THERMOELECTRIC BULK MATERIALS FROM THE EXPLOSIVE CONSOLIDATION OF NANOPOWDERS

David Nemir[1], Jan Beck[1], Lawrence Murr[2]

[1] TXL Group, Inc.

[2] University of Texas at El Paso

MOTIVATION

THERMOELECTRIC COEFFICIENT OF PERFORMANCE

$$Z = \frac{S^2 \sigma}{\kappa_e + \kappa_L}$$

INCREASE Z BY REDUCING κ_L

IN A BULK MATERIAL THIS IS CAN BE DONE BY ENGINEERING THE MATERIAL TO HAVE A NANOCRYSTALLINE STRUCTURE

THIS PROVIDES A HIGH INCIDENCE OF GRAIN BOUNDARIES AND LATTICE DEFECTS

GENERAL APPROACH: START WITH A NANOPOWDER. MAKE IT INTO A DENSE BULK SOLID WHILE PRESERVING NANOSCALE FEATURES.

BULK TO NANOPOWDER TO BULK

1. MAKE THE NANOPOWDER:
   a) BALL MILLING
   b) CRYOMILLING
   c) CHEMICAL SYNTHESIS
   d) GAS ATOMIZATION
   e) MELT SPINNING

2. GREEN COMPACT (WITH OR WITHOUT A BINDER)

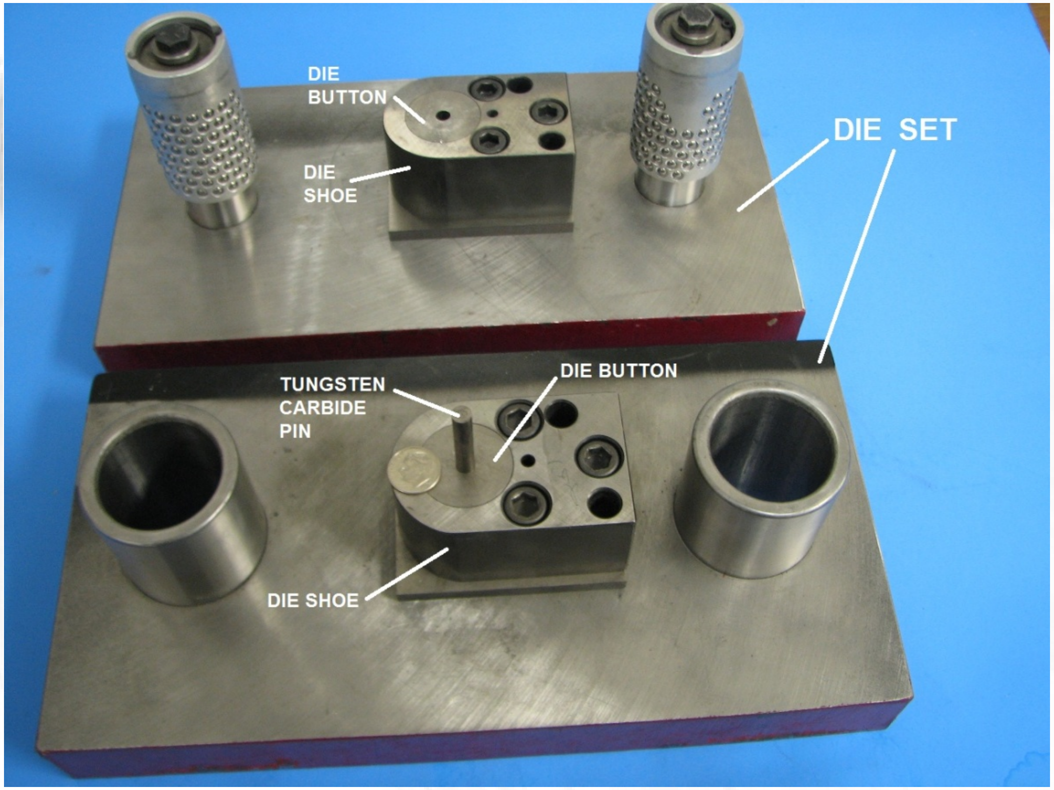
3. POST PROCESSING TO GET GOOD INTERPARTICLE BONDING
   a) HOT PRESSING
   b) ANNEALING
   c) SPARK PLASMA SINTERING

PROBLEM: STEP 3 INVOLVES TIME & HEAT & CAN LEAD TO GRAIN GROWTH

OUR STUDY -- CONSOLIDATE WITHOUT GRAIN GROWTH

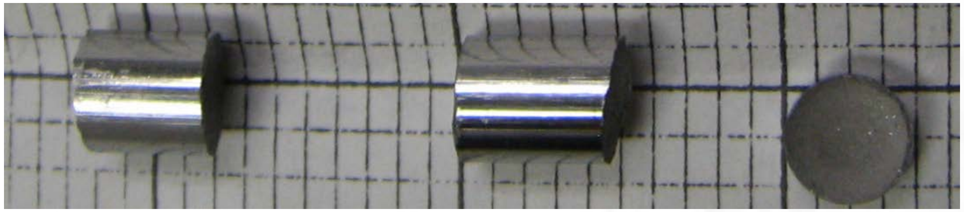
d) HIGH PRESSURE COLD PRESSING
e) SHOCKWAVE CONSOLIDATION

