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# Critical detonation thickness in vapor-deposited hexanitroazobenzene (HNAB) films with different preparation conditions

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# Introduction

- Detonation failure
  - Occurs when size (diameter or thickness) of explosive is decreased
  - When losses to confinement dominate behavior
- Data for small-scale behavior of high-density pure explosives are scarce
  - Difficult to prepare small-scale samples
  - Failure length scales are often sub-millimeter

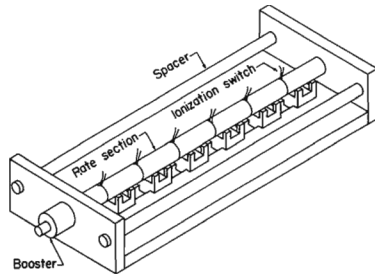
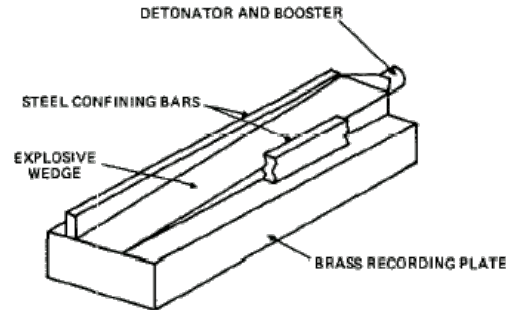
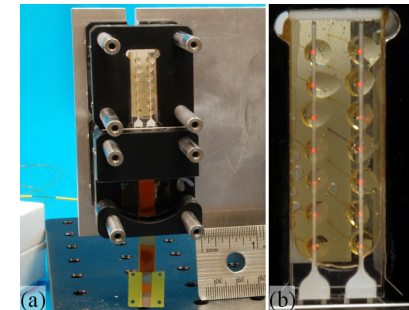


Fig. 1. Schematic of a typical rate-stick assembly.

## Rate stick experiment.



## Detonation failure experiment.



## Critical thickness experiment.

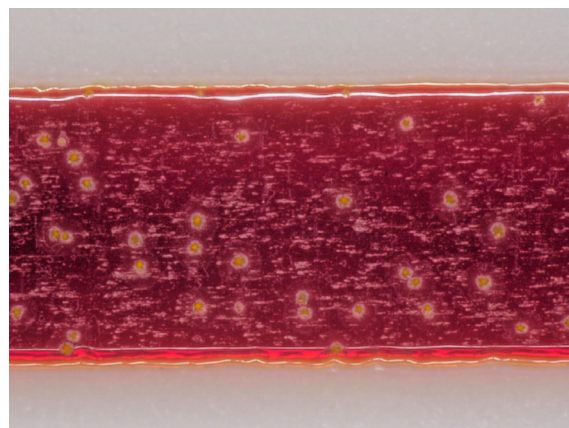
Campbell, A.W. and Engelke, R., "The Diameter Effect in High-Density Heterogeneous Explosives," 6th Symposium (International) on Detonation, Coronado, CA, August 24-27, 1976, pp. 642-652.

Gibbs, T.R. and Popolato, A., LASL Explosive Property Data, Detonation Failure Thickness, pp. 289-290. Berkeley, Los Angeles, London: University of California Press, 1980.

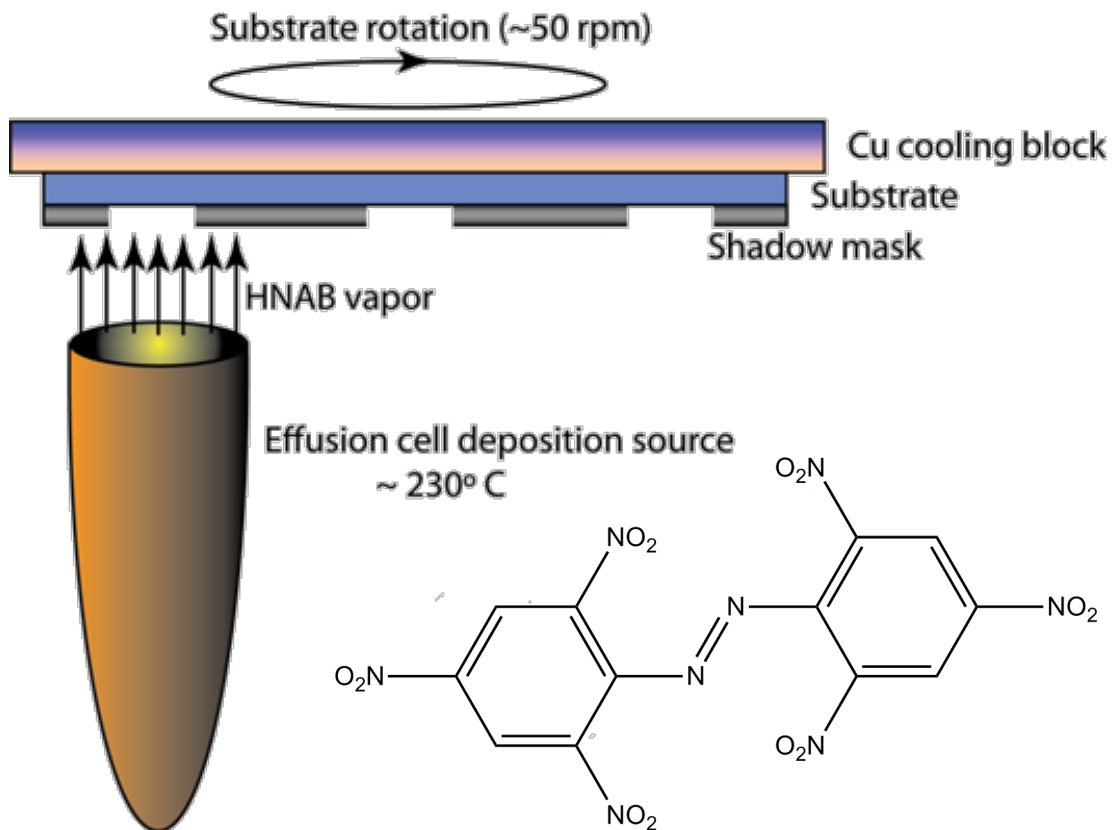
Tappan, A.S., Knepper, R., Wixom, R.R., Marquez, M.P., Miller, J.C., and Ball, J.P., "Critical Thickness Measurements in Vapor-Deposited Pentaerythritol Tetranitrate (PETN) Films," 14th International Detonation Symposium, Coeur d'Alene, ID, April 11-16, 2010.

