Rupture of RDX——A method towards evaluation of the quality of Energetic Crystals

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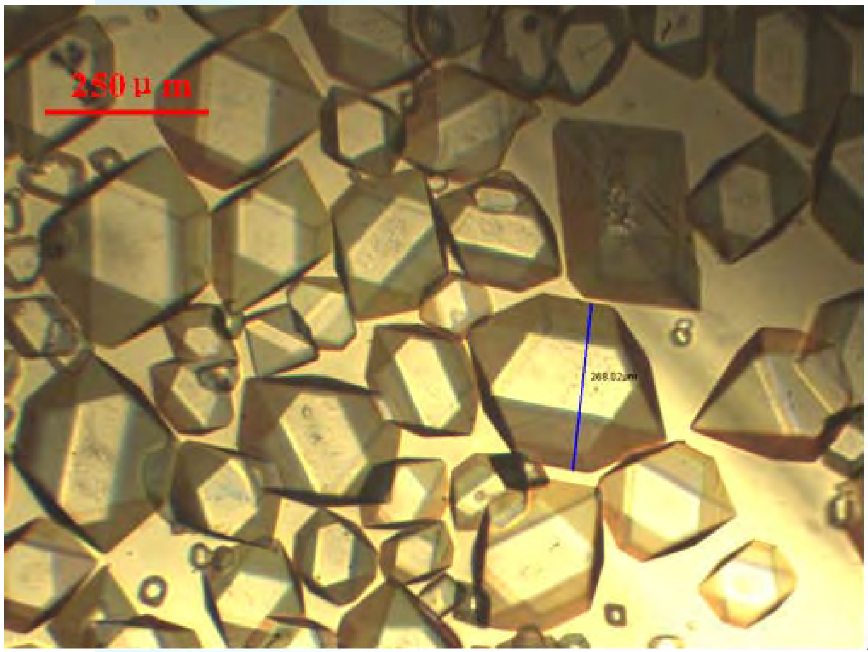
Outline

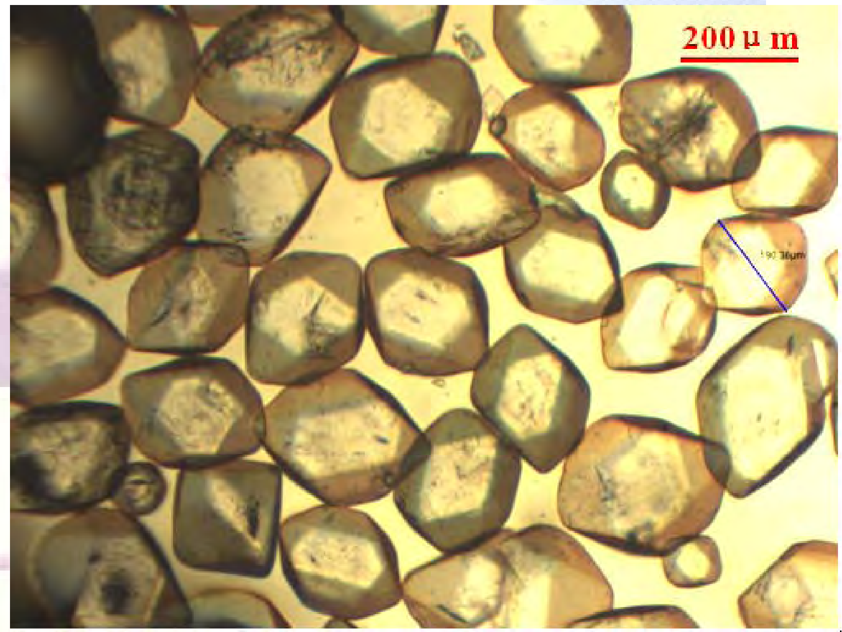
- Recent advances of RS-Energetic Crystals (ECs) at ICM
- Defects and Performance of Energetic Crystals (ECs)
- Ensembles of Energetic Crystals (EEC)
  - How to differentiate Good and Bad ESCs
  - A New Method to test Mech. Prop. of EEC
  - A Bridge between Quasi-static Mech. and Gap tests
  - Compared with other methods
- Summary

Background:

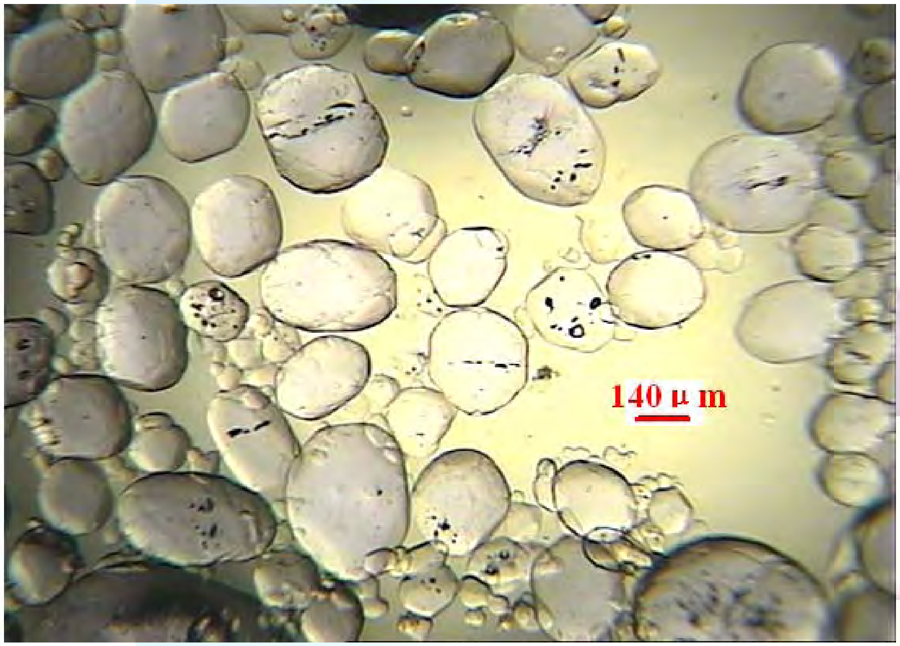
Recent advances of RS-Energetic Crystals (ECs) at ICM

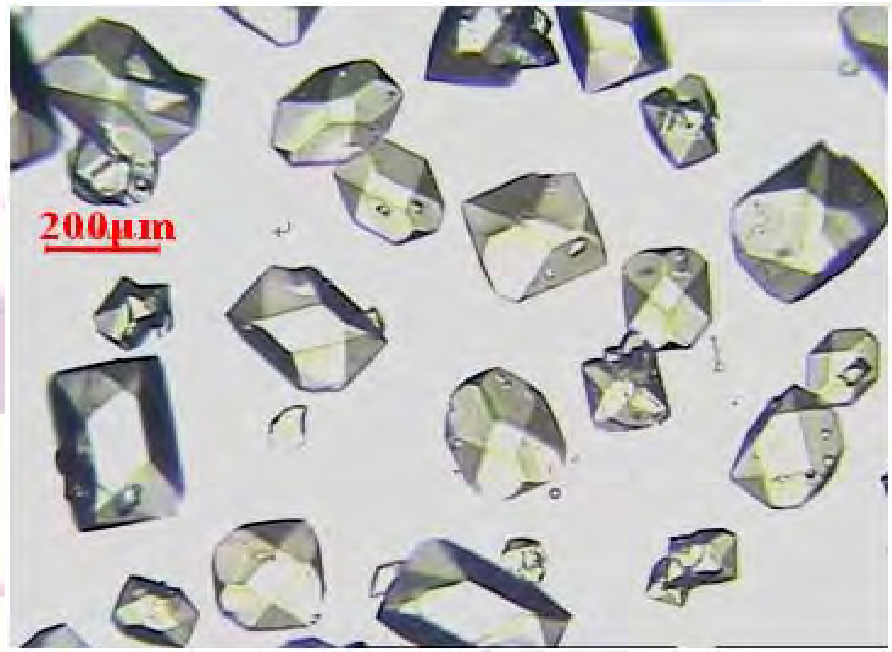


(a)A coarse D-HMX lot composed totally of perfect single crystals within which the twins have been totally eliminated.