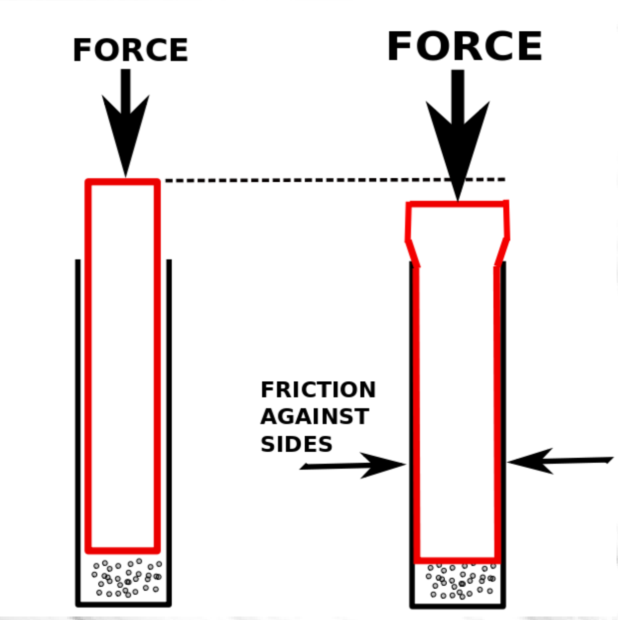
AXIAL PRESSING A PELLET



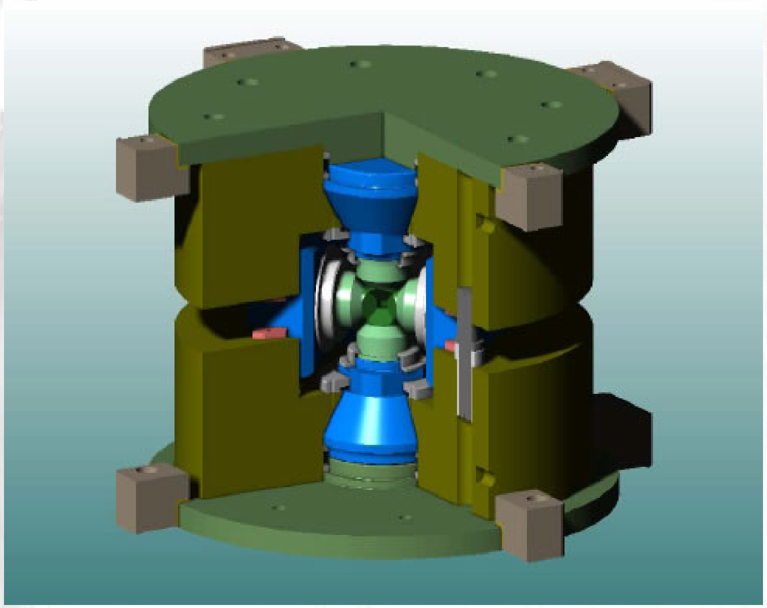
BY PLACING THE DIE SET IN A 70,000 lb PRESS, PRESSURE ON A 0.25” DIAMETER PIN OF UP TO 9.8 GPA. LOAD LIMIT ON PIN OF 3.8 GPA. DIMINISHING RETURNS AT 1.5 GPA.

COMPRESSIVE LOAD CAUSES PIN TO EXPAND (SWELL IN HOLE) REDUCING PRESSURE ON POWDER.