# HNAB physical vapor deposition

- Polycarbonate substrates
- Amorphous HNAB films
- 100% dense

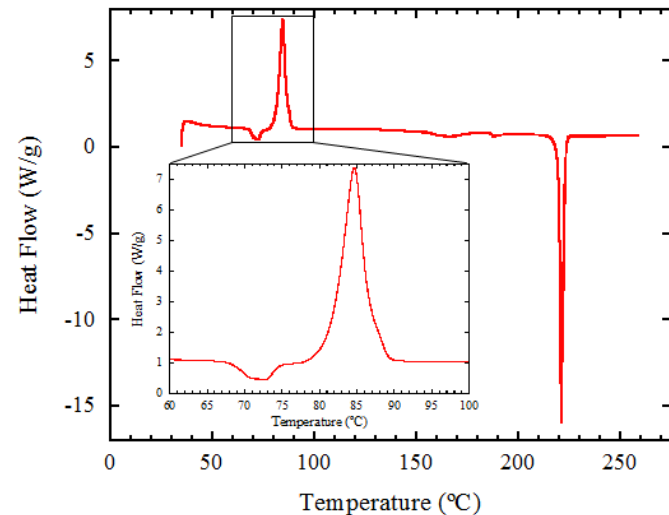
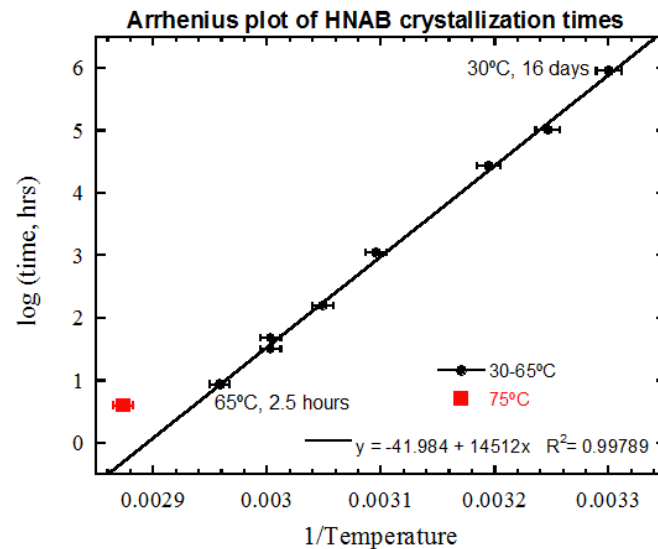


**Optical micrograph  
of as-deposited  
HNAB.**

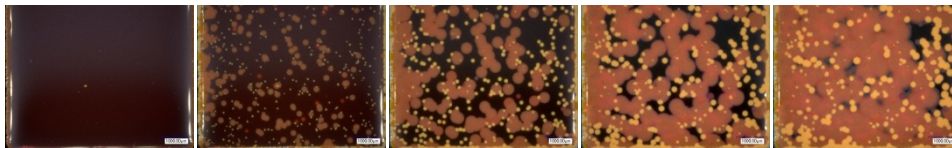


# HNAB crystallization

- Amorphous HNAB films crystallize over time
- Pronounced difference in crystallization above glass transition temperature ( $\sim 70\text{--}75^\circ\text{C}$ )



