Production and Coating of Nano-RDX using Wet Milling

NDIA IM/EM

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Outline

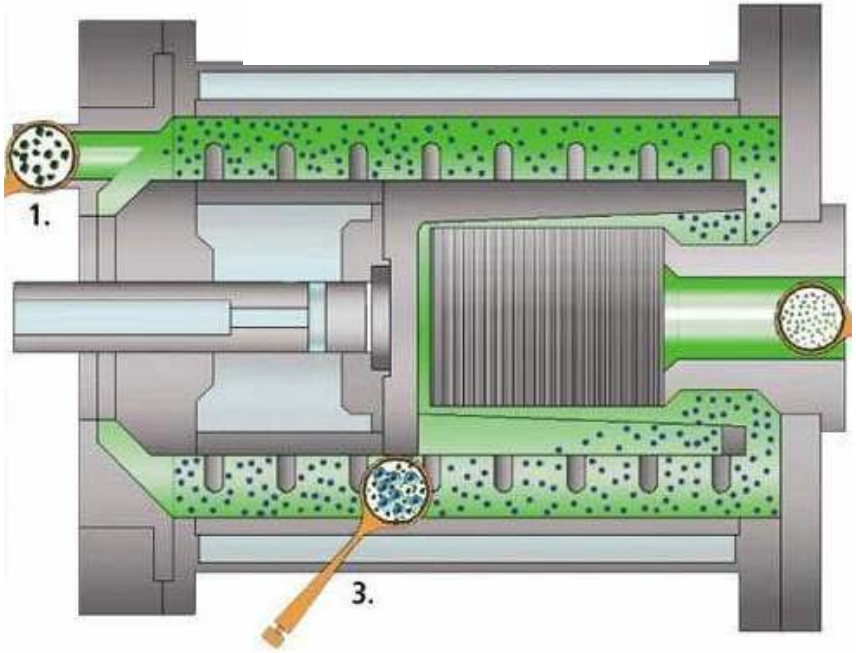
- Background
- Explanation of Wet Milling
- Particle Size Characterization
- Initial Problems
- Coating Process
- Conclusions
- Future Studies

Background to Nano-RDX

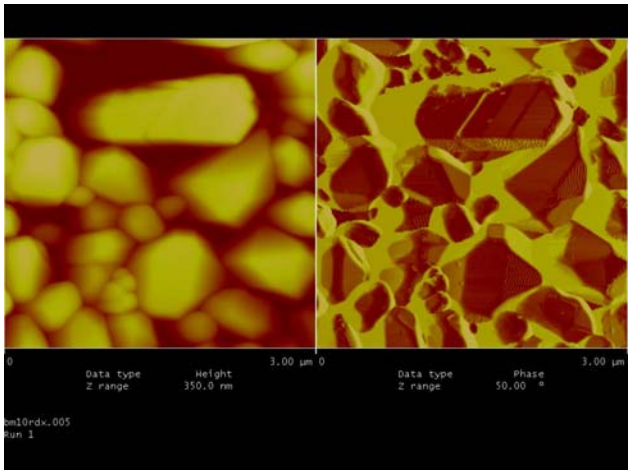
- Army has significant interest in developing insensitive munitions
- Smaller Particle Size for RDX causes Reductions in Sensitivity
- Many researchers are attempting to create Nano-RDX
- One successful method is wet milling

Wet Mill

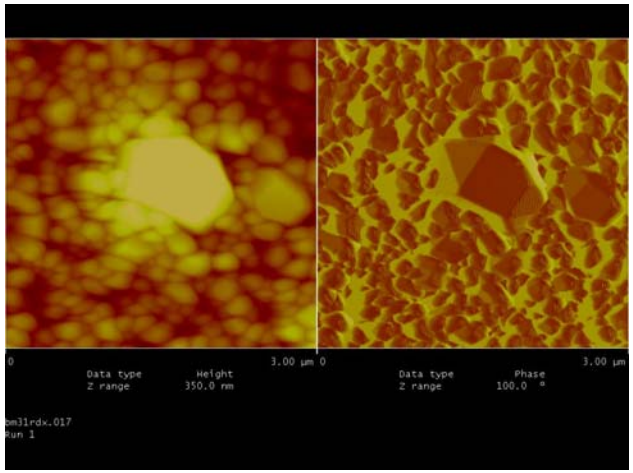
- A wet milling process can be used to quickly reduce micron sized particles



