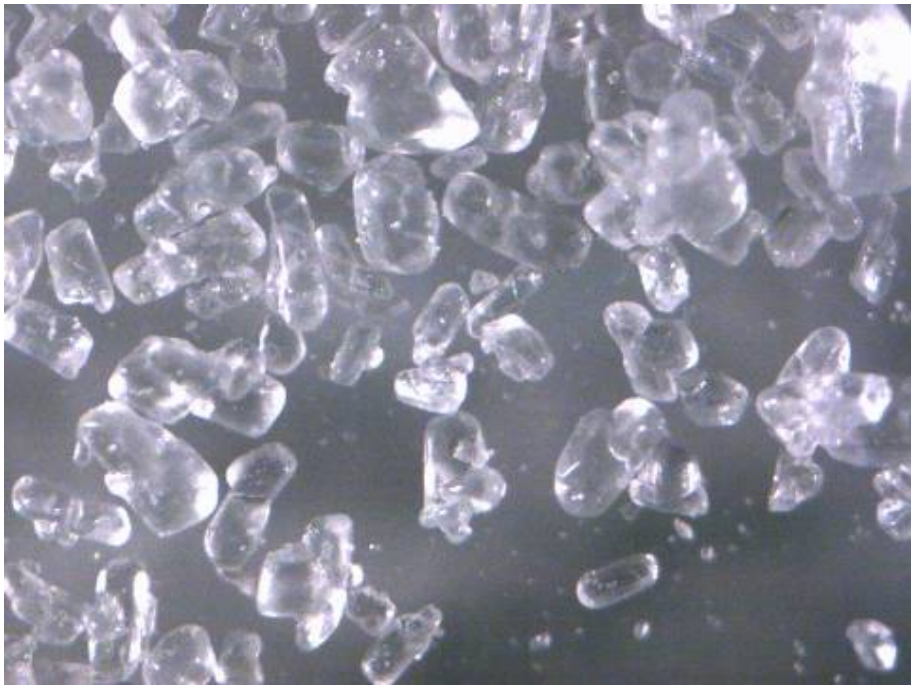
Production Process Focus





- 4200 lb Batch generated using standard, **qualified** HMX Class 3 Process
  - Target manufacturing process sweet-spot that produces more-spheroidal material
- Comparable to Lab Trial #2
  - EOM Viscosity of PBXN-110 premix: 319K – 459K cP; similar visual shape/size

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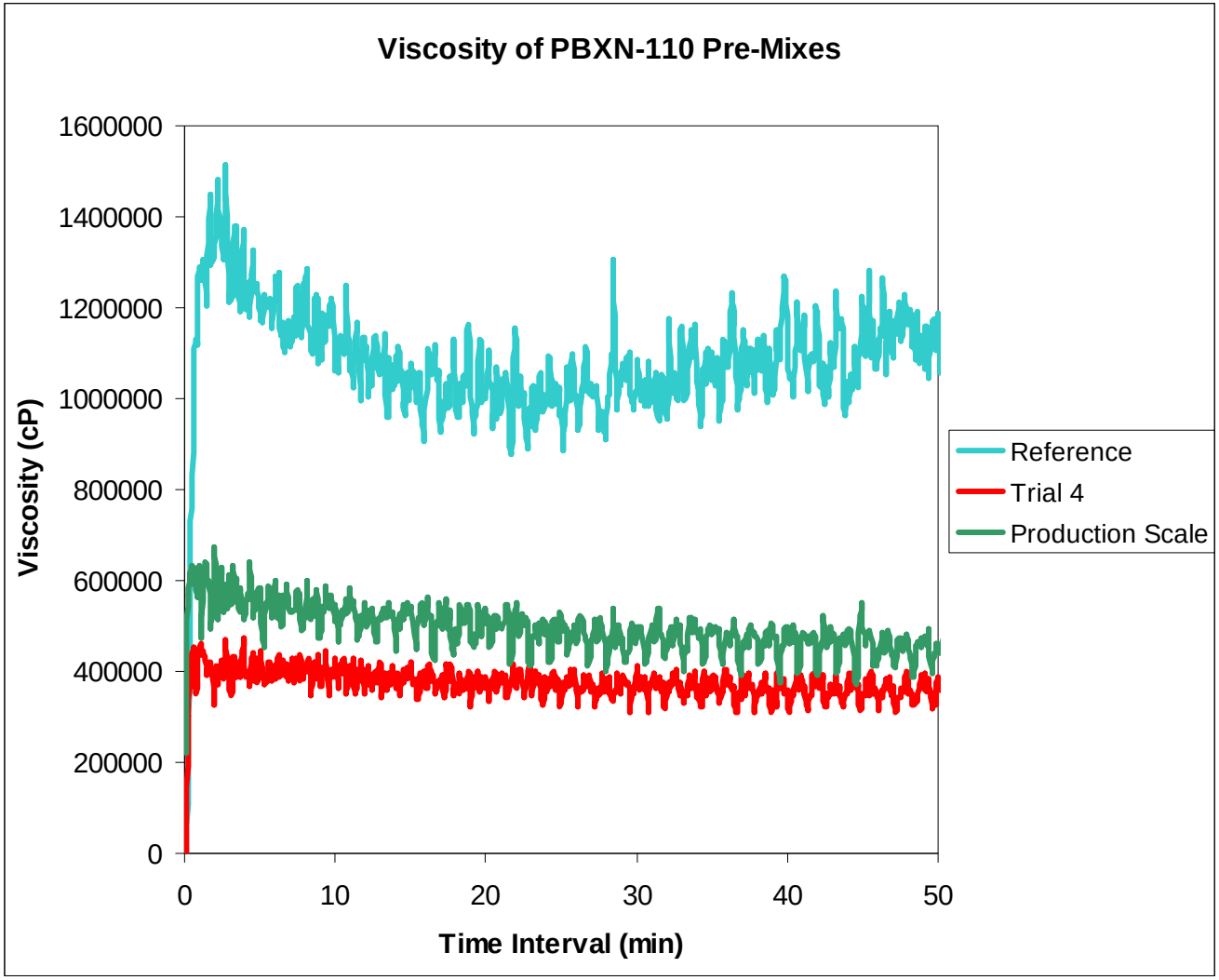
Comparison of EOM Viscosities – Lab to Production