**DSC data from an amorphous HNAB film heated from 40–250°C at 5°C/min.**



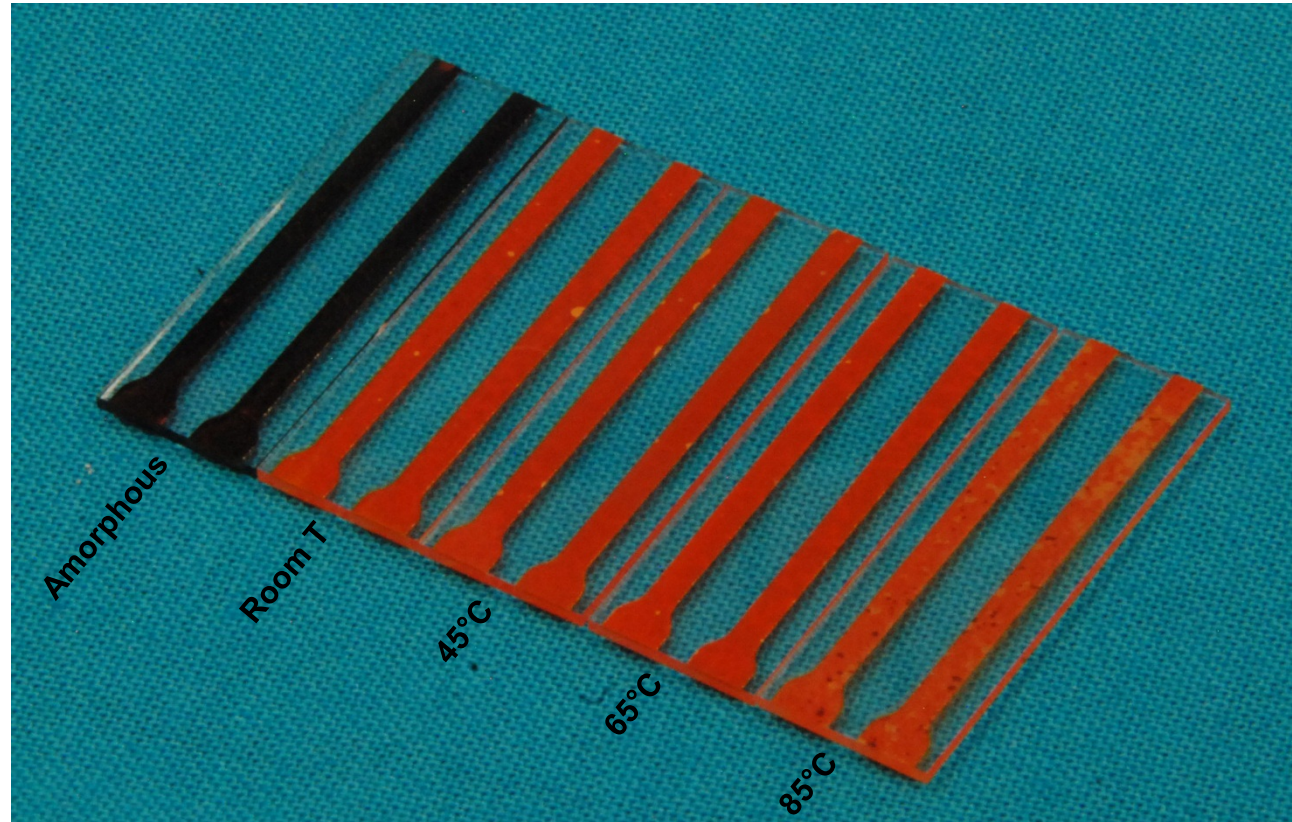
**Time-lapse HNAB crystallization, 65°C, 24 min./image.**

Knepper, R., Browning, K., Wixom, R.R., Tappan, A.S., Rodriguez, M.A., and Alam, M.K., "Microstructure Evolution during Crystallization of Vapor-Deposited Hexanitroazobenzene Films," *Propellants, Explosives, Pyrotechnics*, vol. 37, pp. 459 – 467, 2012.



# HNAB samples

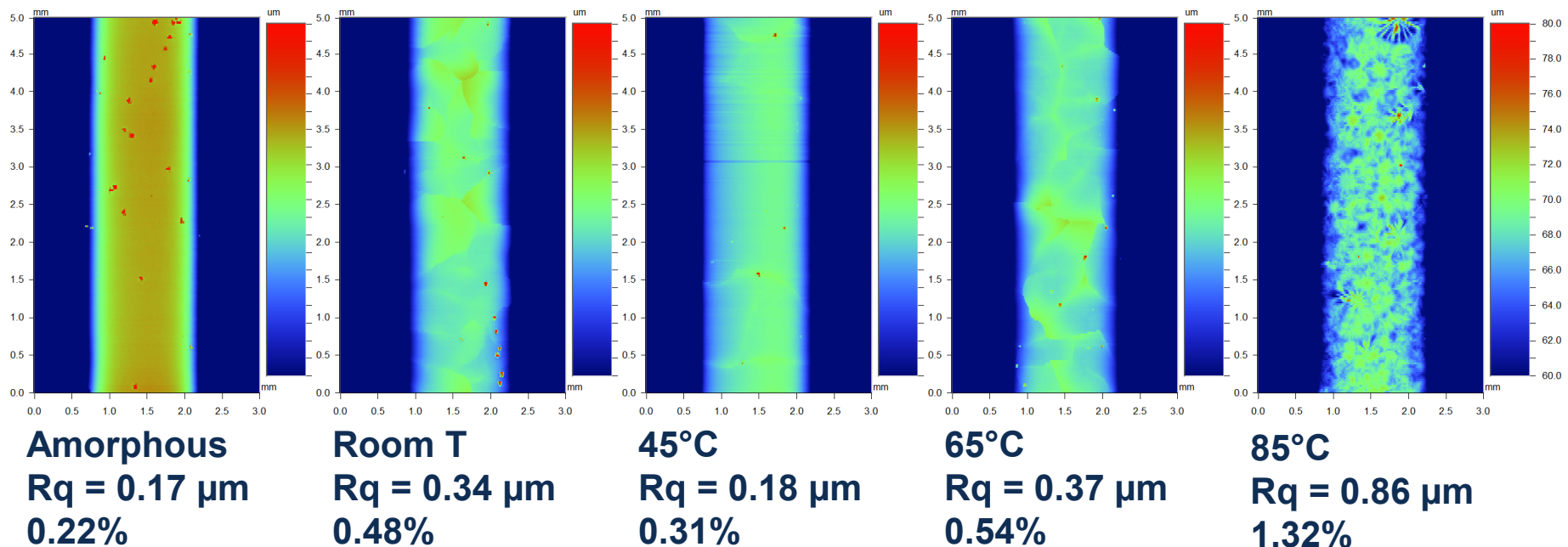
- Polycarbonate substrates
- Four crystallization conditions
  - Amorphous
  - Room temperature
  - 65° C
  - 85° C
- 45° C condition not tested