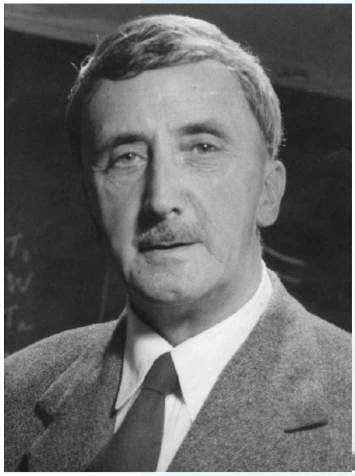
(b) A coarse D-HMX lot composed of spherioidized crystalline crystals





D-RDX crystalline particles **D-CL20X crystalline particles**

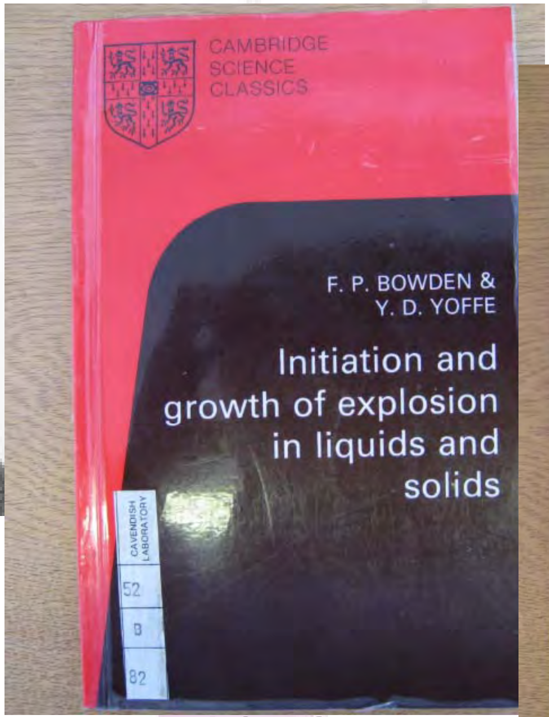
1 Defects and Performances of ECs



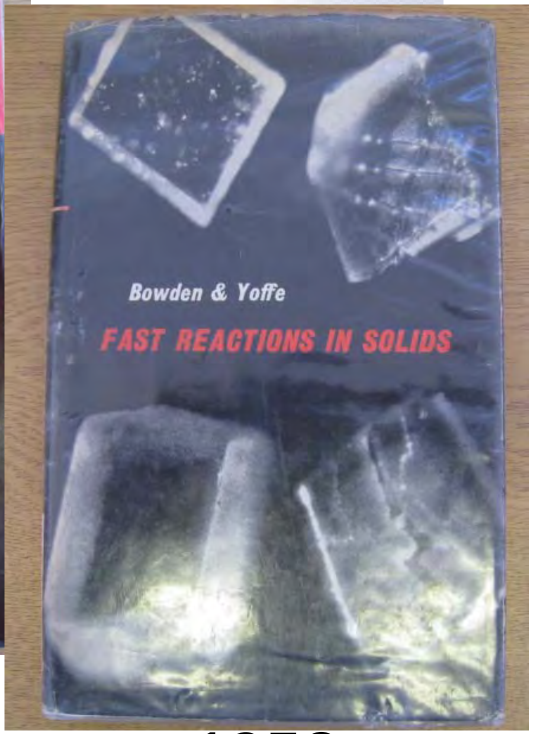
F.P. Bowden
(1903 - 1968)

A.D. Yoffe
(1919~)

From: http://www.phy.cam.ac.uk



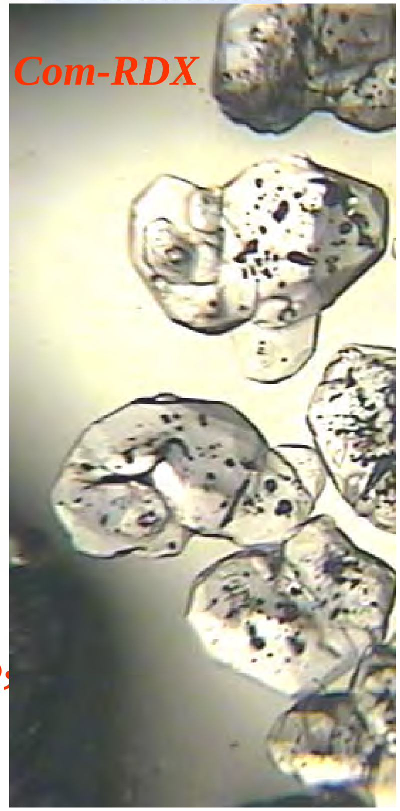
1952



1958

We know very well that "Hot spots" can dramatically sensitize the ECs.

Crystal defects: the sources of the hot spots



Points (0-D, lattice dislocations, etc.)
Lines (1-D, edge dislocations, etc)
Area (2-D,Grain boundaies, microcracks,)
Volume (3-D,gas or liquid inclusions,) **important!**

from U. Teipel, Energetic Materials,2005

That is the reason of Why **Reduced Sensitivity-ECs,** for instance, **RS-RDX, RS-HMX, RS- CL20**,

Reduce the defects, reduce sensitivity

Baseline Comparisons		
Impact Sensitivity	Sensitivity of neat material to drop impact	These techniques are not expected to show any discrimination between 'normal' and RS-RDX, but are included for the sake of a comprehensive characterization of the starting materials and to assess the robustness of these methods prior to inclusion into STANAG-4022 Edition 4.
Melting Point - DSC	Impurity level (+ phase changes)	
HPLC	Impurities (including HMX)	
GC	Impurities (mainly solvent)	
Low Angle Laser Light Scattering	Particle size distribution	
Wet Sieving		
Discriminators		
Microscopy – matched index fluid	Qualititative analysis of internal crystal defects, etc	Difference in shape of particles; visible difference in number of internal voids and dislocations
Crystal Density Distribution via Flotation	Individual crystal density distribution	Difference in distribution of particle densities.
Bulk Density via Pycnometry (Gas and Liquid)	Average crystal density	Difference in average density of particles
Optional Testing	**Property Examined**	**Possible difference**
Atomic Force Microscopy (AFM)	Analysis of surface defects, etc	RS-RDX may have fewer surface crystal defects
Nuclear Quadrupole Resonance (NQR) Spectroscopy	Crystal morphology, through analysis of line width	RS-RDX expected to show sharper NQR lines, indicating lower defect level within crystals.
Microscopic analysis of crystals	Qualitative and semi-quantitative analysis of crystal morphology	Differences in particle morphology/defect density, based on a scoring system

Many methods used to detect the defects of ECs

From:
R. M. Doherty, *L. A. Nock and D.S. Watt*, ***Reduced Sensitivity RDX Round Robin Programme-Update (R4)***, *37th ICT, June 2006.*

and:
Newsletter, *Q2,2006, NATO,* ***MSIAC***, *(from Internet WebPages)*

These are from the perspectives of **Explosive People**, how about other people?

