Thermal and impact reaction initiation of mechanically activated Ni/Al reactive systems

B. Aaron Mason, Lori Groven, Steven Son, Alejandro Strachan (PI),
Purdue University, Mechanical Engineering Department, West Lafayatte, IN 47930
Khachatur Manukyan, Alexander Mukasyan
Notre Dame, South Bend, IN, 46556

Research Objectives

The primary goal of this effort is to support the development of a physics-based multi-resolution model in order to gain greater understanding of multi-functional reactive composites through material fabrication and experimental verification.

Approach:

- Reactive intermetallic composites are produced through high-energy ball milling.
- The composite's microstructure is characterized through high resolution electron microscopy.
- Information from atomistic simulations and electron microscopy is used to inform continuum level simulations of high-rate mechanical events.
- Continuum simulations are compared to high-rate mechanical testing performed at Purdue University's Zucrow labs.

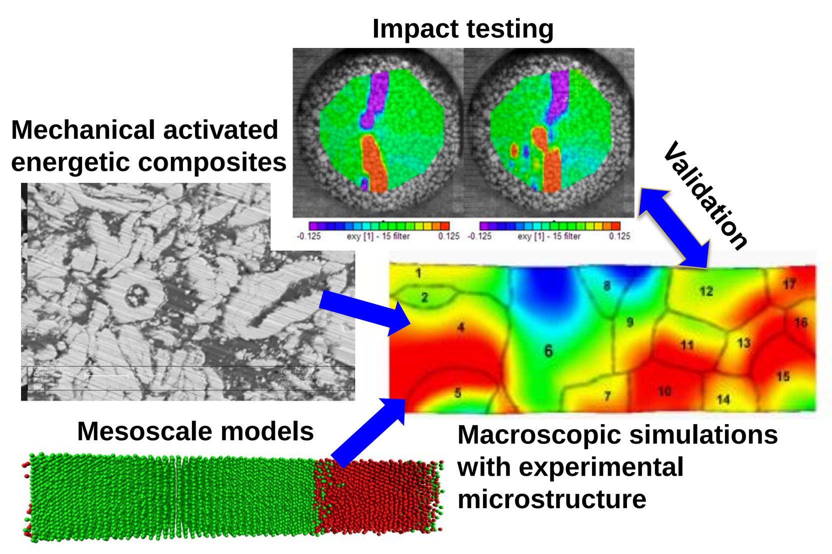


Fig. 1. Schematic of coupled experimental-modeling effort.

For this work intermetallic reactive composites such as Ni/Al and Ti/B are being fabricated by high-energy ball milling and are mechanically impacted to investigate the systems' reaction mechanisms to high dynamic loading.

Experimental Methods

Mechanical Activation

Powder Characteristics:
-325 mesh Al from Alfa Aesar
3-7 μm Ni from Alfa Aesar

Table 1. Milling Conditions .

Milling Parameter	Value
Crash Ratio	5:1
Milling Media	440 CSS, Ø 9.5mm
Dry Milling Time	0-17.5 min
Wet Milling Time	10 min (20 ml hexane)
Critical Milling Time	17.5 (dry)

Compact Formation
2.7g of material pressed at 30 Tons
Dimensions: 20mm x 20mm x 2 mm
Particle size used: 25 μm > d > 53 μm
Average TMD ≈ 70%