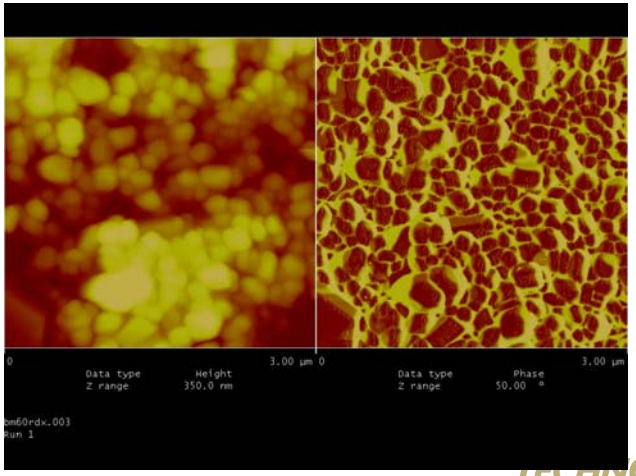
Atomic Force Microscopy



2000 rpm, T=10 minutes



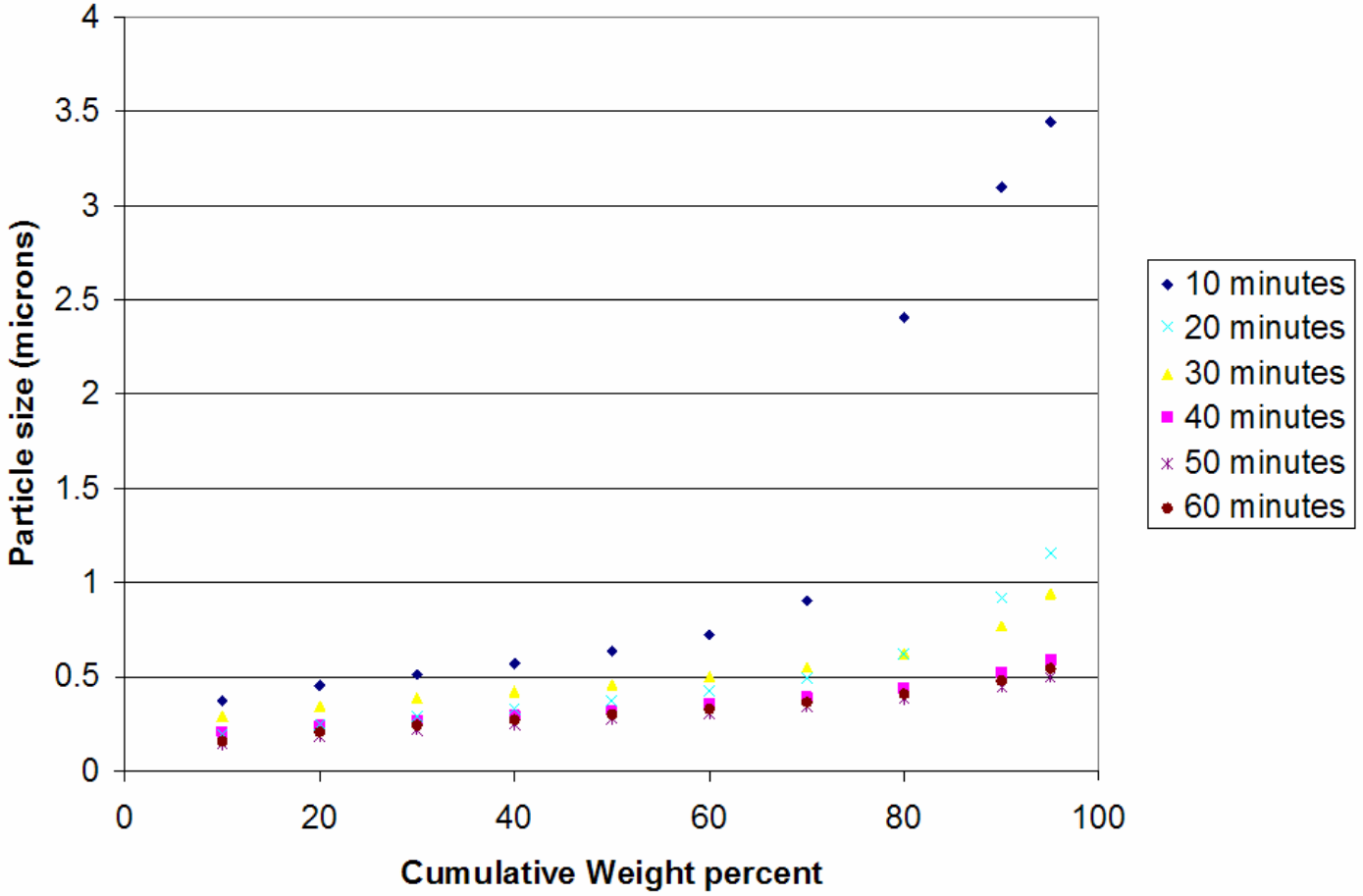
2000 rpm, T=30 minutes



2000 rpm, T=60 minutes

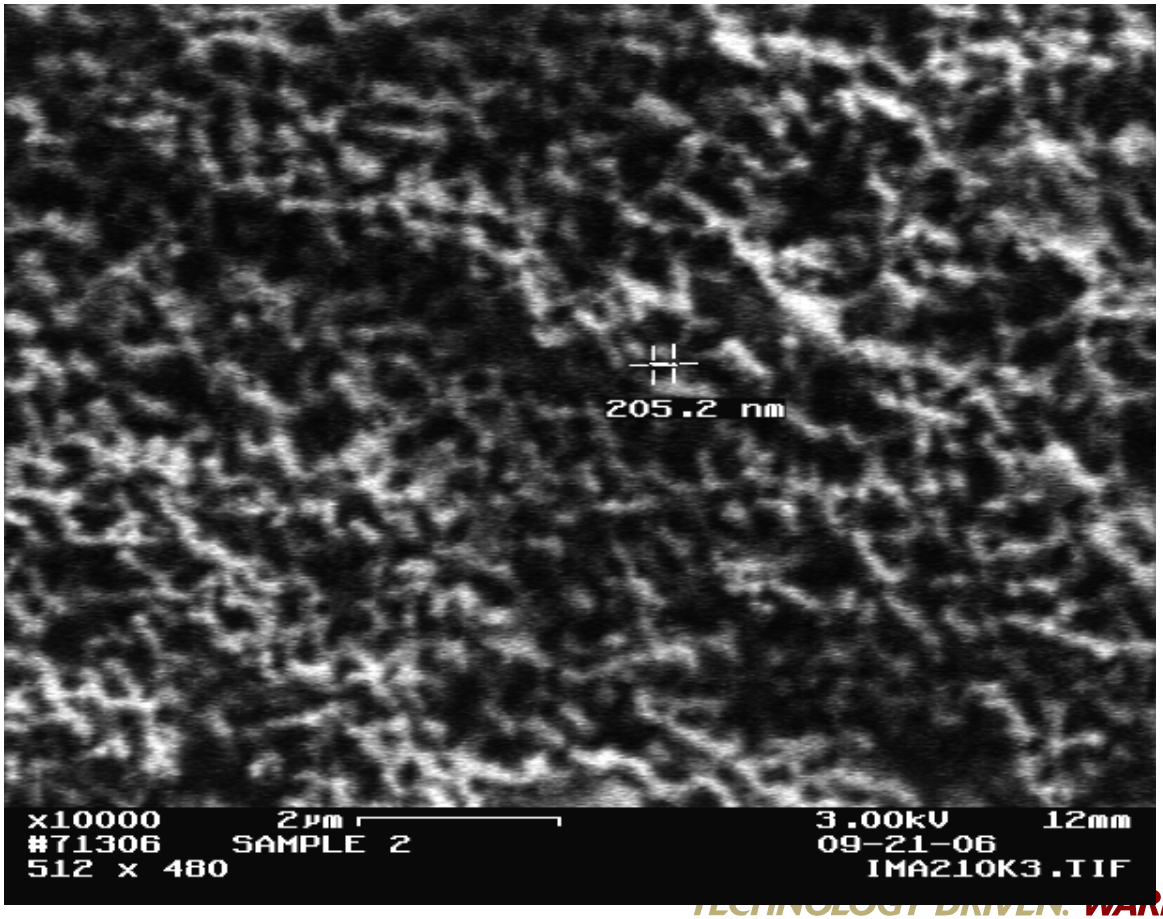
Particle Size Analysis

Analysis with Lecotrac light scattering device generally agreed with visual observation by AFM



Particle Size Analysis

Field Emission Scanning Electron Microscopy provided an additional check to the AFM and Lecotrac data



Problems with Initial Process

- Nano-RDX was difficult to filter out of solution

-Possible solution would to be to coat wetted RDX which comes from milling process and filter out coated particles

- Wet Milling Creates Solution which is well suited for coating

Materials Used in Milling Process

Run #	1	2	3	4	5	6	7
Water (g)	167.62	167.68	167.56	167.82	167.83	167.93	167.93
Isobutanol (g)	10.80	11.22	11.02	11	12.07	20.15	30.07
Triton X-100 (g)	1.42	1.37	1.57	1.7	1.53	1.5	1.5
RDX Class V (g)	19.95	20.01	20.11	20.52	20.66	20.07	20

