

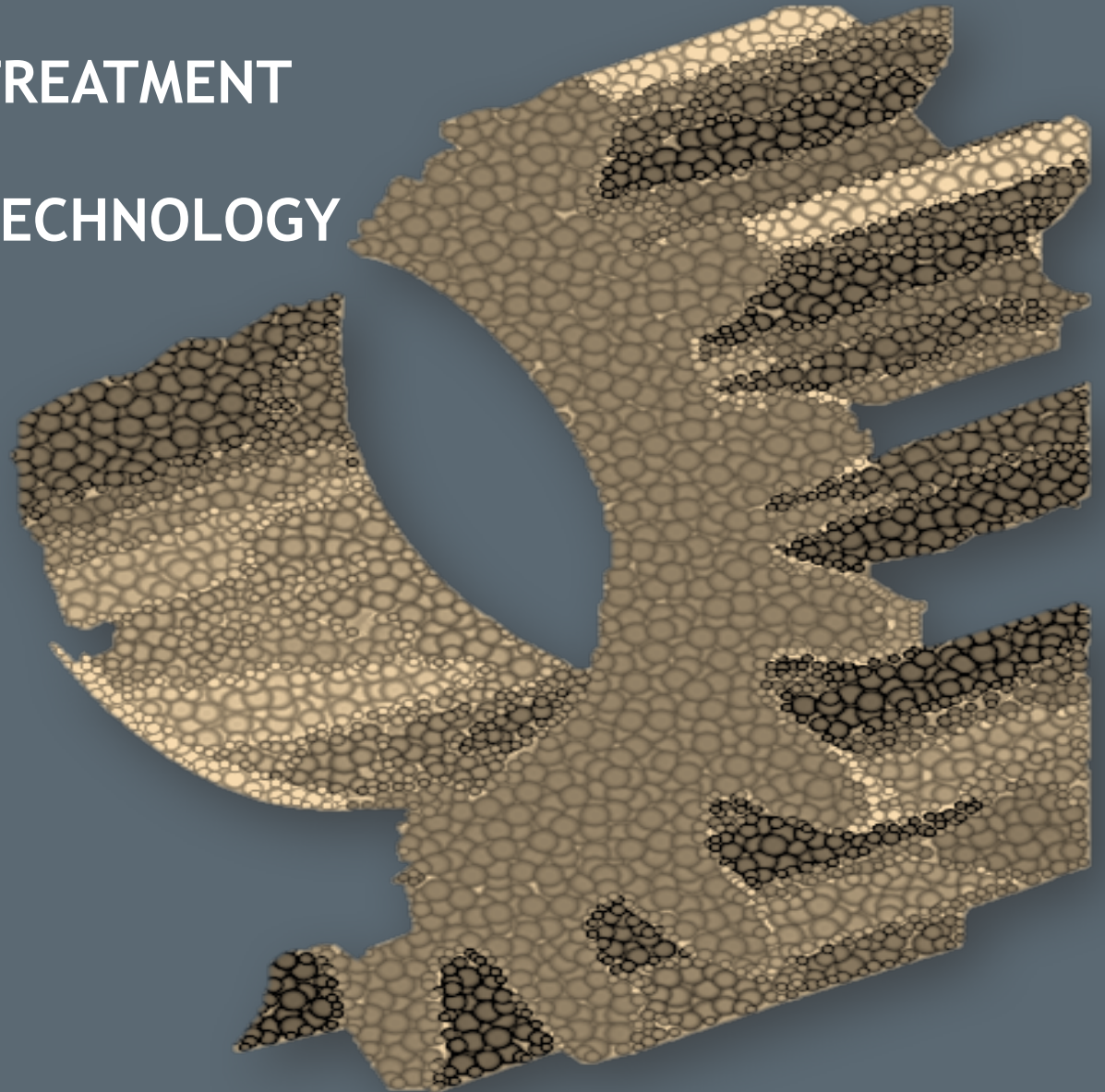
**DEEP CRYOGENIC TREATMENT**

**A GAME CHANGING TECHNOLOGY**

**MIT TECH BRIEFING**

**OCTOBER 30, 2012**

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DCI CORPORATION**



Deep cryogenic processing is a one time immersion of metal objects in - 320 °F for 24 - 75 hours.

**WHY USE CRYOGENICS?** Reduce metal on metal wear by 40 - 70 %  
Reduce multiple types of corrosion 30 - 60 %  
Reduce fatigue cracking by 20 - 60%

**WHAT METALS CAN BE TREATED?** Steel, copper, aluminum, tungsten, super alloys

**WHAT ACTUALLY HAPPENS TO THE METAL?**

The grain structure densifies and releases stress, voids disappear, carbide particles bind weaker elements, the steel becomes wear and fracture resistant, the surface finish is much smoother.

**IN WHAT FORM CAN YOU TREAT ITEMS?**

Raw material/castings/forgings ; fully machined and finished discrete parts up to assembly level.