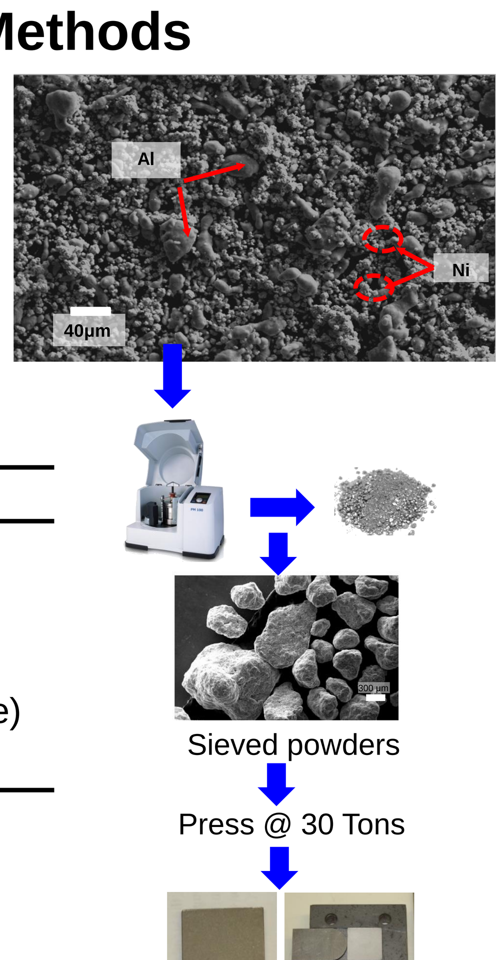
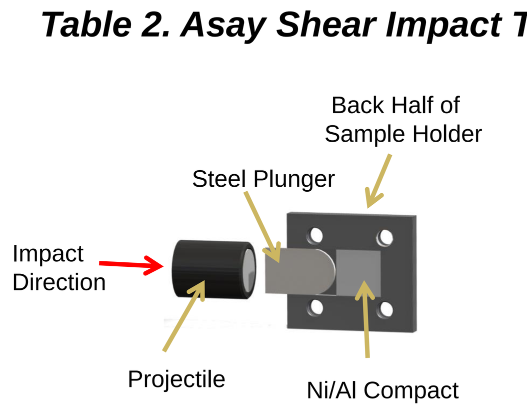


Fig. 2. Schematic of material preparation for impact experiments.

Table 2. Asay Shear Impact Testing Nominal conditions.



Impact Parameter	Value
Plunger Width	20 mm
Plunger Radius	10 mm
Plunger Weight	8.9 g
Projectile Weight	23.9 g
Projectile Impact Velocity	130 m/s
Maximum Velocity	1 km/s

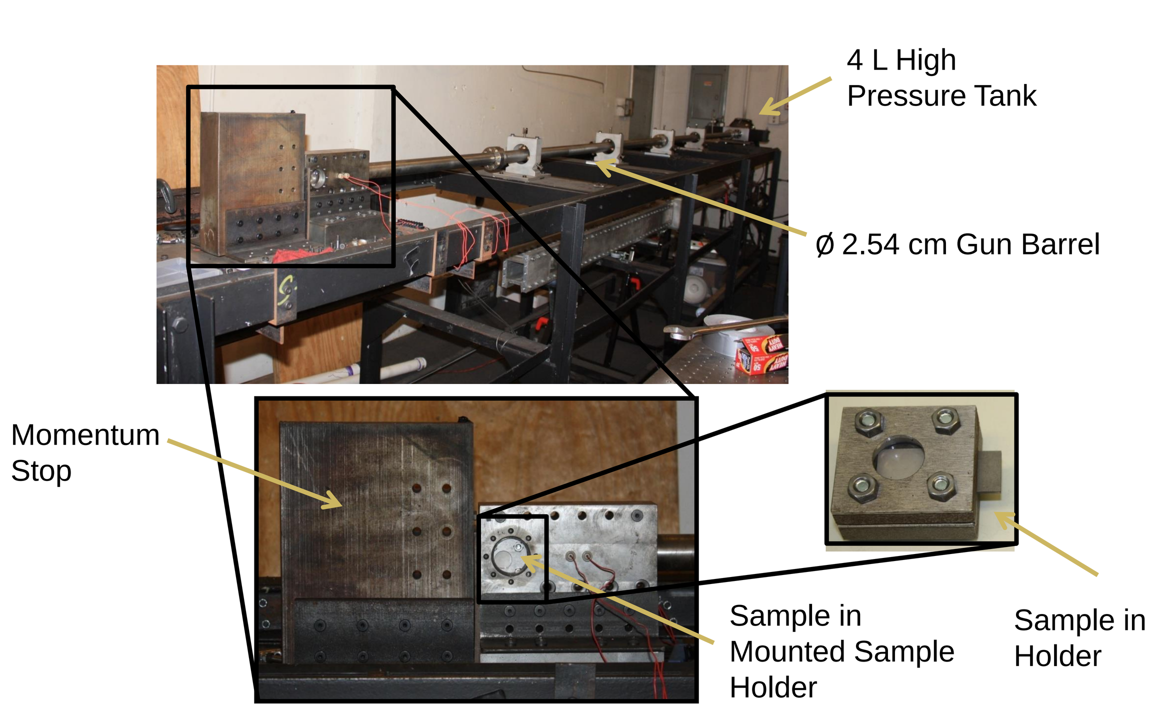


Fig. 3. Gas gun and experimental configuration for impact ignition experiments.