**Photograph of five HNAB films on polycarbonate. Amorphous film on left and increasing crystallization temperature to right.**

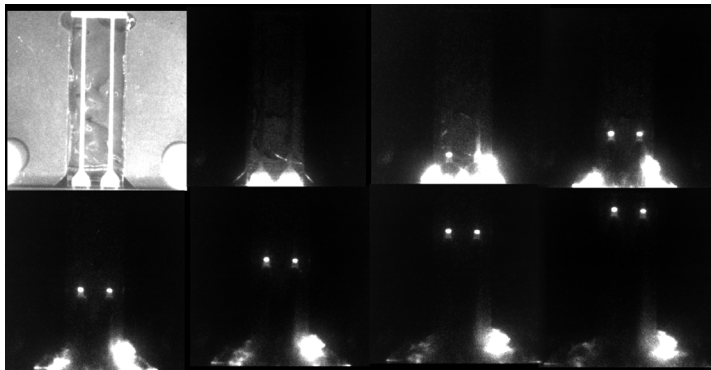
# Surface profiler measurements

- Root mean squared surface roughness ( $R_q$ )
  - Amorphous films are smooth with a low  $R_q$
  - Films crystallized below glass transition temperature ( $\sim 70\text{--}75^\circ\text{C}$ ) have crack networks and slightly higher  $R_q$
  - Film crystallized above  $T_g$  higher  $R_q$

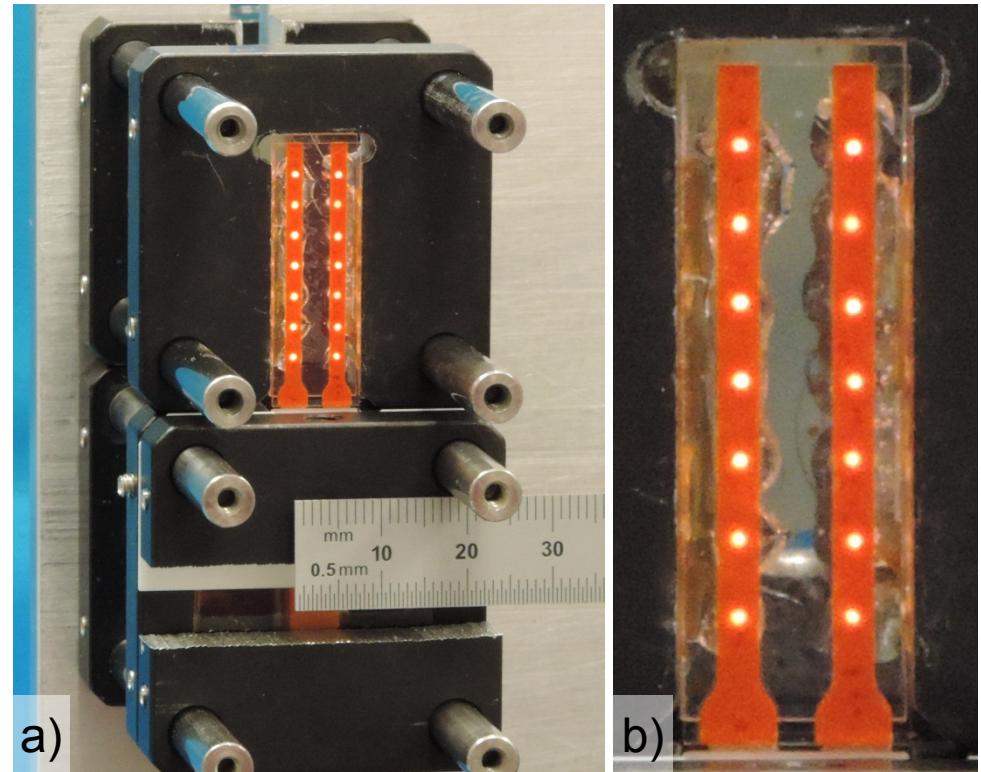


# Critical detonation thickness experiment

- Two experiments (HNAB lines) each shot
- Optical fibers deliver detonation light to photodetector