Defects: the sources that degrade the mechanical properties of Materials——From the perspectives of ***Materials/Mechanics People:***

Griffith, A.A. (1920), **Critical stress/length**
Irwin （1948）,SIF
Wells (1963) , COD
J.R. Rice, G.P. Cherepanov (1967), J integral
……

Cracks

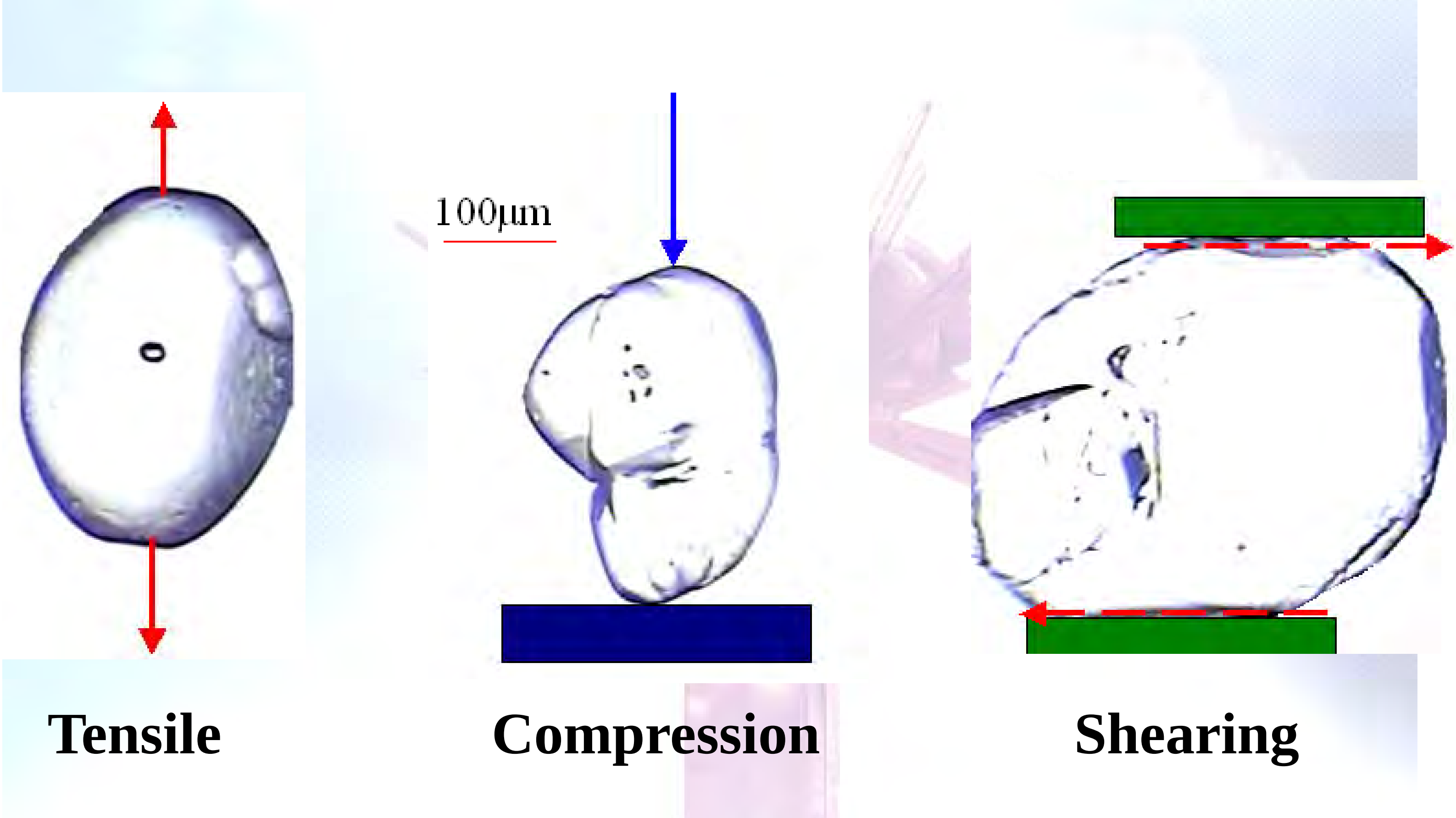
Eshelby (1956), **Eigenstrain**
……

inclusion

C.O. Leiber (2000), **Coherence Strength**
……

Grain boundary

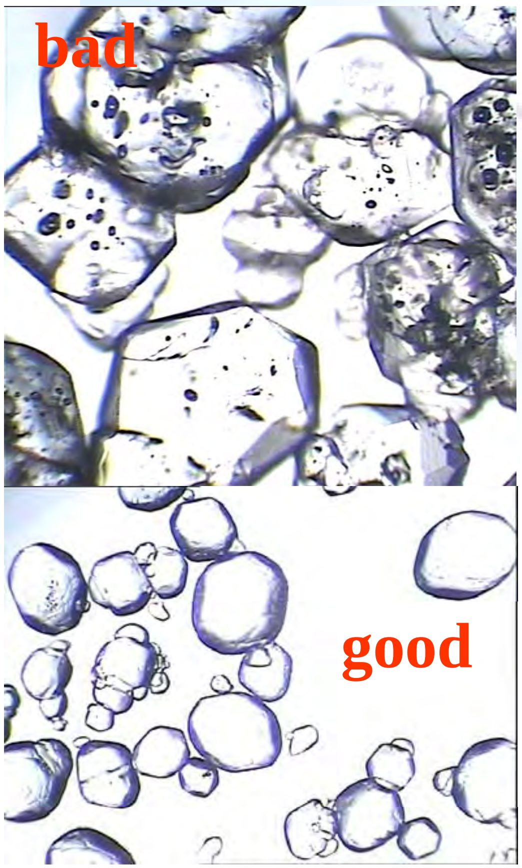
Ideal mechanical experiments for single RDX particle

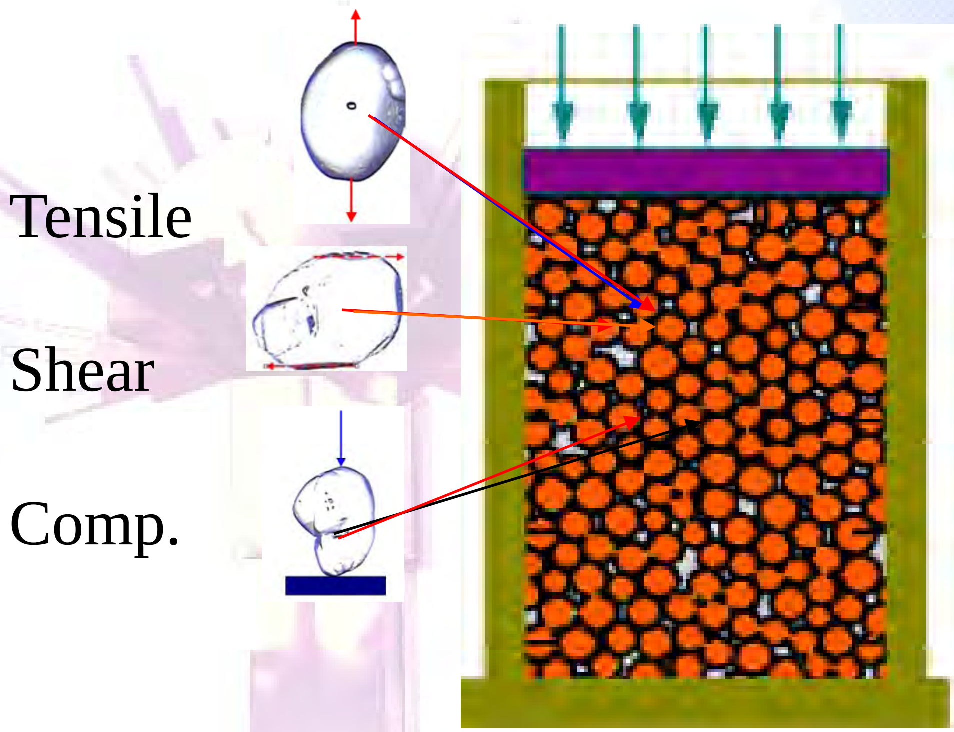


But unpractical !

(maybe for regular shape, i.e., ball particle, it works, as T. Heintz, et al, 39th ICT)

Feasible method—Check the macroscopic response of the ensembles of ECs via pressing.