Results

The Ni and Al are milled first dry and then wetted with hexane. The dry milling heats the Al and Ni promoting plastic deformation and so called cold welding, thoroughly mixing the material down to the nanometer scale. However, the majority of the material is cold welded to the milling media and interior of the jar, leaving an extremely low yield of milled material, and large particles up to 3 mm in diameter. Wet milling promotes brittle fracture and refines the powder to small and more uniform dimensions while restricting the temperature of the material (Fig. 5). Reaction of the material is also limited by the creation of fracture surfaces, rather than continued deformation of the material. Additionally the longer the material is dry milled the finer the resulting microstructure after wet milling, due to increased intermixing.

Detailed Microstructure Characterization

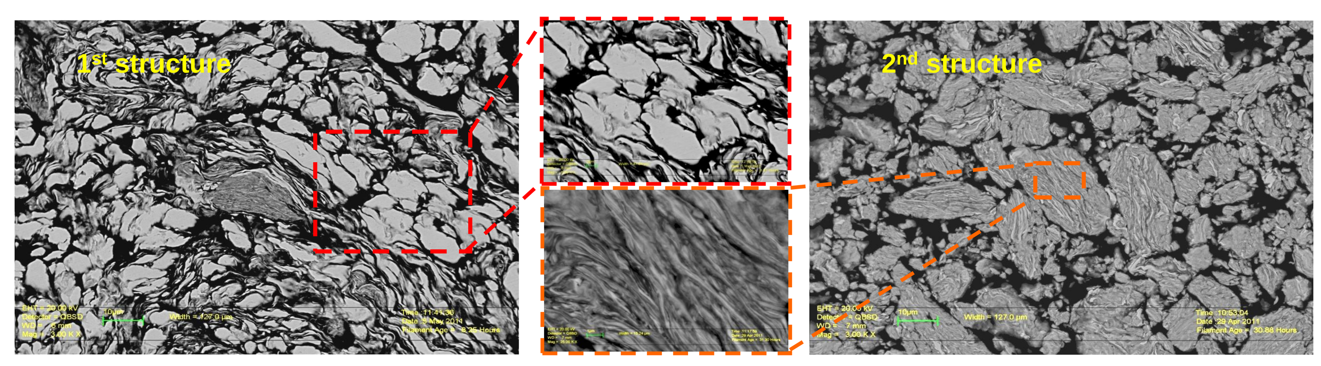


Fig.4. Microstructures observed in the ball milled Ni/Al materials.

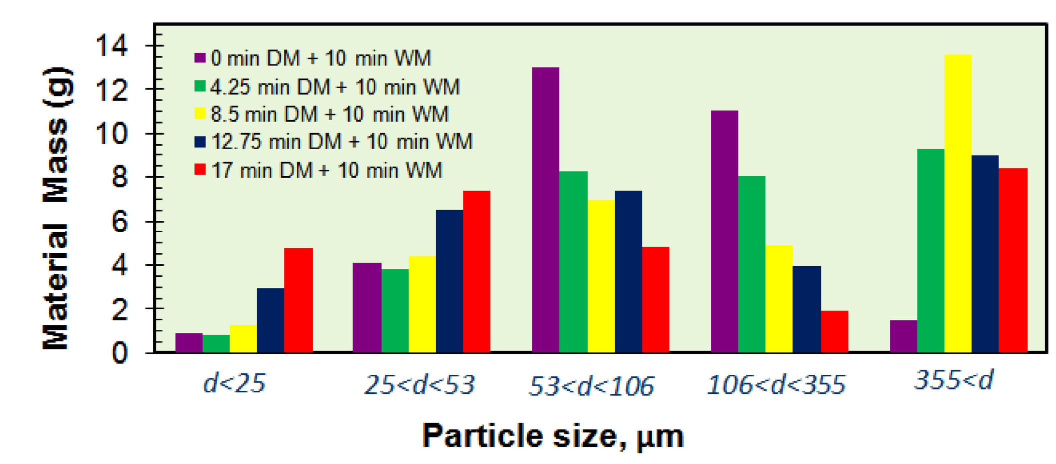


Fig. 5. Material yield of ball milled Ni/Al materials.