- Past studies revealed most effective milling process to create Nano-RDX quickly

Lacquer Preparation

- Coating of Nano-RDX presents interesting challenge because of relatively high surface area
- A polar binder was chosen because of its theoretically high affinity for RDX
- Binder was dissolved in MEK to form a solution which was 20% binder by mass

Slurry Coating Process Run 1 and Run 2

- In these runs, the coating process failed
- 1 L still charged with material from the mill and 300 mL of deionized water
- Slurry was heated with a circulating bath to 50 C with 300 rpm agitation
- Lacquer solution (5 g) was added slowly over a 4 minute period.
- The material could not be filtered out.

Slurry Coating Process Run 3, 4, 5, 6, and 7

Slurry Coating Process (Run 4)

- 30 g of lacquer was used. The product was vacuum filtered to provide a powder and a clear, colorless filtrate.

Slurry Coating Process (Run 5 and Run 6)

- Same as Run 4 except 15 g of lacquer was used.

Slurry Coating Process (Run 3 and 7)

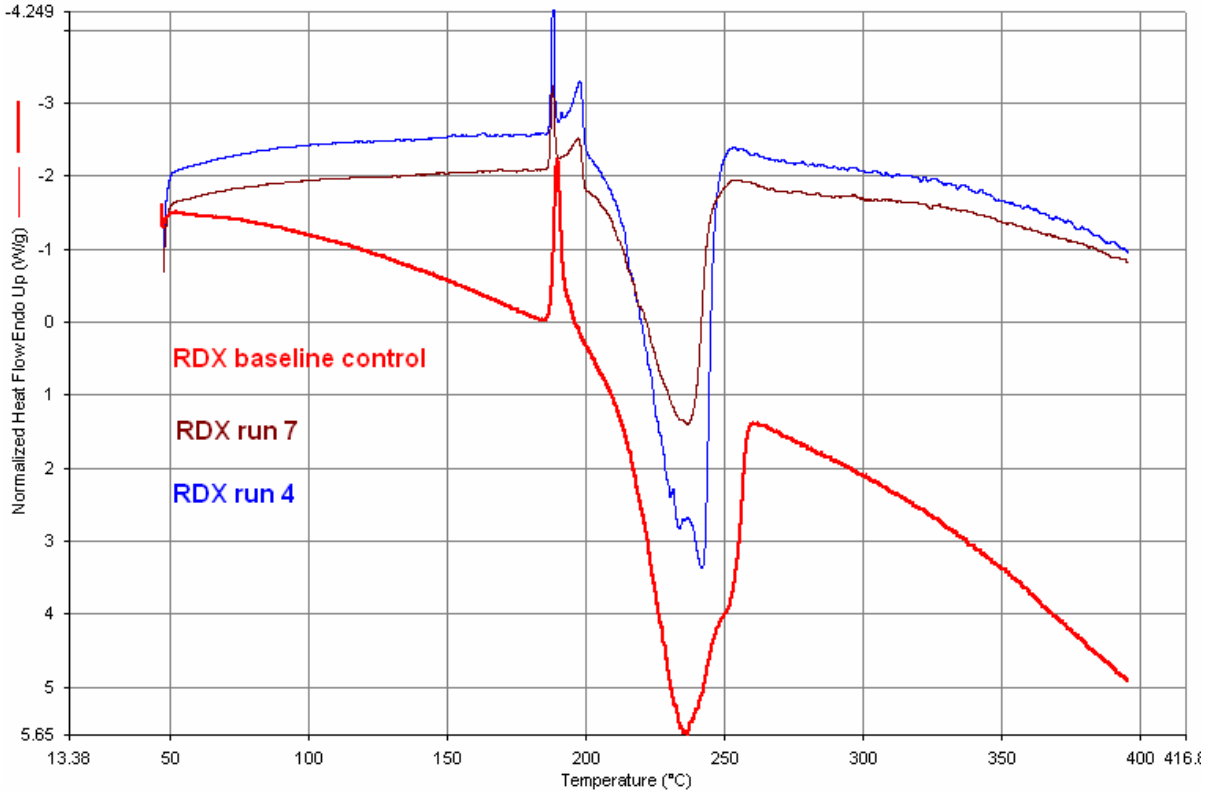
- These runs were the same as Run 5 and Run 6 except after agitation was stopped, the circulating bath temperature was turned down to 30 C and pressure was reduced to remove solvent for 15 minutes, lowering the reaction temperature to 37 C. The product was vacuum filtered to provide a powder and a clear, colorless filtrate.