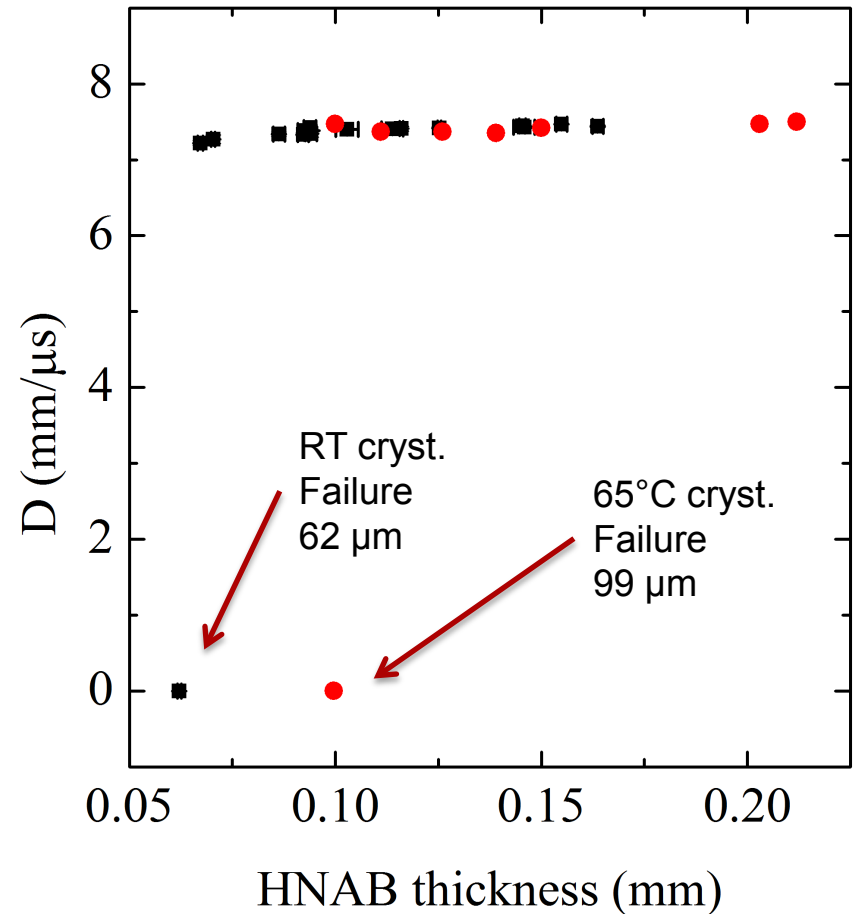
**Framing camera images of critical thickness experiment (PETN). 1.67 Mfps (1/600 ns), 20 ns exposure time.**



**Photographs of critical detonation thickness experiment. Optical fibers illuminated to highlight position.**

# Critical detonation thickness for room temperature and 65° C crystallized HNAB

- Detonation velocity relatively consistent up to failure
- Room temperature crystallized HNAB fails at a thickness of about 62  $\mu\text{m}$
- 65° C crystallized HNAB fails at a thickness of about 99  $\mu\text{m}$



**Detonation velocity versus thickness. One failure point is included.**

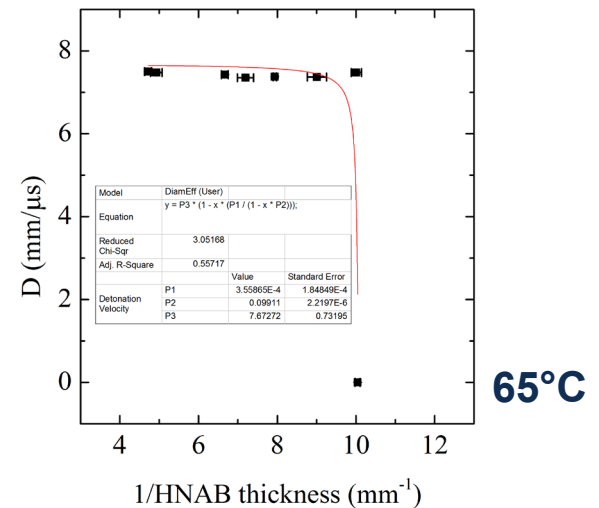
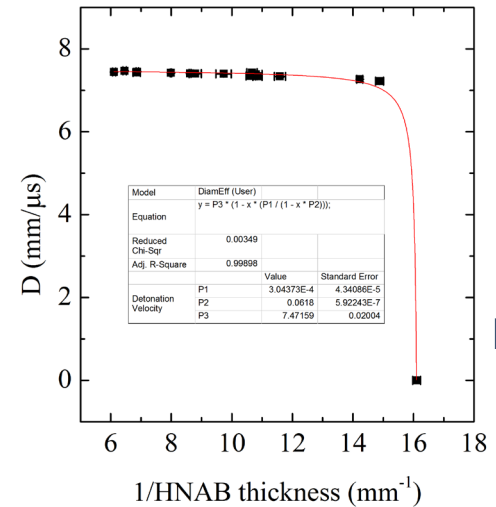


# Critical detonation thickness for room temperature and 65° C crystallized HNAB

- Analysis of critical thickness data is performed in an analogous fashion to Campbell and Engelke (1976)