Two types of structures were observed in mechanically activated Ni/Al materials (Fig. 4). In the 1st structure slightly deformed Ni particles are distributed in the Al matrix. A 2nd "intermixed" laminar nanostructure was observed in the ball milled materials as well. By tuning the mechanical treatment conditions one may produce particles with only the 1st or 2nd structure or particles combining these two structures as summarized in Fig. 6.

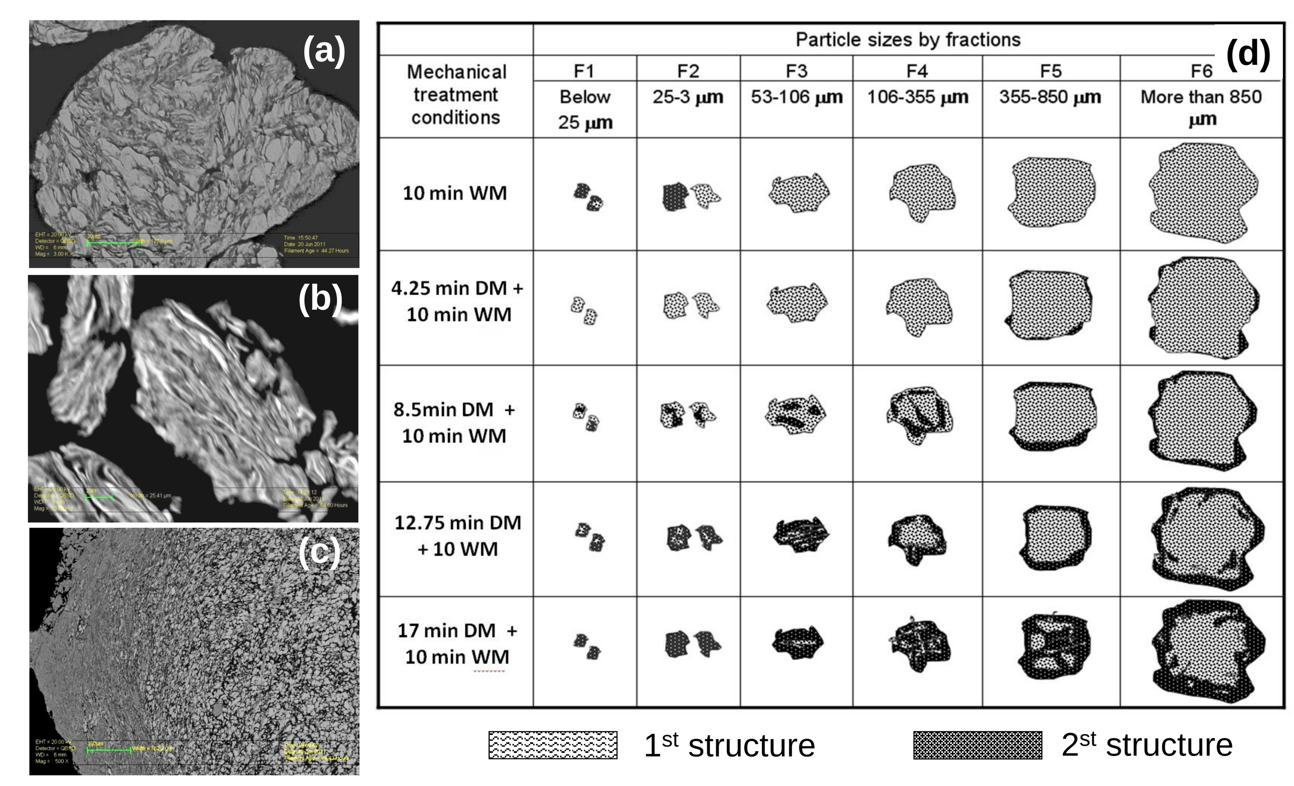


Fig. 6. Ni/Al particles with only 1st structure (a), only 2nd structure (b), combination of both structures (c), and diagram (d) showing all possible combinations.

Impact Experiments

Impact experiments were conducted using the Asay shear test. The test involves a projectile from a gas gun striking a metal plunger which impinges the sample. The geometry of the plunger can be varied to control the shear formation within the sample. The impact causes the material's temperature to rise. If the temperature is great enough a self-propagating reaction will occur. This behavior is termed "shock-assisted" chemical reaction.

Table 3. Impact Ignition Results.