- EOM viscosity significantly lower than reference particles (although not as low as lab trial #4)
- Overall, 30-40% reduction of EOM viscosity achieved by more rounded particles

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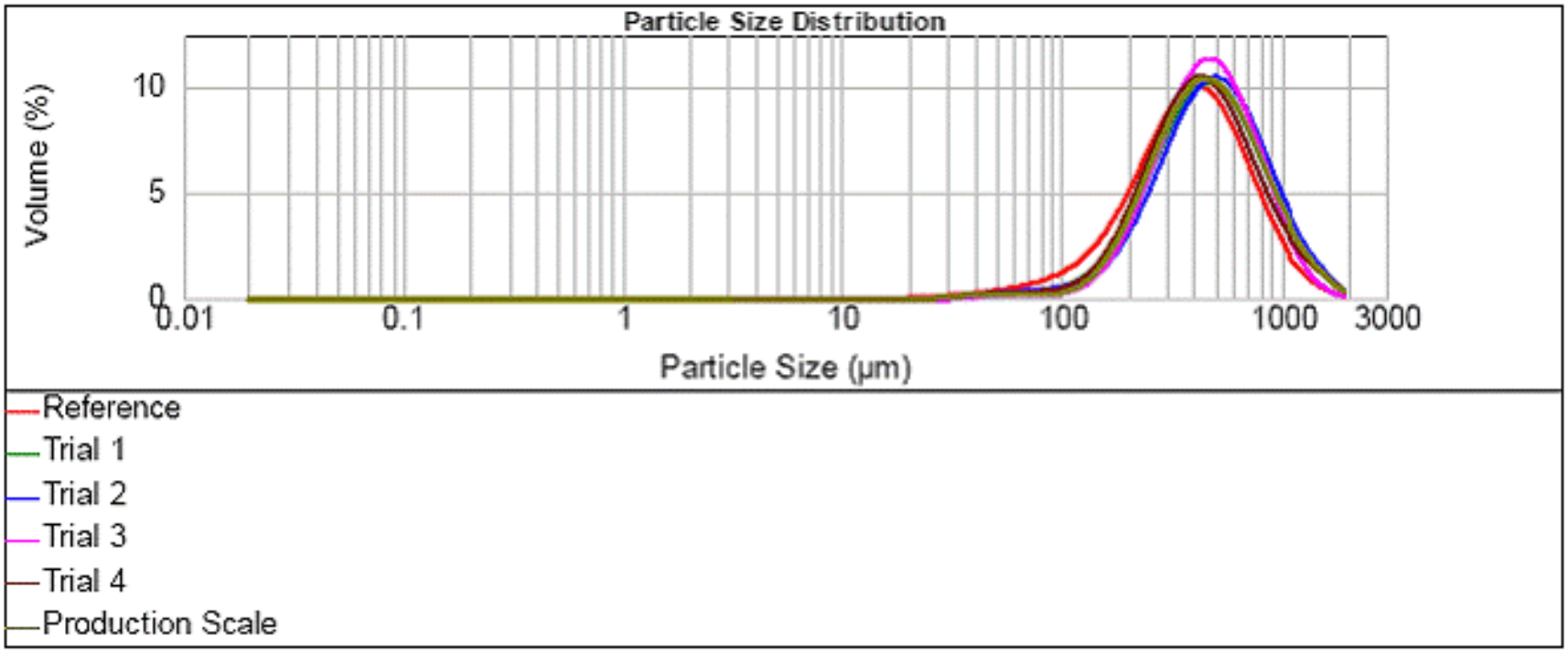
Particle Size Distribution

USSS#	CL3 Spec (%Pass)	Starting Material No Treatment	Lab Trials				Production Scale
			Trial 1	Trial 2	Trial 3	Trial 4	
12	99-100	100	100	100	100	100	100
20		100	98	98	99	99	95
50	25-55	53	52	49	54	53	54
100	10-30	15	16	16	14	12	14
200	0-20	5	4	5	5	4	6



Conclusions

- Standard manufacture of HMX Class 3
  - Sharp-edged, irregular crystals occasionally produced
    - High viscosity of PBX premixes
  - Spheroidal (to varying degrees) occasionally produced
    - Lower end-of-mix viscosity
- Targeting the sweet-spot of the HMX Class 3 Manufacturing Process
  - Results in manufacture of the desirable more-spheroidal HMX Class 3
  - Significantly lowers EOM viscosity in premixes of PBXN-110
    - 30-40% reduction in EOM viscosity compared to untreated crystals
    - Longer pot-life of PBXN-110 as a result
- This process investigation is also applicable to other recrystallized products used in the manufacture of cast-cured products