$$D(t) = D(\infty) \left[ 1 - A / (t - t_c) \right]$$

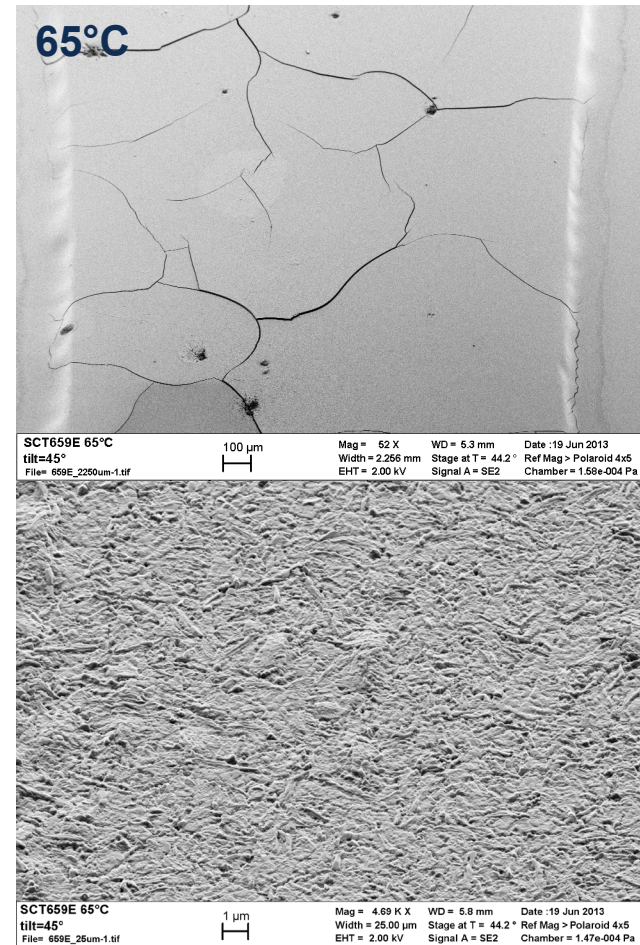
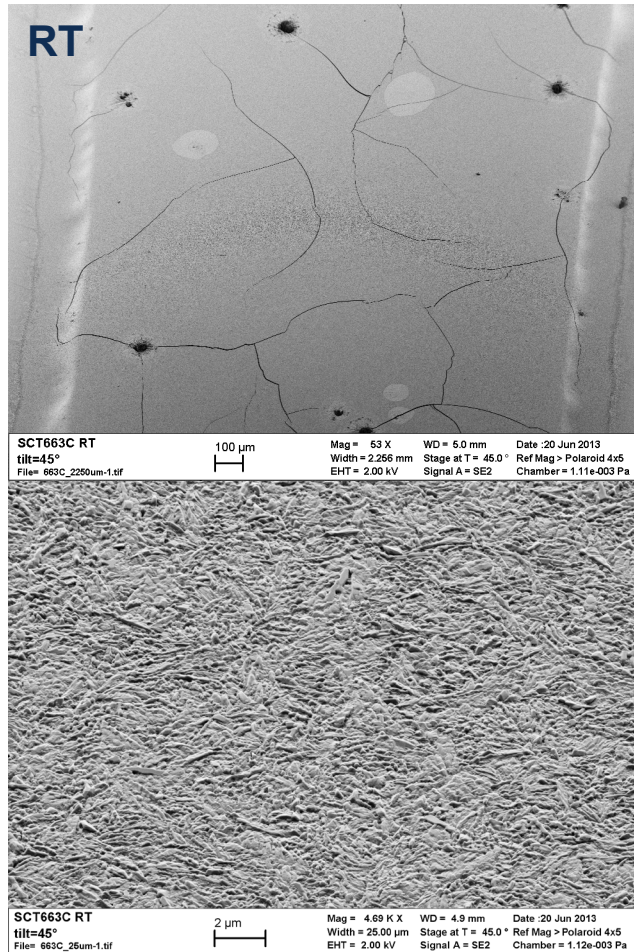
Condition	Detonation Velocity at Infinite Thickness, $D(\infty)$	Critical Thickness, $t_c$	Length Parameter, $A$
	mm/ $\mu$ s	mm	mm
Room T	$7.47 \pm 0.02$	$0.062 \pm 0.000$	$0.0003 \pm 0.00004$
65° C	$7.67 \pm 0.73$	$0.099 \pm 0.000$	$0.0004 \pm 0.0002$



Campbell, A.W. and Engelke, R., "The Diameter Effect in High-Density Heterogeneous Explosives," 6th Symposium (International) on Detonation, Coronado, CA, August 24-27, 1976, pp. 642-652.