Dry Milling Time (min)	% t_{cr}	TMD	Ignition@130 m/s
0	0	75	No-Go
4.25	24	72	No-Go
8.50	49	70	Go
12.75	73	73	Go
17.00	97	70	Go

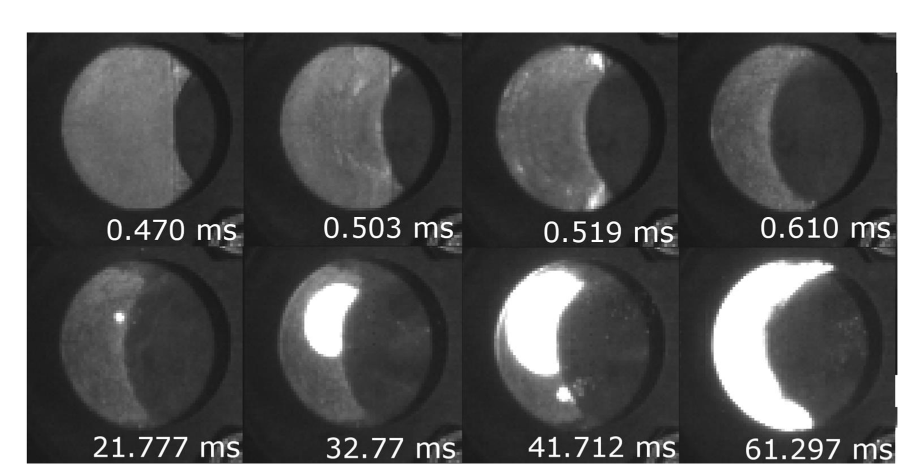


Fig 7. Impact ignition test of Ni/Al dry milled for 12.75 min and wet milled for 10 min with a plunger of 0.4" radius at 130m/s.

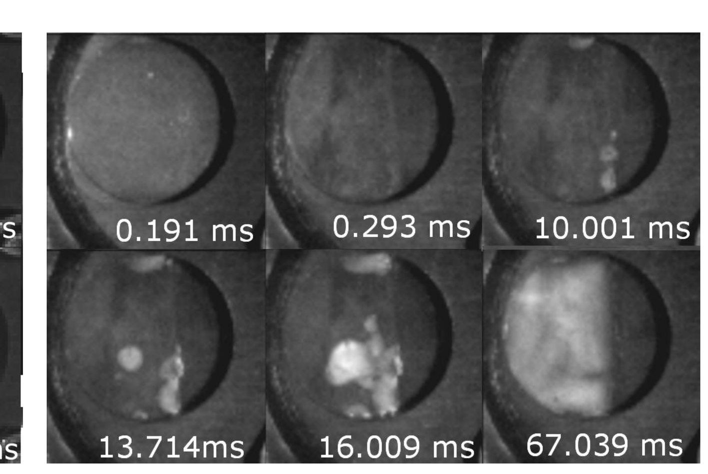


Fig. 8. Impact ignition test of Ni/Al dry milled for 17 min and wet milled for 10 min with a flat plunger at 130 m/s.