M. Li, *et al*, **JSA, Vol41(8), 609-622, (2006).**
Acta Mech. Sin. (2003)

The points of the proposed method :

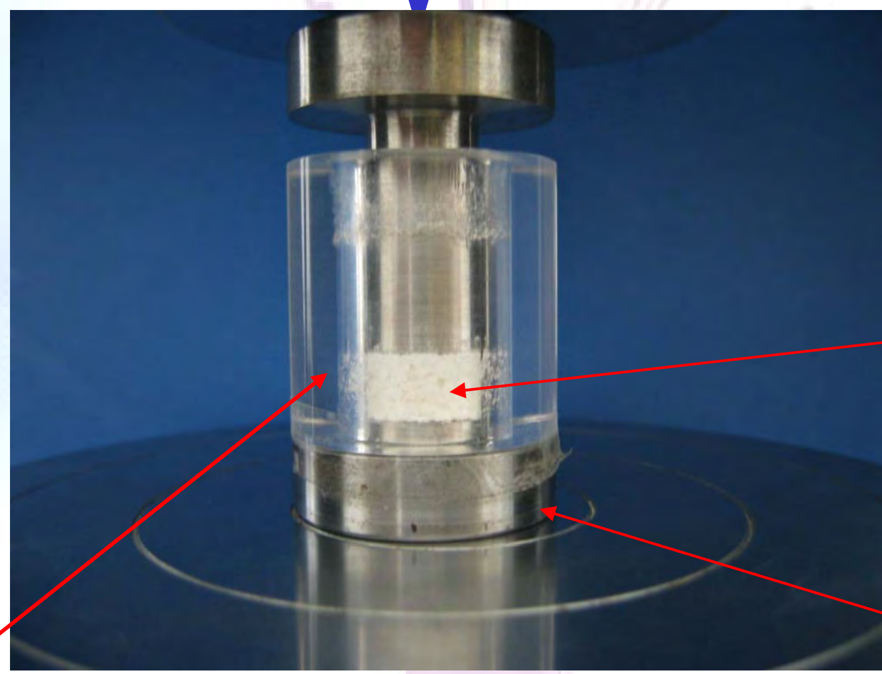
During the compression, the particles are to be crushed, the more defects insides the particles, the less coherence strength to resist to external force and this would be shown on the compression curves.

I named this method as **Compressive Stiffness Test (CST) and I don’t want to call it compressive strength test!**

Setup of CST- Uniaxial compression

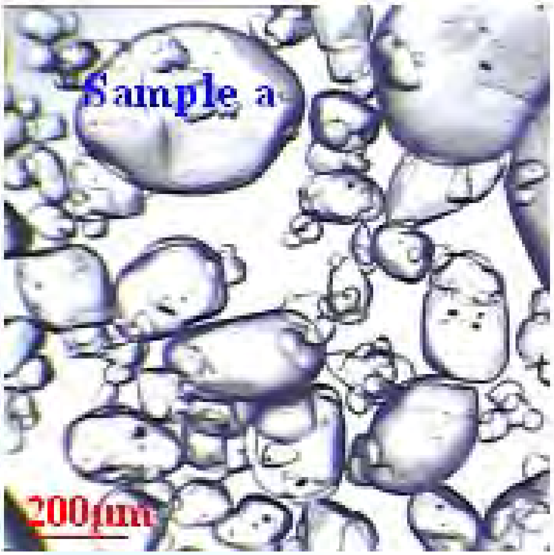
Compression speed:0.05mm/min

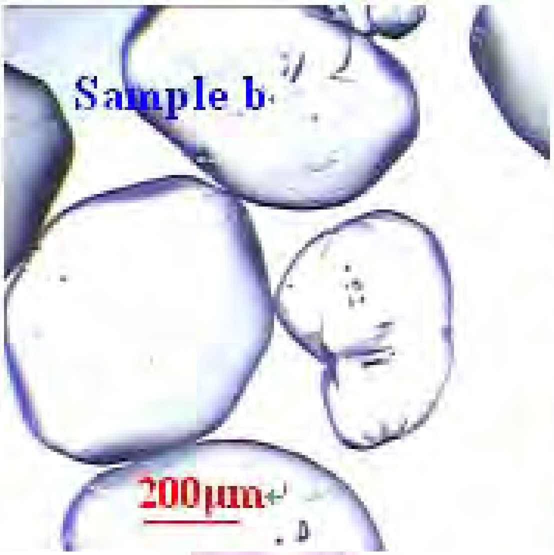
Compared with hammer speed of about 180m/min in sen. test

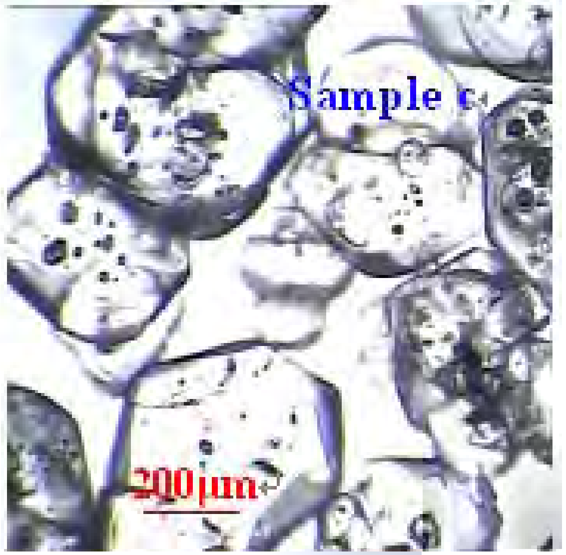


The sleeve is made of high strength stainless steel in practice using.
The internal diameter is 15mm and the thickness of the wall is 10mm.

CST-1(RDX) -Preliminary impression:







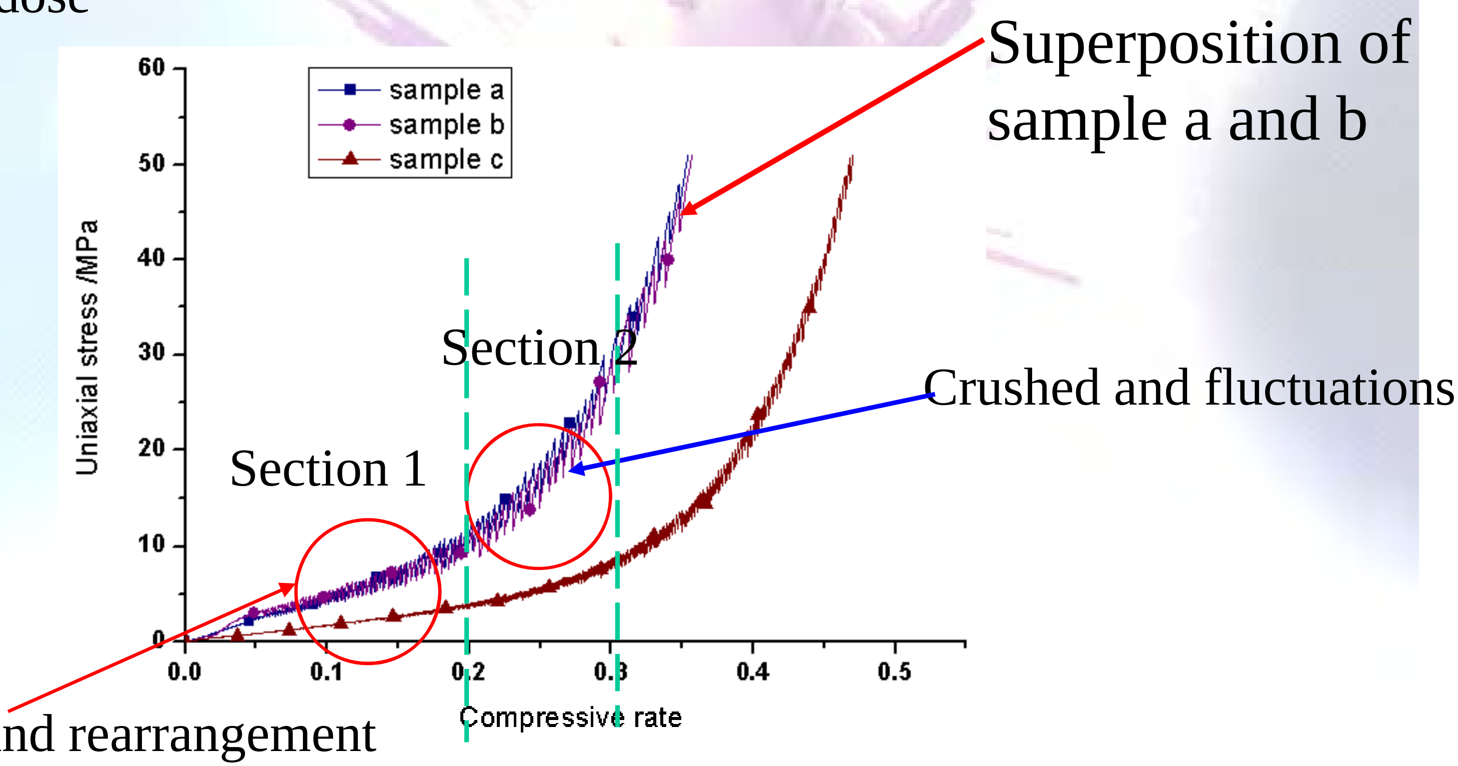
Sample a
Recrystalized

Sample b
Spheroidized

Sample c
Coarse raw particles

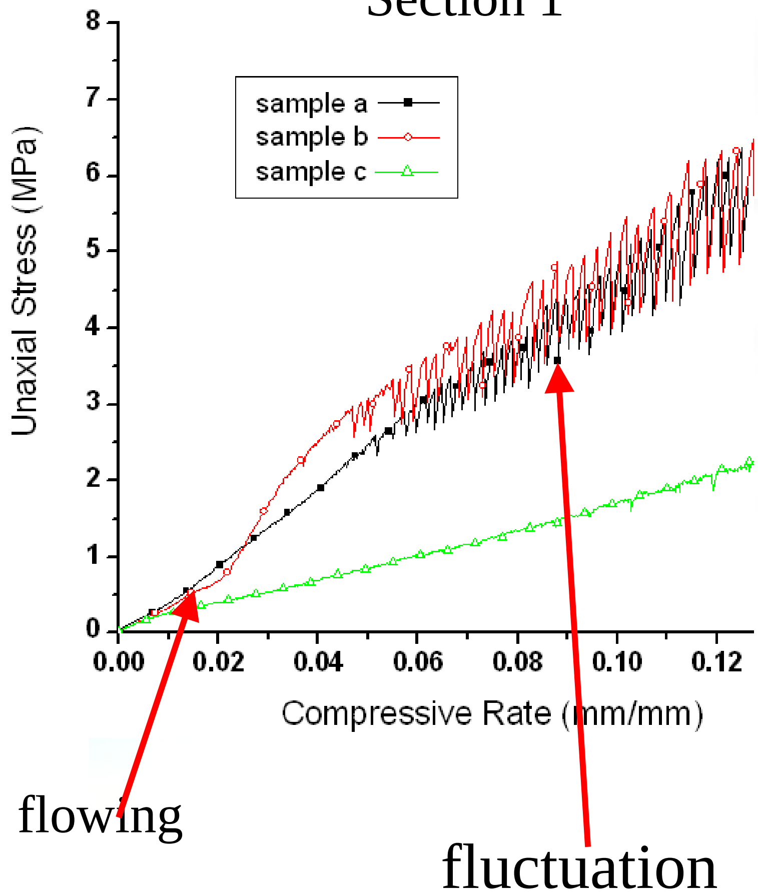
Characteristics of the Compression Curves (Test 1)

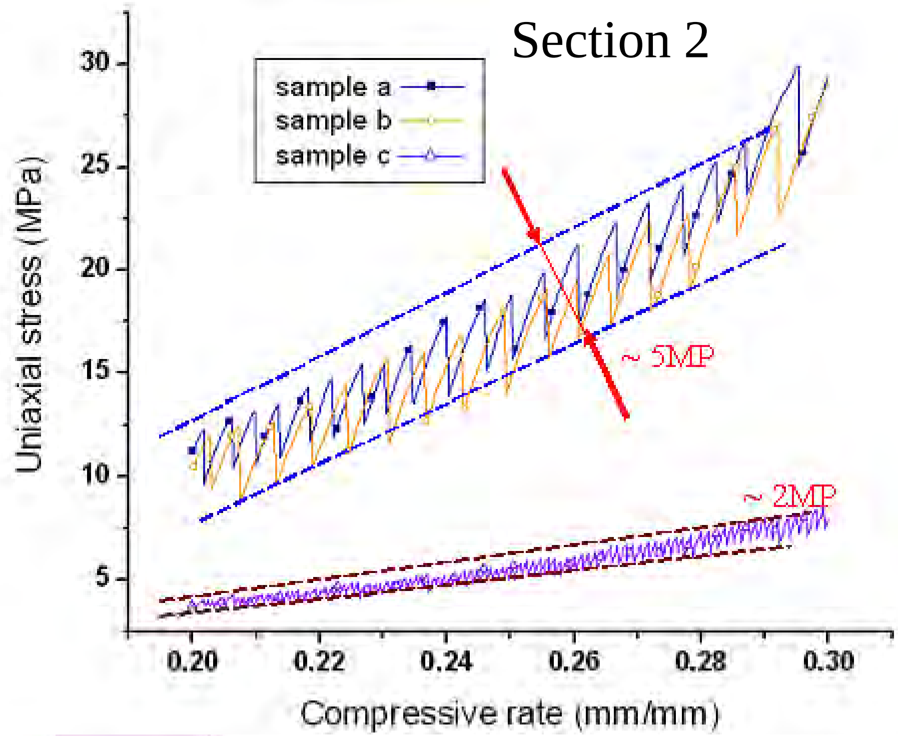
Sample a (Recrystalized)
Sample b (Spheroidized)
Sample c (Coarse raw particles)
About 2g-dose



Stages-(1) flowing (2) fragmentation

Allowable failure stress





M. Li, et al,
Chinese J. of Energetic materials,2006

CST-2 : (RDX), Gives further details

Lot	Mean size µm	D_{10} µm	D_{50} µm	D_{90} µm	source
1	603.0	172.7	635.9	882.7	commercial
2	536.6	363.9	542.3	720.1	commercial
3	335.0	102.8	332.5	570.9	recrystallized
4	382.8	140.2	375.6	631.2	recrystallized
5	276.4	93.2	283.2	540.5	Recrystallized + spheroidized
210	180	63.5	175.3	300.1	commercial

lot 1 above 40 sieve
lot 2 between 40~60

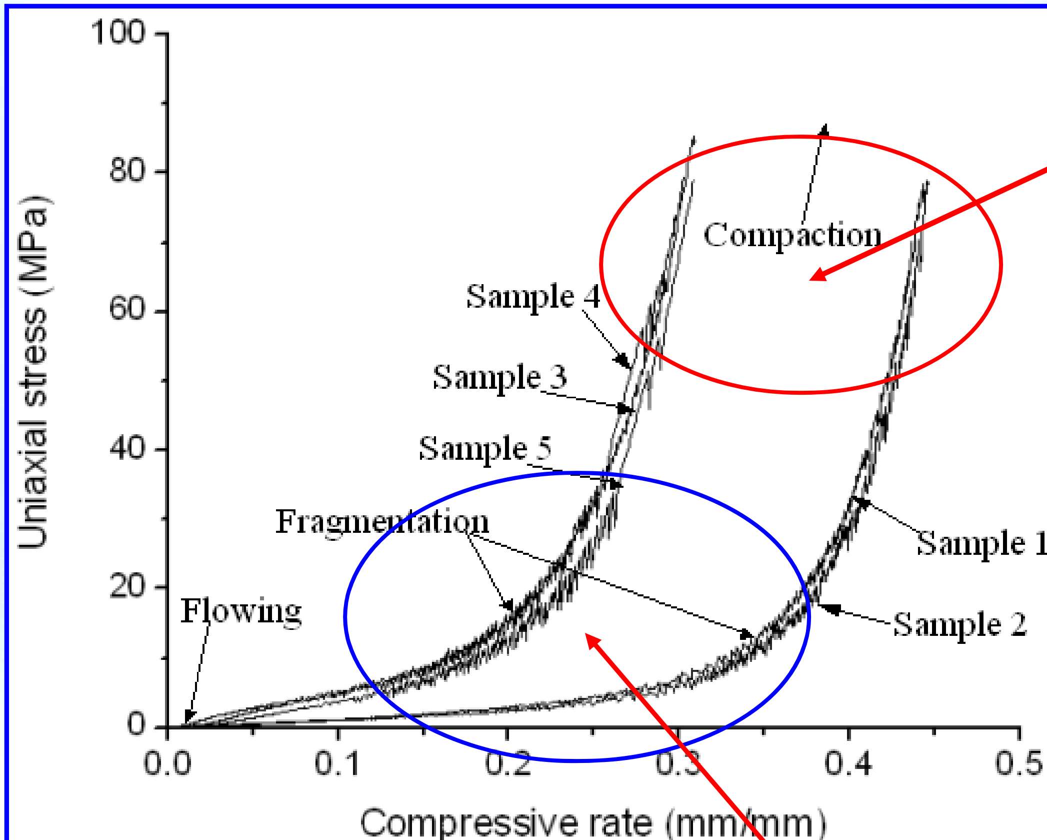
screened ← **RDX raw materials** → recrystallized Lot3,4,5