HPLC Analysis

- HPLC analysis was performed to determine %RDX
- Results indicate Runs 5, 6, and 7 have close to what should be the theoretical amount of RDX (87%).

Sample ID RDX	
	RDX
	HPLC
Solid Run 3	59.2
Solid Run 4	67.6
Solid Run 5	87.7
Solid Run 6	83.0
Solid Run 7	82.9

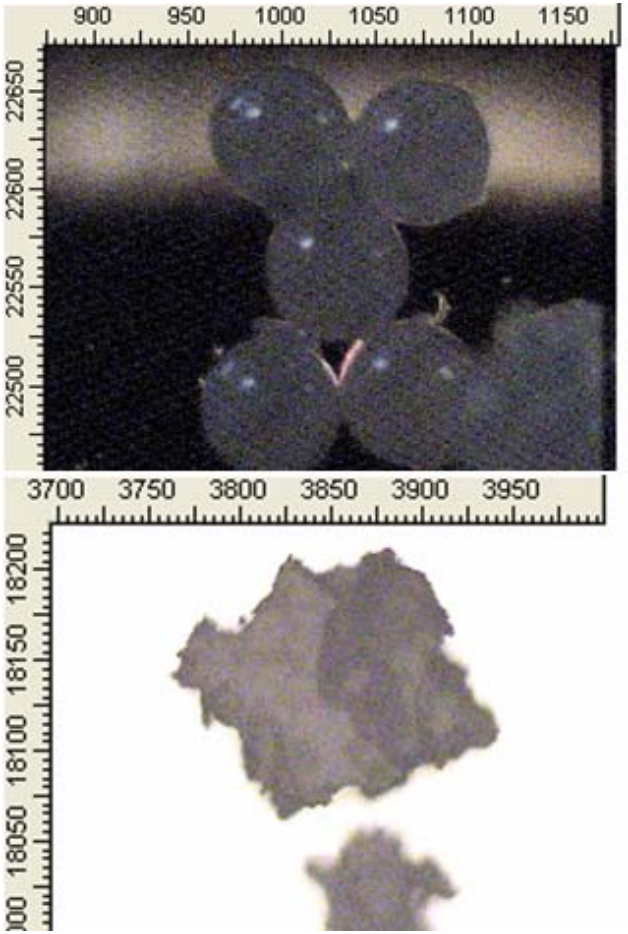
DSC Analysis of Coated RDX



- DSC's show coating causes little changes on thermal properties. Onset is little changed.

Optical Microscopy of Coated RDX

- Top image is of Run 3, where the coating was mostly on the surfactant
- Bottom image is of Run 7



Small-Scale Gap Tests with Coated RDX

In order to provide further testing of sensitivity as well test consistency of product, process repeated multiple times and small scale gap tests were performed.

Lot	Result (dBg)	Class 5 RDX (15% wax)
R8	6.063	5.526
R9	6.313	
R10	6.438	
R11	6.688	
R12	6.688	

Conclusions

- Nano-RDX from the wet milling can be coated effectively without adverse effects on its thermal properties
- Nano-RDX can be created using wet milling and coated to cause further reductions in sensitivity
- The Nano-RDX produced is reasonably consistent
  - Sensitivity
  - Particle size distribution

Future Studies

- Creation of Nano-HMX and other Nano-Energetics
- Larger Scale Sensitivity Testing
- Electron microscopy and surface area analysis of Nano-RDX to determine true particle sizes
- Testing with more Binders

Summary

- Coated Nano-RDX presents an interesting opportunity to gain IM properties
- Further experimentation is needed to optimize the process
- Future experimentation will provide a clearer view of the future of the nano-energetics created through wet milling

References

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- Stepanov, V., A. Di Stasio, Yakovlev, Yakovlev, S., Yi-Feng Su, Woo Lee, and Libera M. *High-Resolution Spectroscopic Imaging of Wax-Coated RDX Nanoparticles* ARDEC Technical Report.

- Stepanov, V., L.N. Krasnoperov, I.B. Elkina, and X. Zhang, *Production of Nanocrystalline RDX by Rapid Expansion of Supercritical Solutions.* Propellants, Explosives, Pyrotechnics 2005. **30**: p. 178-183.

- Stepanov, V., V. Anglade, A. Bezmelnitsyn, and L. Krasnoperov. *Production and Characterization of Nanocrystalline Explosive RDX*. in *AIChE Annual Meeting*. 2006. San Francisco.