# Top surface microscopy of room temperature and 65° C crystallized HNAB

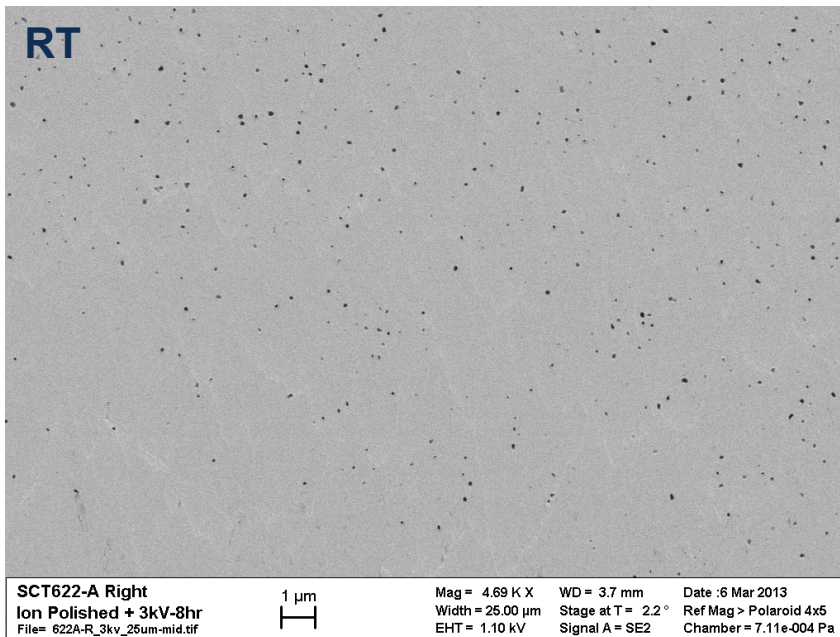
- Similar top surface appearance



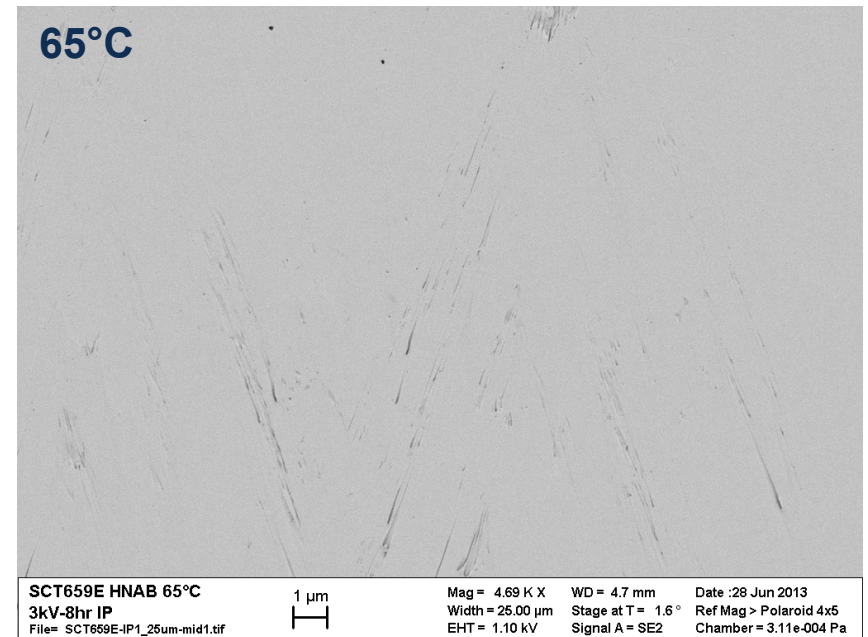


# Ion-polished cross-sections of room temperature and 65° C crystallized HNAB

- Pores (~ 100 nm) are distributed throughout room temperature crystallized HNAB, but not in 65° C crystallized HNAB



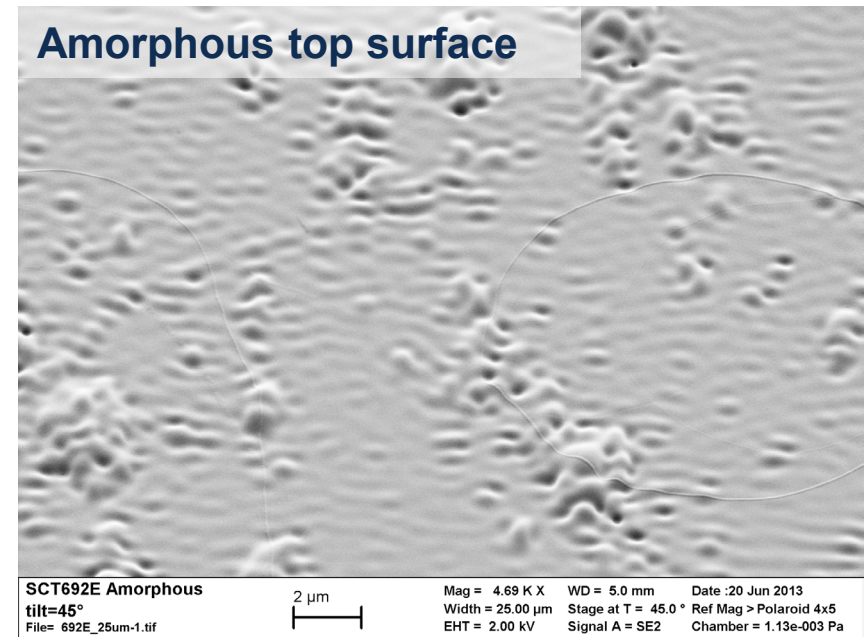
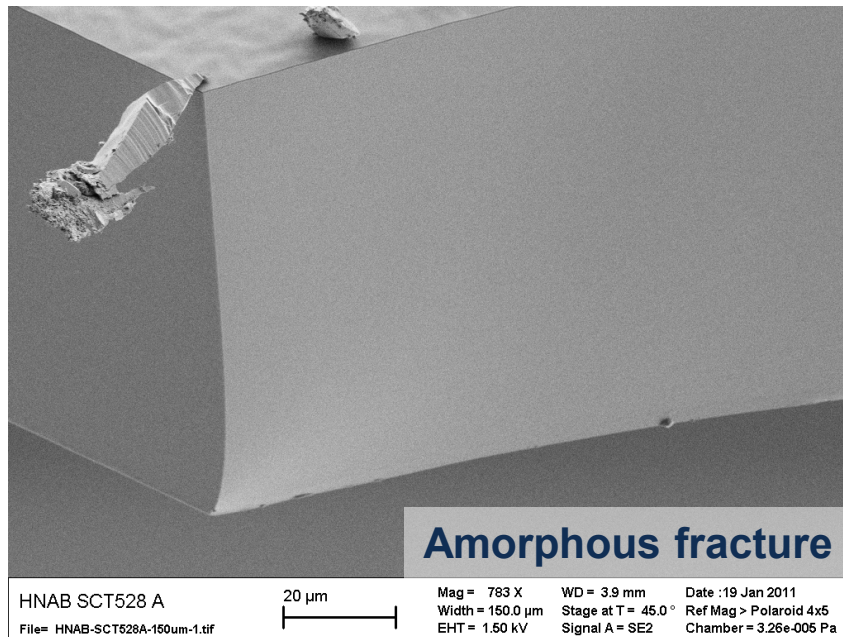
**Scanning electron micrograph of ion polished room temperature crystallized HNAB. Image width 25.00 μm.**