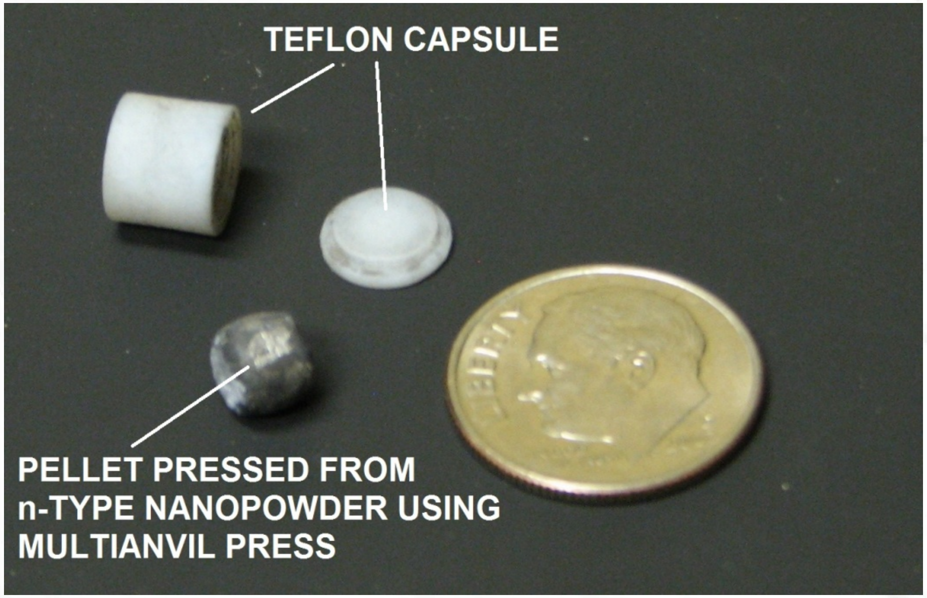




ANOTHER APPROACH TO STATIC PRESSING



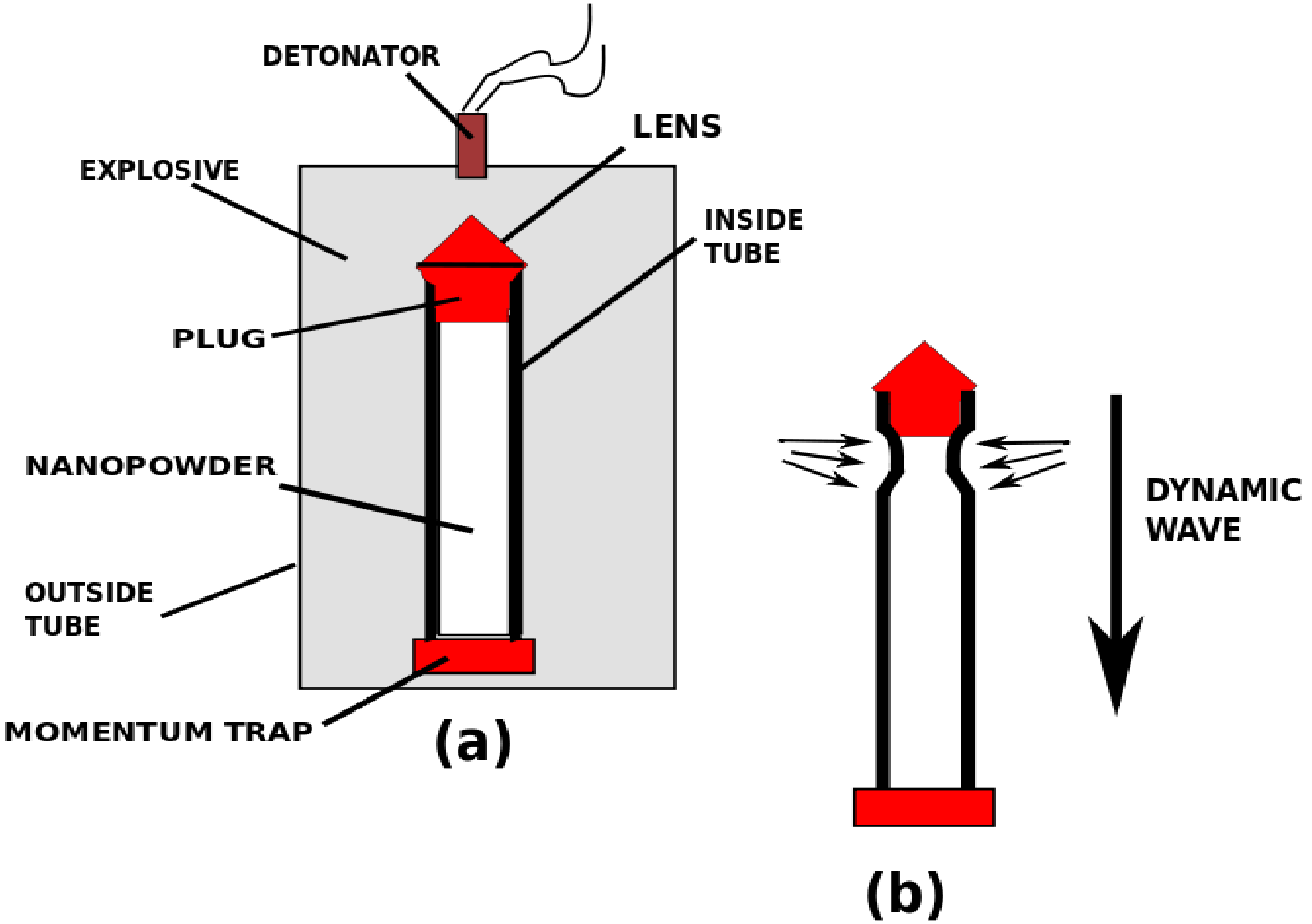
MULTI-ANVIL PRESS TO APPLY 5 GPA PRESSURE TO NANOPOWDER SAMPLES TO MAKE A PELLET (ARIZONA STATE HIGH PRESSURE LAB).



MECHANICALLY: WE OBTAINED STRONG, HOMOGENEOUS PELLETS.

TRANSPORT PROPERTIES: VARIABLE THERMOPOWER, INDICATING POOR INTERPARTICLE BONDING. MADE CONSISTENT BY ANNEAL.