Information of five samples in one test group

Sample	Mass (g)	H_0 (mm)	Tap Density (g/cm^3)	ISM (MPa)	Apparent Density (g/cm^3)	Range of Apparent Density (g/cm^3)
sample 1/lot 1	2.001	12.184	0.929	36.5	–	–
sample 2 /lot 2	2.006	12.194	0.931	34.6	1.7925	1.7866~1.7966
sample 3 /lot 3	1.998	10.036	1.127	85.7	–	–
sample 4 /lot 4	1.999	9.873	1.147	84.8	–	–
sample 5 /lot 5	2.003	10.065	1.126	82.3	1.7992	1.7982~1.7995
sample6/lot210	2.006	11.085	0.979	45.2	1.7931	1.7887~1.7987

Results-compression curves

compaction



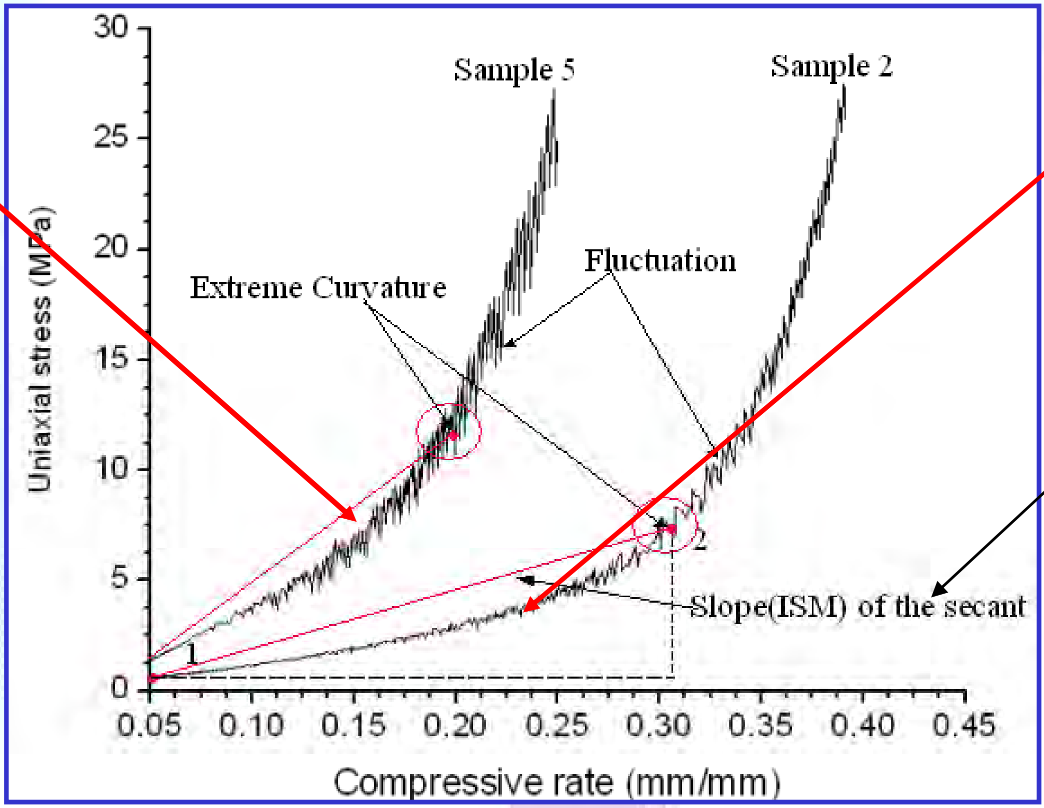
Uniaxial stress vs. compressive rate for five types RDX.

fragmentation

Quantitative method

(1)85.7MPa
(2)84.8MPa
(3)82.3MPa

Note: ISM is different from crystal elastic modulus, See, ***M. Li*** et,al PEP, (in press) and J. Zaug, 1998



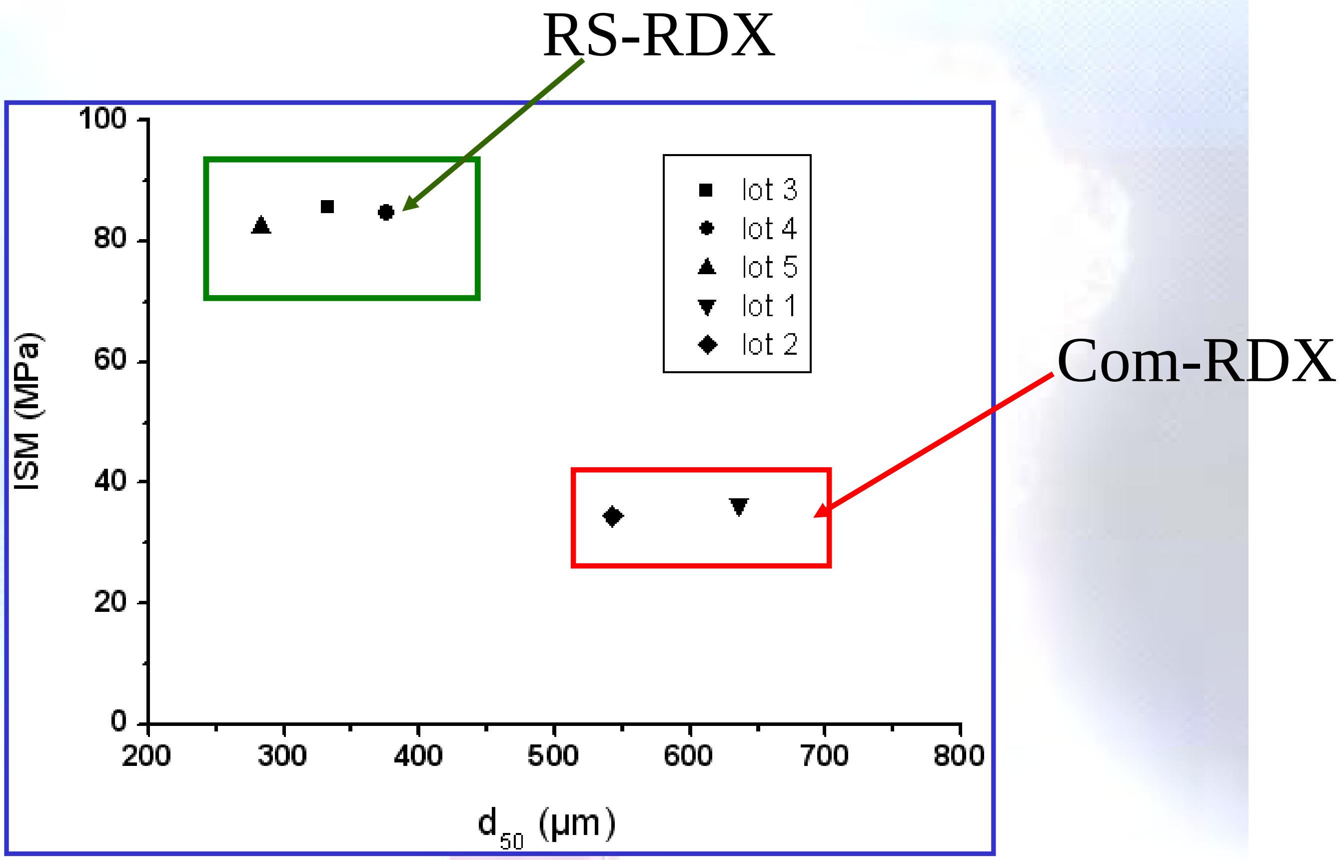
(1)36.5MPa
(2)34.6MPa

Define:
ISM
(Initial secant modulus)