Thermal Ignition

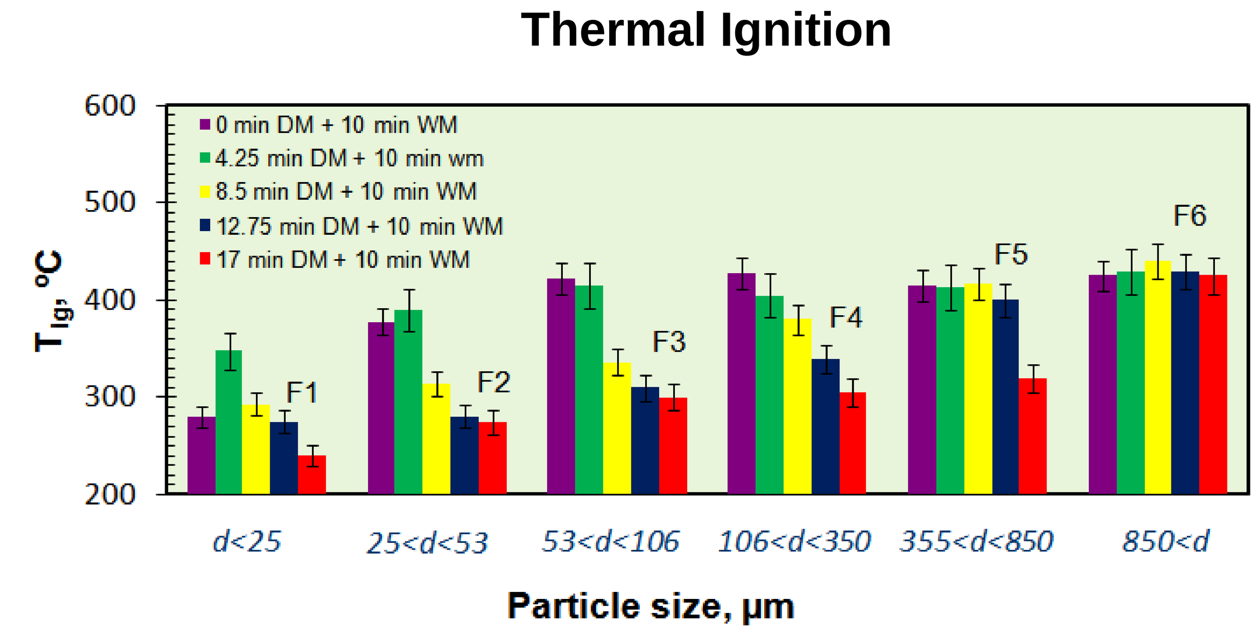


Fig. 9. Ignition temperature for Ni/Al materials obtained at various milling conditions as a function of particle size.

The thermal ignition temperature (T_{ig}) for these materials was measured using a setup which includes a hot plate, a quartz tube and a high-speed infrared thermography camera (FLIR SC6000 HS) to visualize the ignition process and to measure the temperature-time history of the reactive Ni/Al powders (Fig. 9). The T_{ig} of the mechanically activated Ni/Al materials depends on the resulting particle size and microstructure (Fig. 10). The ignition temperature of particles with the 1st structure is about 430°C. Particles having predominately the 2nd structure ignite at temperatures below 300°C.

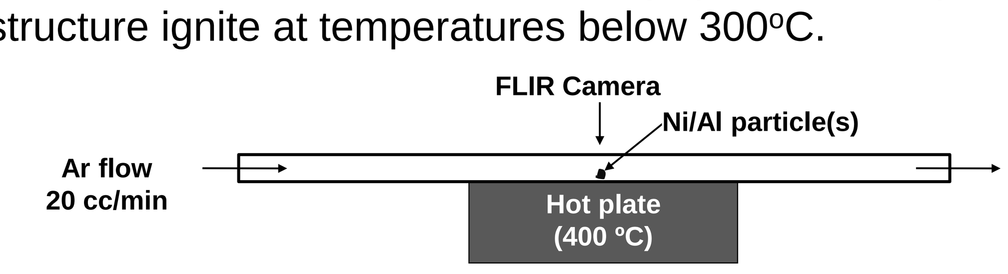


Fig. 10. Experimental setup for determining thermal ignition temperature of mechanically activated Ni/Al materials. The FLIR SC6000 HS has a spatial resolution of 1.5 microns with a frame rate of 36 kHz.

Conclusions

- The Ni/Al composite's microstructure can be tailored by adjusting the milling conditions.
- Dry milling times of ≥ 8.5 min result in highly intermixed Ni + Al laminar nanostructures.
- The development of very intermixed laminar nanostructure results in very low thermal ignition temperatures (< 300°C).
- Impact ignition was observed for plunger velocities as low as 130 m/s for materials dry milled for ≥ 8.5 min.
- The impact and thermal ignition corresponds nicely to the second microstructure observed indicating the strong dependence of microstructure/nanostructure for tuned ignition (both thermal and impact).

Acknowledgement

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Future Research Plans – Next Steps

- Further study of the structure and morphology of ball milled Ni/Al materials by Transmission Electron Microscopy (TEM), Electron Backscattered Diffraction (EBSD) and Dual Beam Field Emission Scanning Electron Microscopy techniques.
- Thermal ignition and explosion characteristics of materials produced under various MA conditions will be studied by thermal analysis (TGA/DSC) and electrothermal explosion (ETE) methods.
- Impact behavior as a function of plunger geometry.
- Microstructure/Thermal/Impact study on only wet milled materials.
- Determination of impact ignition thresholds for coupling with modeling efforts (macroscale).