USE EXPLOSIVE CONSOLIDATION TO APPLY DYNAMIC PRESSURE

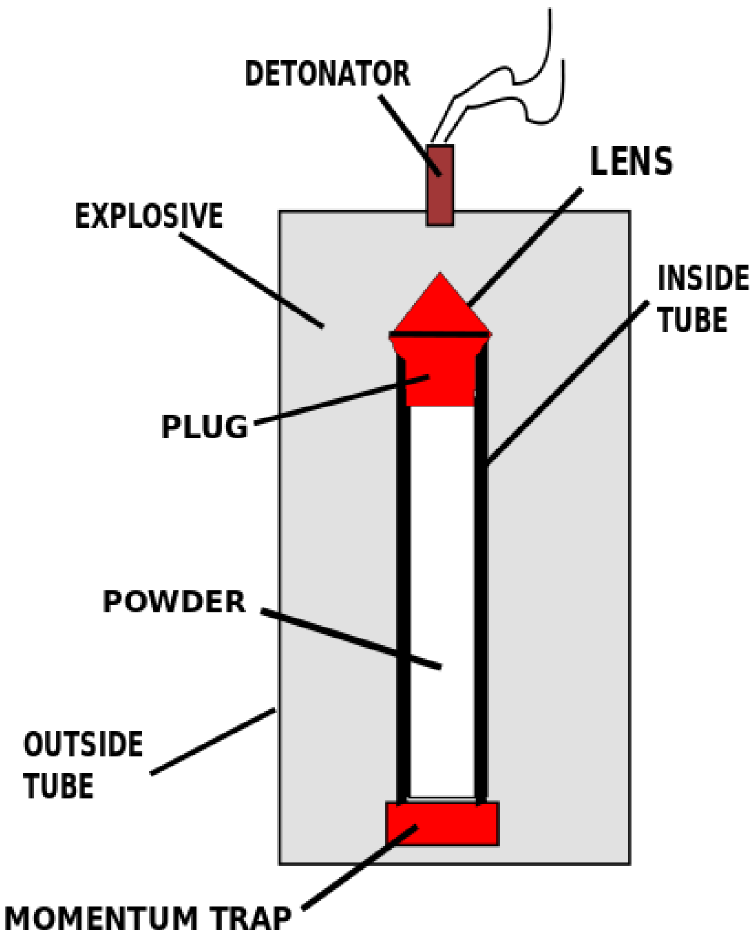


HOW IT WORKS

THE COMPACTION WAVE IS ACCOMPANIED BY PLASTIC DEFORMATION AND REARRANGEMENT WITH MOST OF THE ENERGY MANIFESTING AS HEAT ON THE SURFACES OF INDIVIDUAL PARTICLES. THIS CAUSES MELTING AT INTERFACES WHICH ARE THEN COOLED BY THE PARTICLE INTERIOR.

LOCALIZED ENERGY DEPOSITION AND MELTING OCCURS AT THE INTERFACES WHICH OCCURS AS THE PARTICLES UNDERGO SHEAR AND DEFORMATION DURING DENSIFICATION.

VARIABLES



DETONATOR/BOOSTER TYPE

LENS DESIGN

EXPLOSIVE TYPE (WE USE ANFO)

POWDER
- **CHEMISTRY**
- **PARTICLE SIZE DISTRIBUTION**
- **PRECOMPACTION**

POWDER TUBE DIMENSIONS

EXPLOSIVE TUBE DIMENSIONS

MOMENTUM TRAP DESIGN

DETONATION

DETONATION – SUPERSONIC COMBUSTION THAT PROCEEDS THROUGH A SHOCK FRONT. DETONATOR IS REQUIRED TO INITIATE.

OUR EXPLOSIVE IS AMMONIUM NITRATE/FUEL OIL (ANFO) WHICH REACTS AS:

$$C_{0.365}H_{4.713}N_{2.000}O_{3.000} \rightarrow 0.322CO_2 + 2.356H_2O + N_2 + 0.043C$$

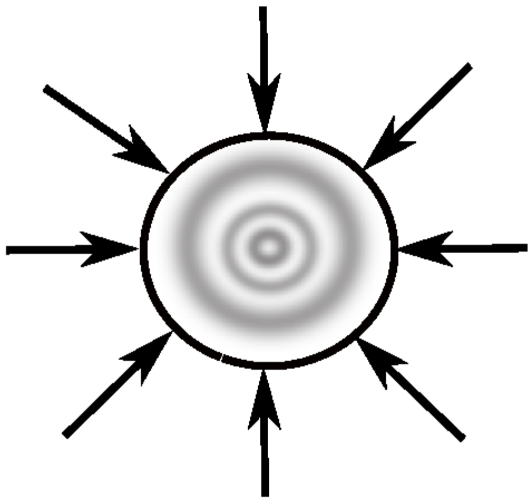
A SOLID QUICKLY BECOMES GAS, GAS ON THE RIGHT TRIES TO FIT IN THE SAME VOLUME AS THE SOLID, THE REACTION PRECEEDS AS A WAVE RESULTING IN A PRESSURE PULSE

$$P_D = \frac{1}{4}\rho_o V_D^2$$

where P_D is detonation pressure,
ρ_o is initial density of explosive and
V_D is the detonation velocity

DETONATION VELOCITY OF ANFO = 3200 m/sec
DENSITY OF ANFO = 840 kg/m^3
SO DETONATION PRESSURE OF ANFO = 2.3 GPa

PRESSURE WAVE CONVERGES INTO CENTER OF TUBE



TWO COMPETING PHENOMENA. PRESSURE INCREASES. BUT ALSO, ENERGY IS USED UP IN CONSOLIDATING POWDER PARTICLES. TOO LITTLE EXPLOSIVE PRESSURE AND NO CONSOLIDATION. TOO MUCH LEADS TO MACH STEM & CRACKS.

TOO LITTLE YIELDS PACKED POWDER

TOO MUCH YIELDS MACH STEM

EXPERIMENTAL SET-UP

STEEL TUBE, 1" DIAMETER, FILLED WITH POWDER, CAPPED & LENS ON TOP

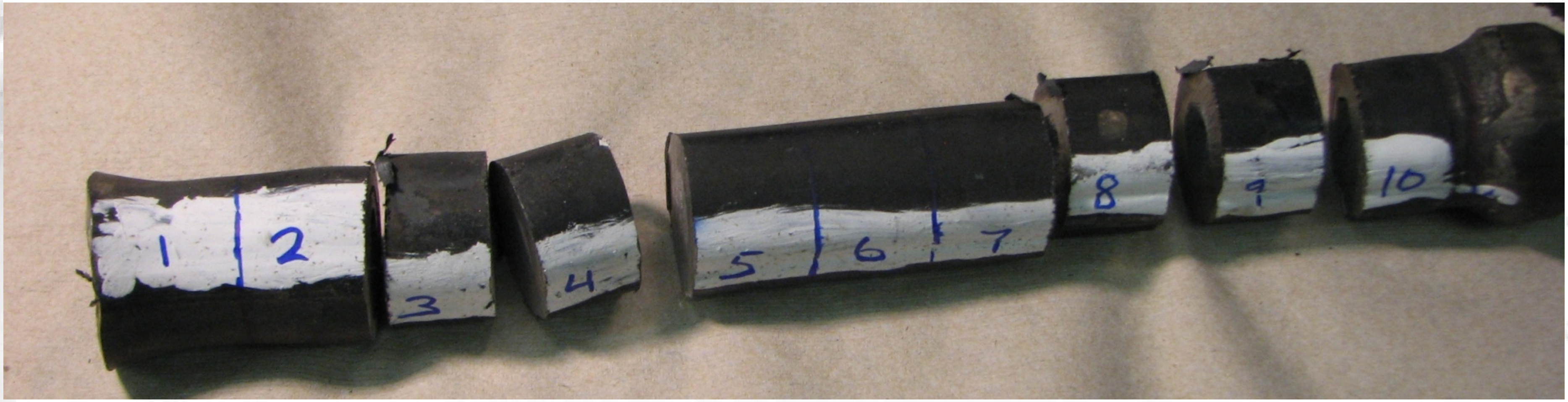
PLACED IN A PVC OUTER TUBE.