Uniaxial stress vs. compressive rate in fragementation period for sample 5 and sample 2

Other than ISM, another method can be found in ***W.J. Tan, M. Li, H. Huang***, ***CJEP***,*2007*

Discussion—Effects of the particles size

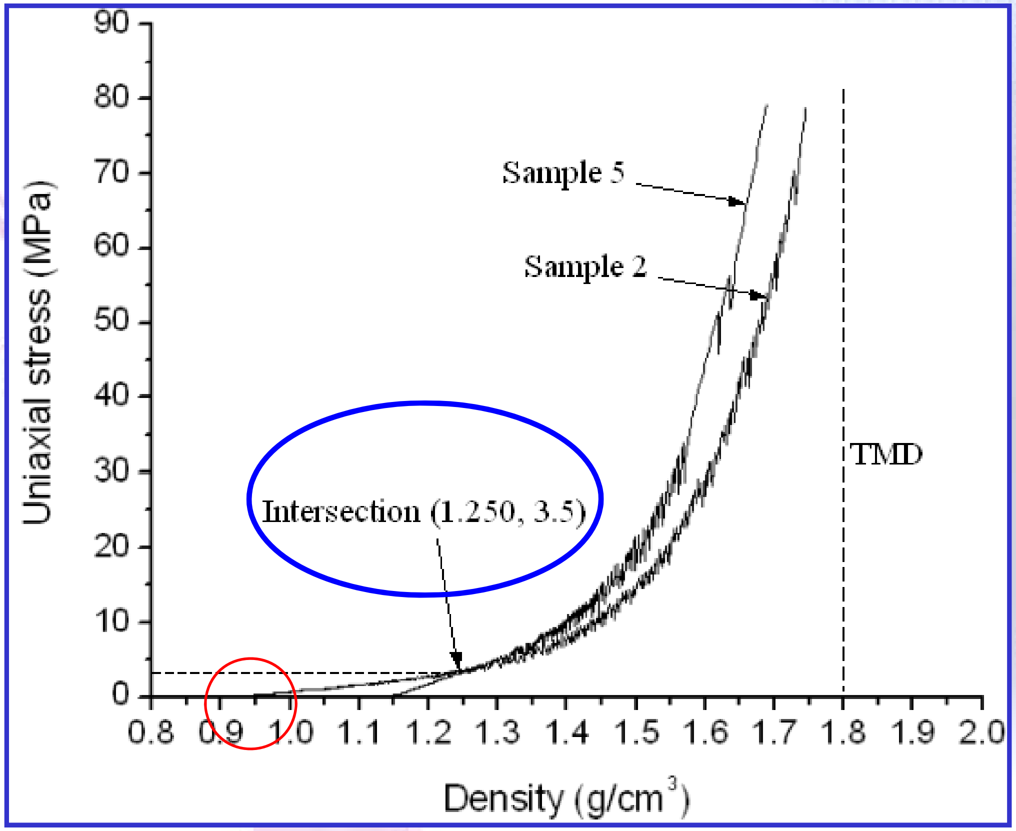


D_{50} vs. ISM

The effects of size on ISMs are very limited ! Same with L.Boren(1998,2002)

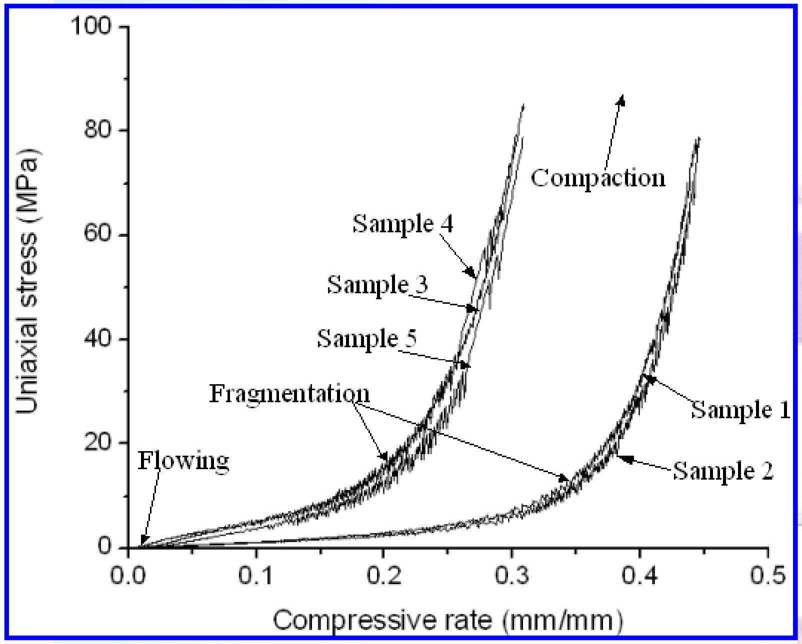
Discussion—Effects of the tap density

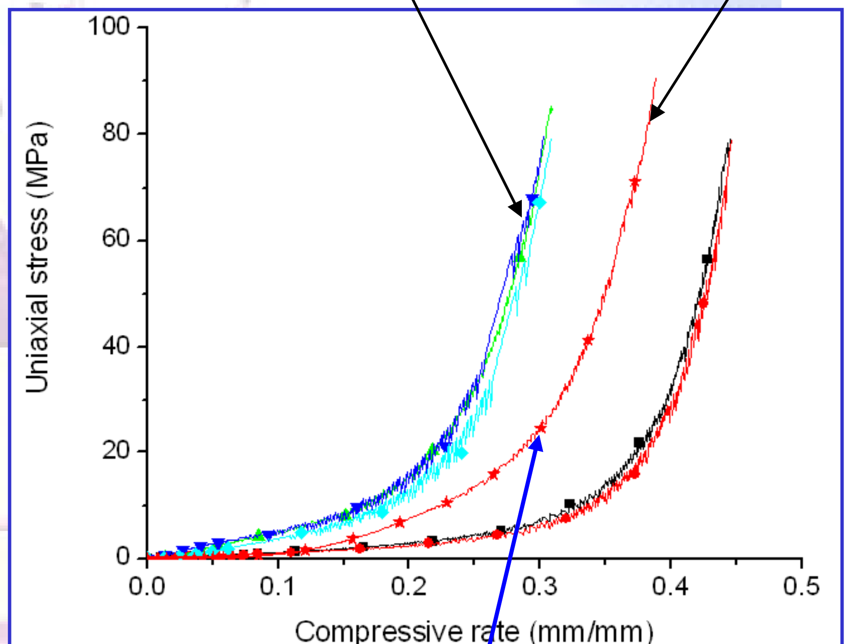
	Tap Density (g/cm^3)
lot1	0.929
lot2	0.931
	1.127
	1.147
lot5	1.126