**Scanning electron micrograph of ion polished 65°C crystallized HNAB. Image width 25.00 μm.**

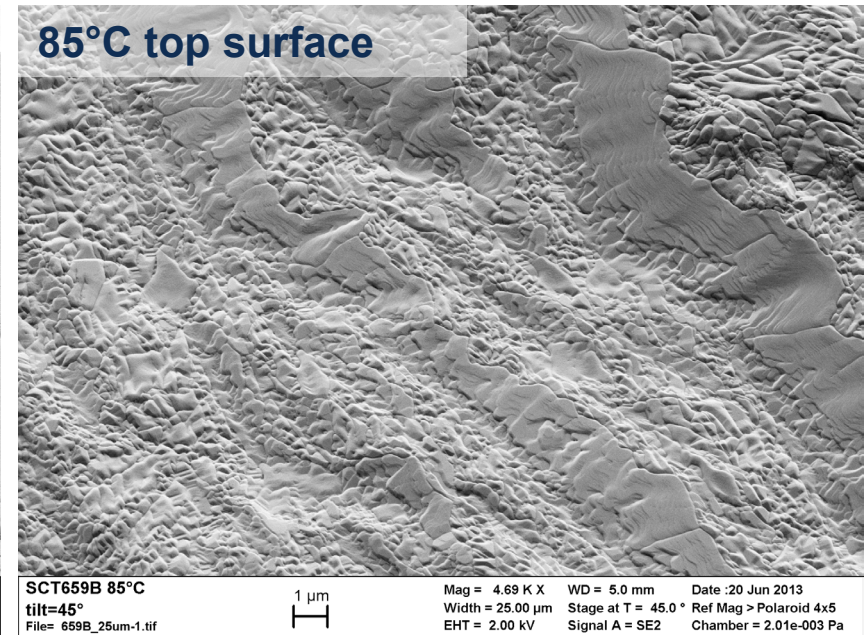
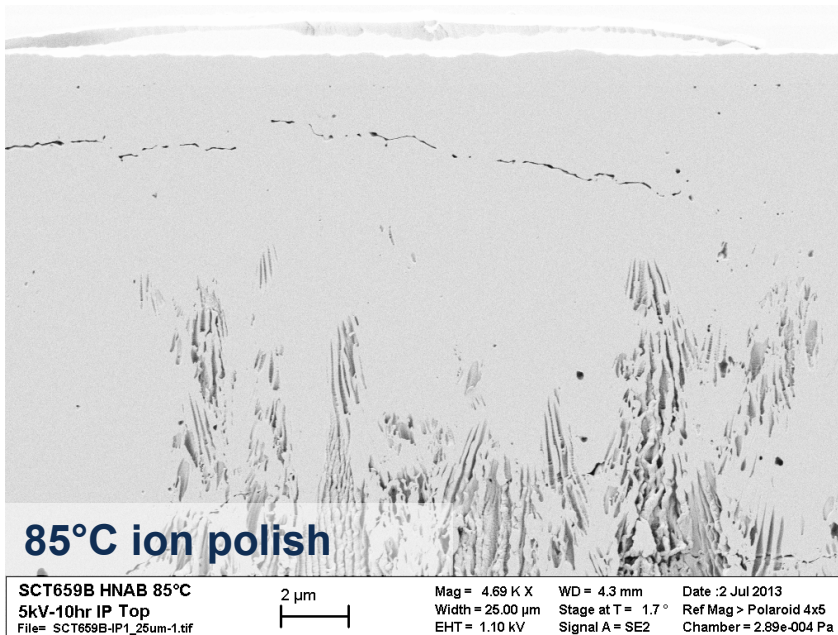
# No detonation observed in amorphous HNAB or 85° C crystallized HNAB

- In films up to  $\sim 200\text{ }\mu\text{m}$  thick
- No porosity in amorphous films



# No detonation observed in amorphous HNAB or 85° C crystallized HNAB

- In films up to  $\sim 200 \mu\text{m}$  thick
- Crystallization at 85° C is above glass transition temperature





# Conclusions

- Detonation in HNAB films depends on crystallization conditions
  - Similar detonation velocities for films crystallized at room temperature and 65° C
  - Different critical thickness for films crystallized at room temperature and 65° C
  - Amorphous films lack porosity and do not detonate up to ~ 200  $\mu\text{m}$  thick
  - Films crystallized at 85° C (above  $T_g$ ) do not detonate up to ~ 200  $\mu\text{m}$  thick
- Acknowledgements
  - M. Barry Ritchey, Ryan R. Wixom
  - Jill C. Miller, Bob Patton
  - The Joint Department of Defense/Department of Energy Munitions Technology Development Program