FILL OUTER TUBE WITH EXPLOSIVE. ADD DETONATOR/BOOSTER TO TOP.

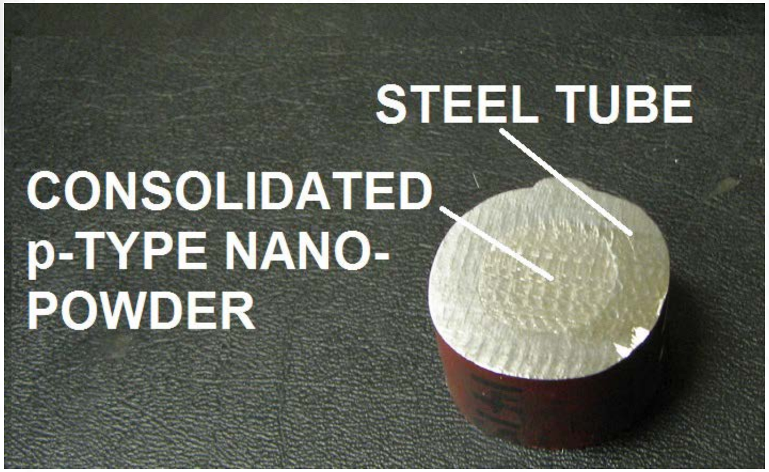
IN THE FIELD


JPL Consolidation Experiment
350 Mesh Silicon
11/04/2011
BEFORE
AFTER

MECHANICAL ANALYSIS



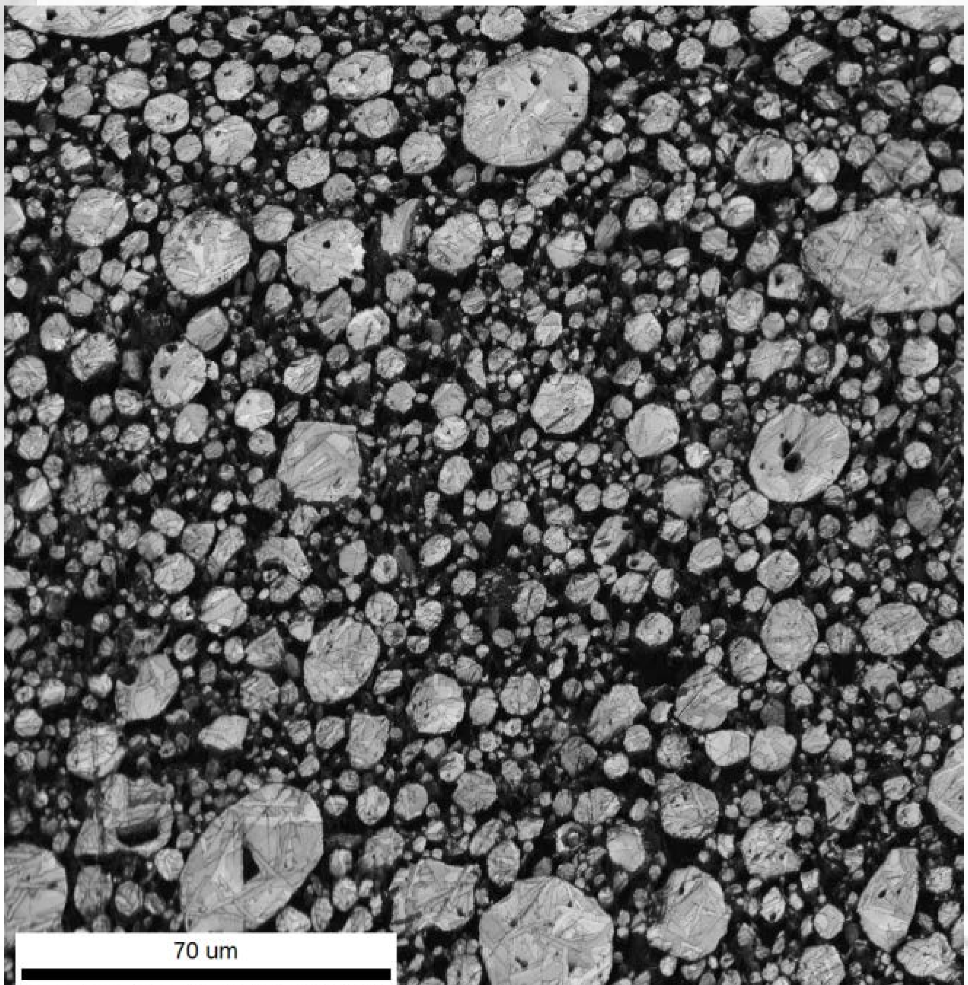
PIPE IS NUMBERED AND SECTIONED TO ANALYZE POSITIONAL DIFFERENCES



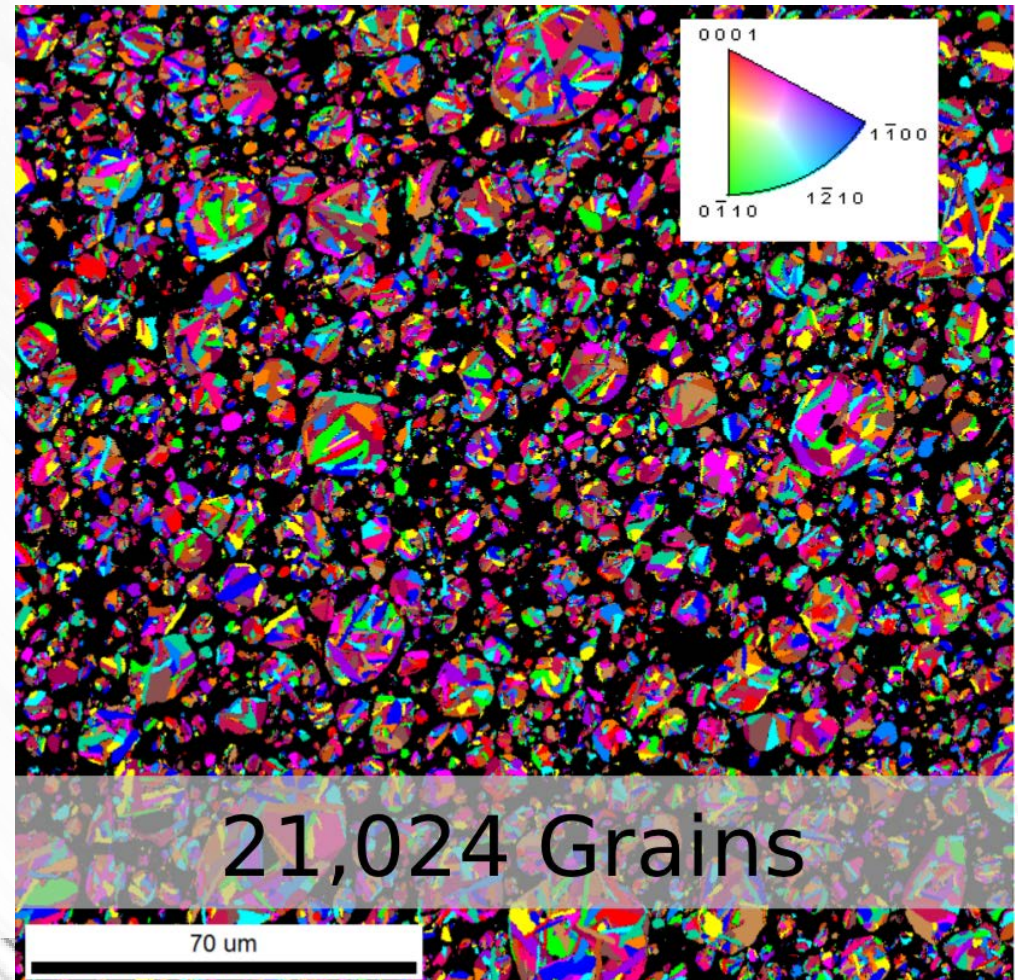
CORE SAMPLES ARE
CUT FOR TRANSPORT
PROPERTY ANALYSIS

Bi_2Te_3 NANOPOWDERS

SEM

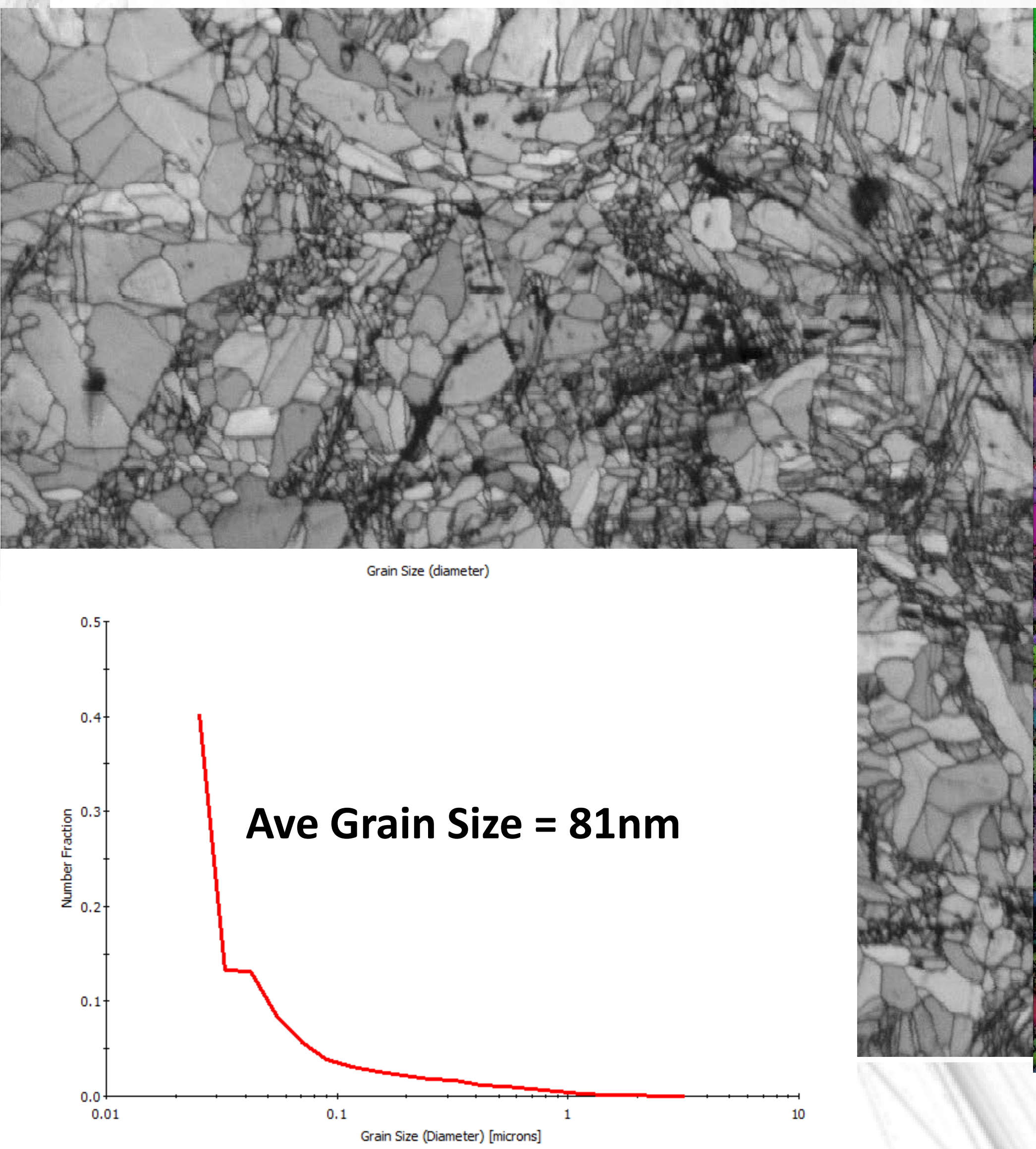


EBSD