Li Ming et al., PEP., Vol.32(5), 401-405 , 2007

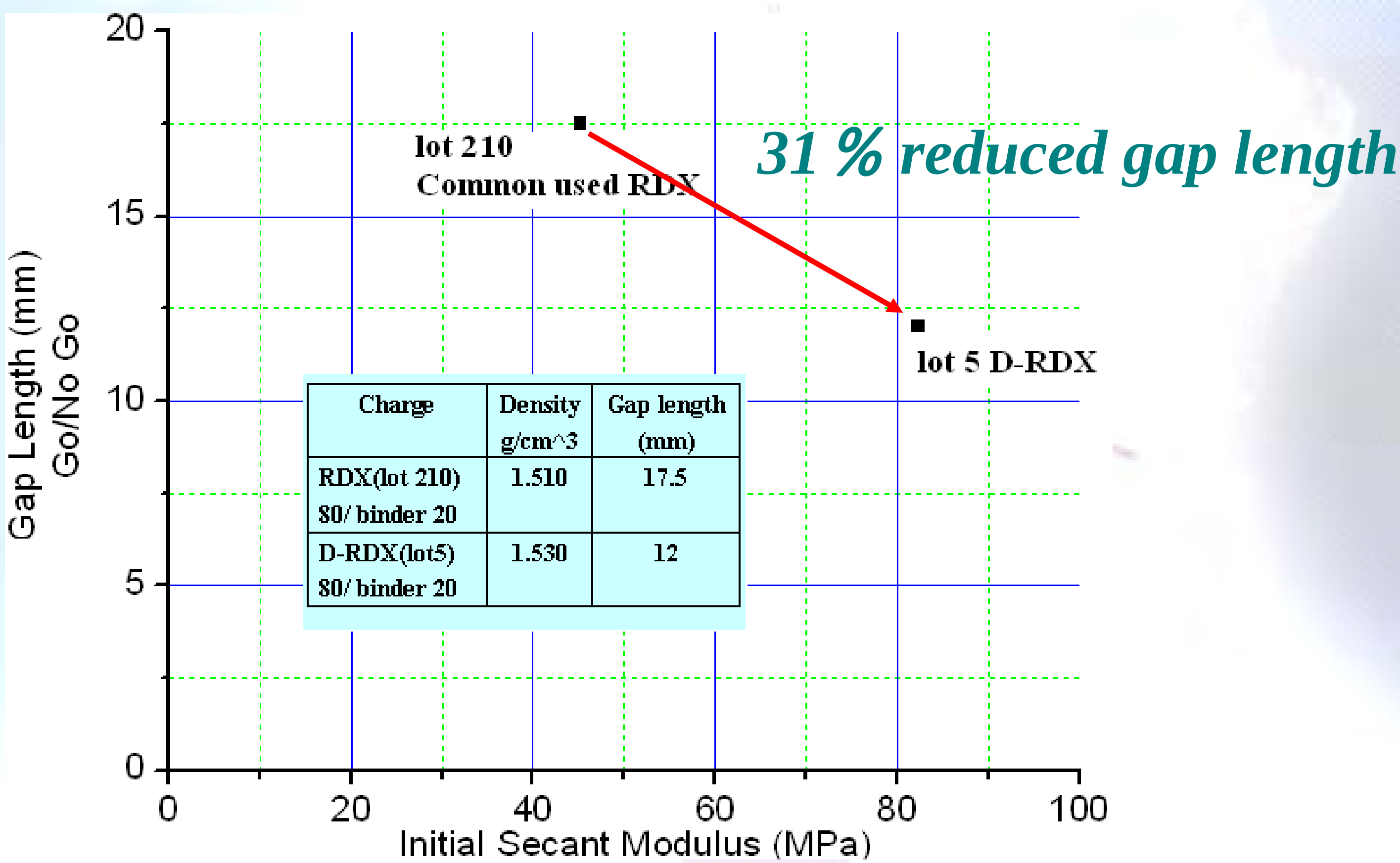
What Lot210 shows





** D-RDX developed by ICM*

2. Bridging Quasi-static Mech. to Gap tests



Charge	Density g/cm^3	Gap length (mm)
RDX(lot 210) 80/ binder 20	1.510	17.5
D-RDX(lot5) 80/ binder 20	1.530	12

ISM vs. Shock sensitivity (Li Ming et al, submitting)

3. Compared with other commonly used methods

(a) Optical Microscopy with Matched Refractive index

Qualitative observation for internal defects

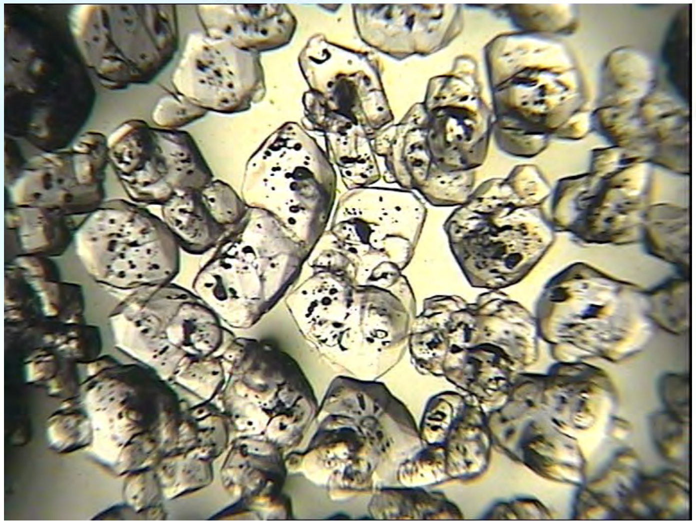
(b) Density Gradient Tube

Quantitative measurement of the particle apparent density (PAD)

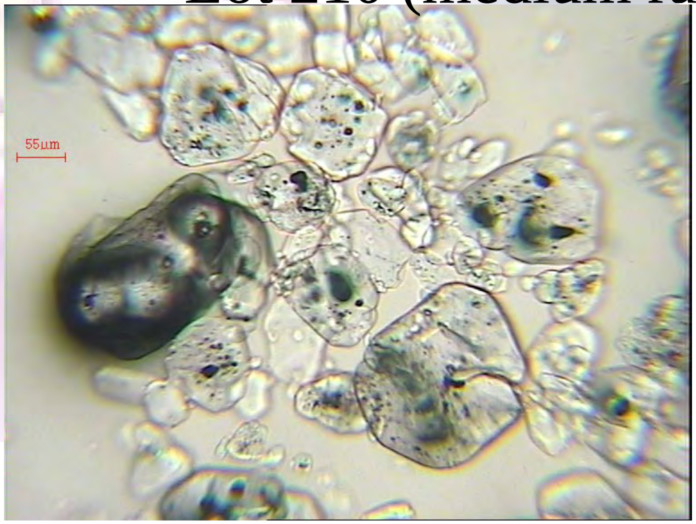
OM pictures

D-RDX（lot 5）

lot 1（coarse raw）



Lot 210 (medium raw)



PAD by DGT

Lot 2 (Coarse）：
1.7925(1.7866~1.7966)

Δρ≈0.001

Lot 210(Medium）:
1.7931(1.7887~1.7987)

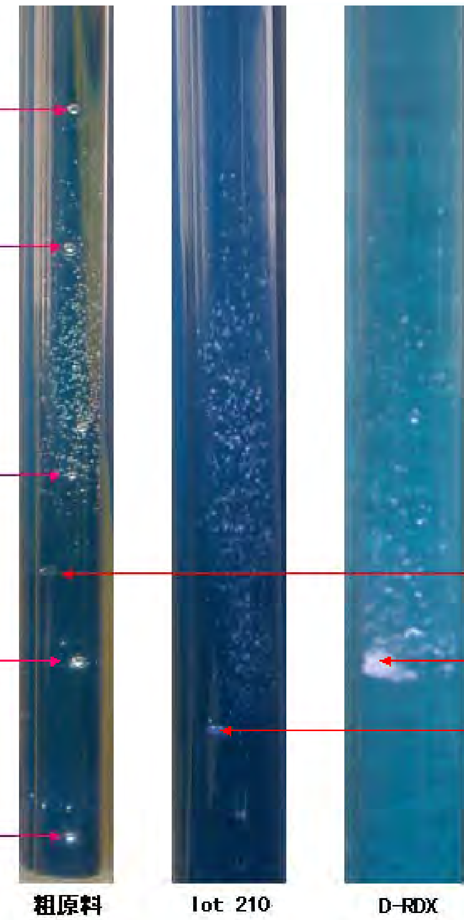
Δρ=0.0061

D-RDX（lot 5）:
1.7992(1.7982~1.7995)

Δρ≈ 0.0067

Note the TMD of RDX: 1.806g/cm^3

glass floaters



Suspending Single crystals

Expanded uncertainty< 0.0004g/cm^3

Summary

CST is a very easy and cheaper method which can definitely differentiate/ evaluate the quality of commonly used RDX and RS-RDX(D-RDX here).

The Initial secant modulus (ISM) are defined to quantify the stiffness of the ensembles of the energetic crystals. It shows that the size/shape effects are limited and the major comes from the internal defects.

The results from CST are very consistent with those from OM and DSG and Gap test.

The results from HMX, CL-20 as well as more details related to gap tests will be discussed in the future, hopefully, next Workshop.

Thanks for attention!