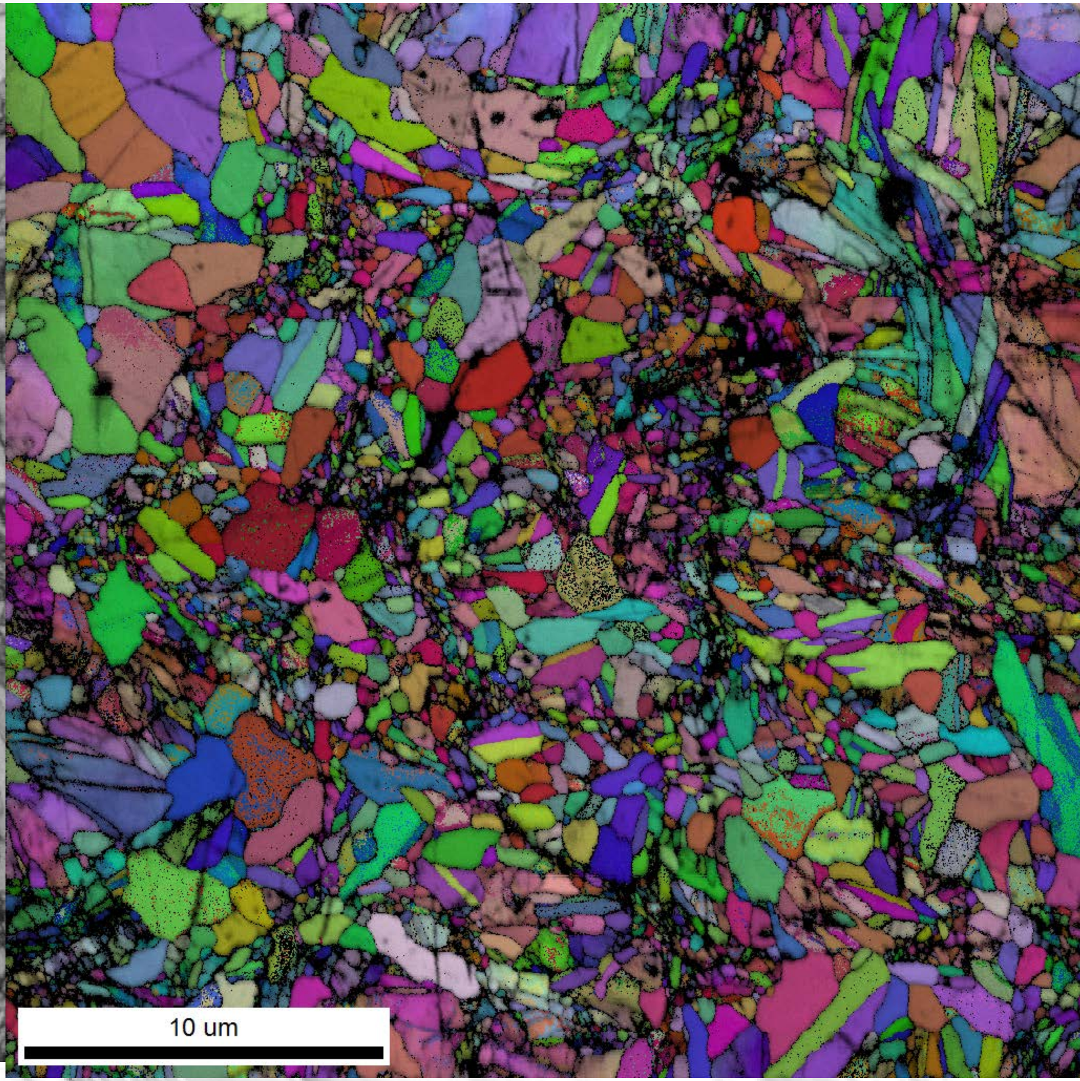


AFTER CONSOLIDATION

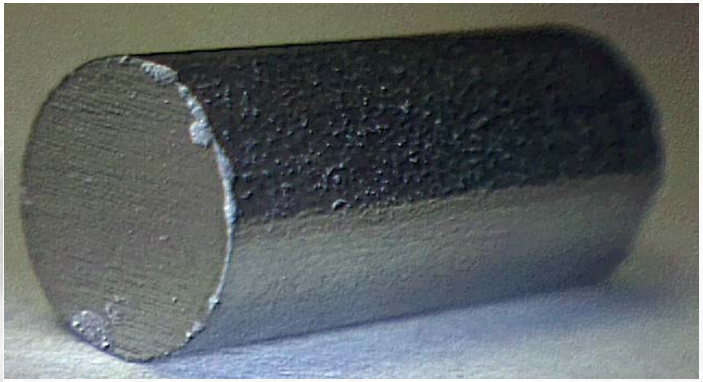
SEM



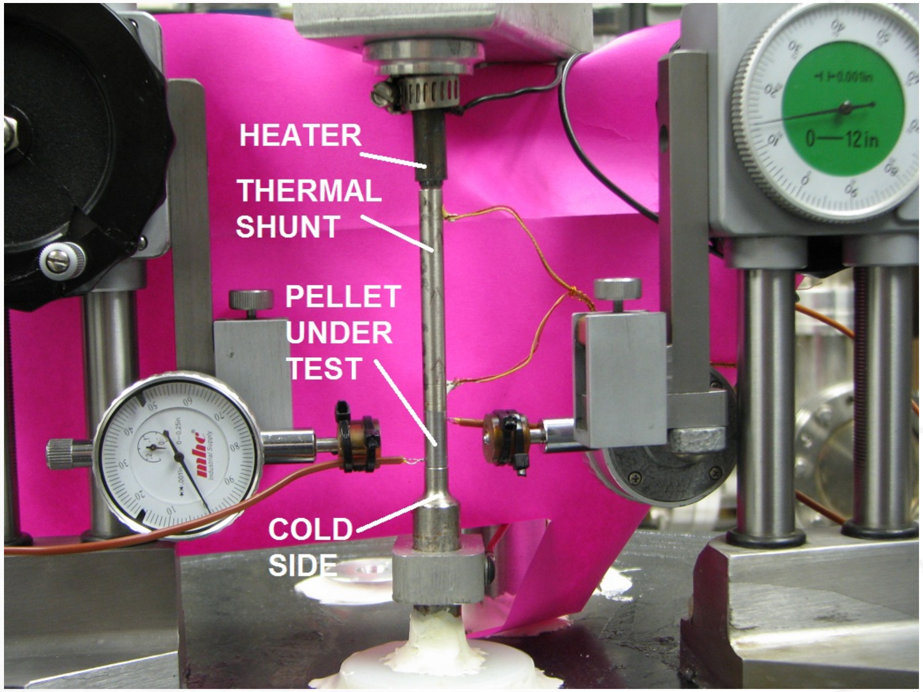
EBSD



TRANSPORT PROPERTY TESTING



Bi_2Te_3 PELLET DIAMETER 0.25” (6 mm)
LENGTH 0.6” (15 mm)
DENSITY >97% OF THEORETICAL

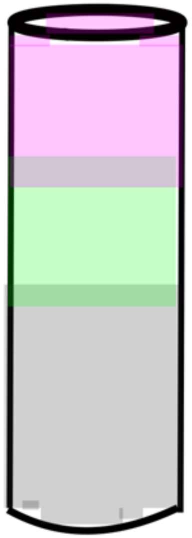


FUTURE WORK

1. CONFIRMATION OF TRANSPORT PROPERTY MEASUREMENTS
2. REDESIGN TO REMOVE THE REQUIREMENT FOR A POST ANNEAL STEP AND/OR OPTIMIZE THE ANNEAL
3. ANALYZE n-TYPE AND p-TYPE TRINARIES: $Bi_2Te_{2.7}Se_{0.3}$ & $Bi_{0.5}Sb_{1.5}Te_3$ THESE ARE AVAILABLE & HAVE BEEN CLASSIFIED INTO DIFFERENT POWDER SIZES
4. SHOCKWAVE CONSOLIDATION TO

   PRODUCE GRADED THERMOELECTRICS
5. CONSIDERATION OF OTHER MATERIALS. SILICIDES PRESENT

   SOME OF SAME CHALLENGES AS BiTe



GRADED LAYERS

PROS & CONS

ADVANTAGES

PROVEN MANUFACTURING TECHNIQUE

PRESERVES NANOSTRUCTURE WHILE PRODUCING HIGH DENSITY BULK MATERIAL

PROVIDES A MEANS TO QUICKLY PROCESS A LOT OF MATERIAL (150 μsec, 1 kg)

CONSUMMABLES ARE CHEAP (STEEL TUBES, EXPLOSIVES). LABOR & NANOPOWDER ARE THE BIGGEST COSTS.

SCALE UP IS STRAIGHTFORWARD

DISADVANTAGES

IT IS HARD TO DO SMALL SCALE EXPERIMENTS

IN THE U.S. , EXPLOSIVES WORK VERY REGULATED (BUREAU OF ALCOHOL, TOBACCO, FIREARMS)

ACKNOWLEDGEMENTS

SHOCKWAVE CONSOLIDATION

IT DOESN'T ALWAYS WORK

---- BUT IT'S STILL A LOT OF FUN

THIS WORK WAS SUPPORTED BY NASA UNDER CONTRACT #NNX10CB69C

DAVID NEMIR
TXL GROUP, INC
david@ txlgroup.com