# DEEP CRYO HAS BEEN AVAILABLE SINCE WW II

**COST/BENEFIT** Cost is ~ 5-8% of the original item to double service life.

**DOES DIMENSIONAL SIZE REMAIN CONSTANT?** Yes.

**WHERE IS IT USED NOW?** Bearings, brakes, engines, guns, turbines, machine tools, dies, landing gears.

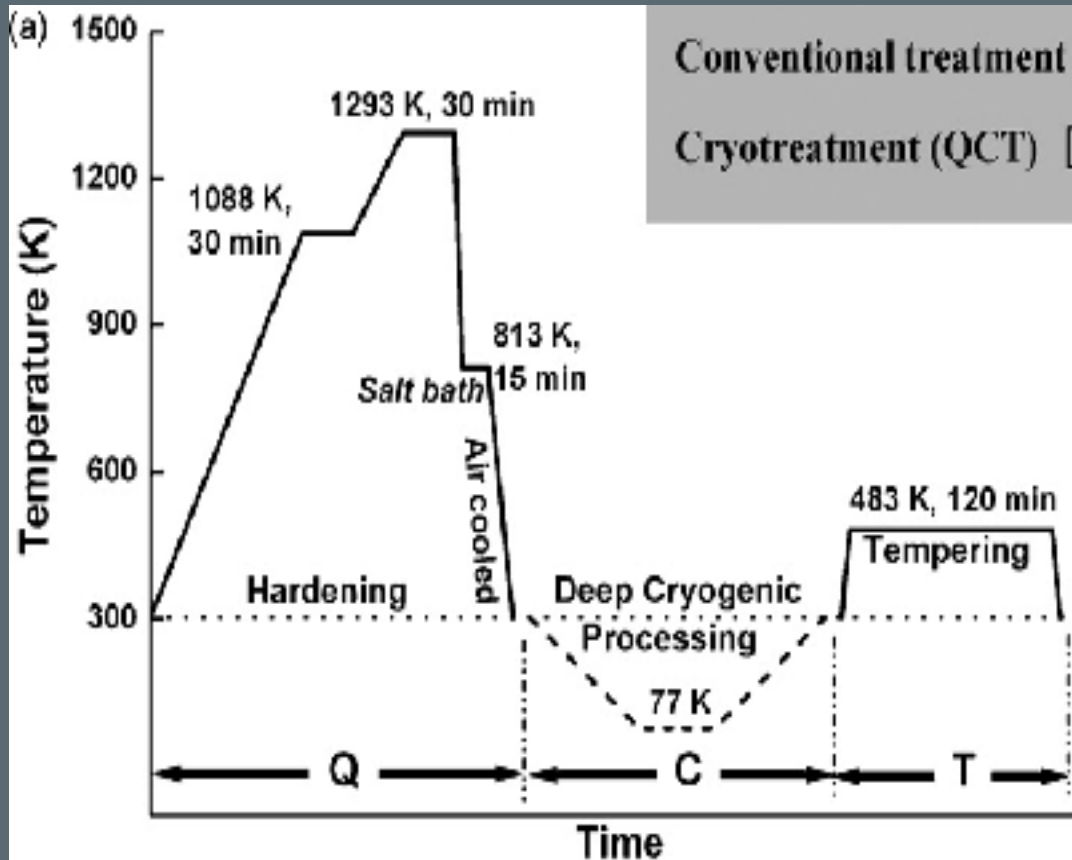
**WHERE COULD IT BE USED NEXT?** Gas and oil drilling equipment, jet engines, ships and naval applications, all metal-on-metal wear parts, windmill gearboxes, electric and gas automobiles, industrial bearings and power units.

## **WHY HASN'T THIS TECHNOLOGY BEEN EXPLORED FURTHER?**

- Lack of explanation for the phenomena behind the cryogenic effect.
- No existing procedure for validating or certifying cryogenic application.
- No prior business 'champions' pushing extended life of metal wear items.
- An absence of industrial, commercial & military funded cryo research.

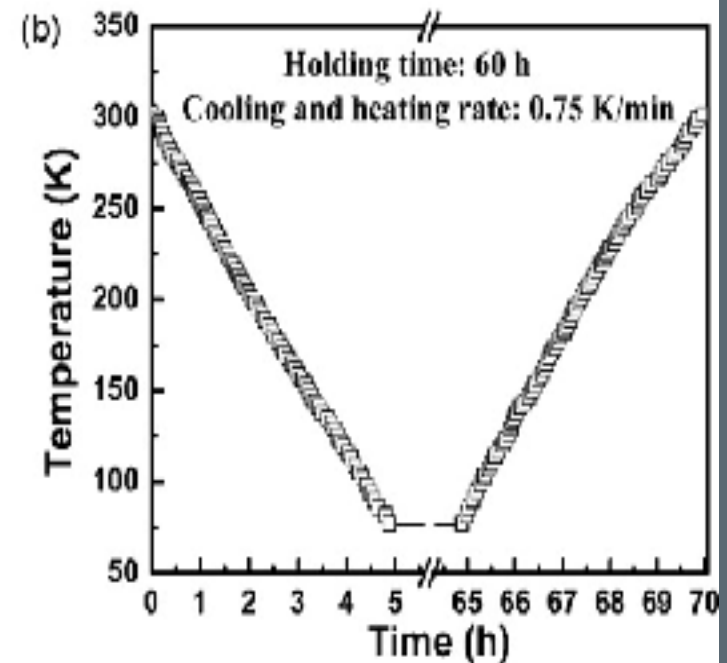
# THE DEEP CRYOGENIC TREATMENT PROCESS

(SAMPLE TREATMENT RECIPE - EACH MATERIAL REQUIRES A DIFFERENT PROCEDURE)



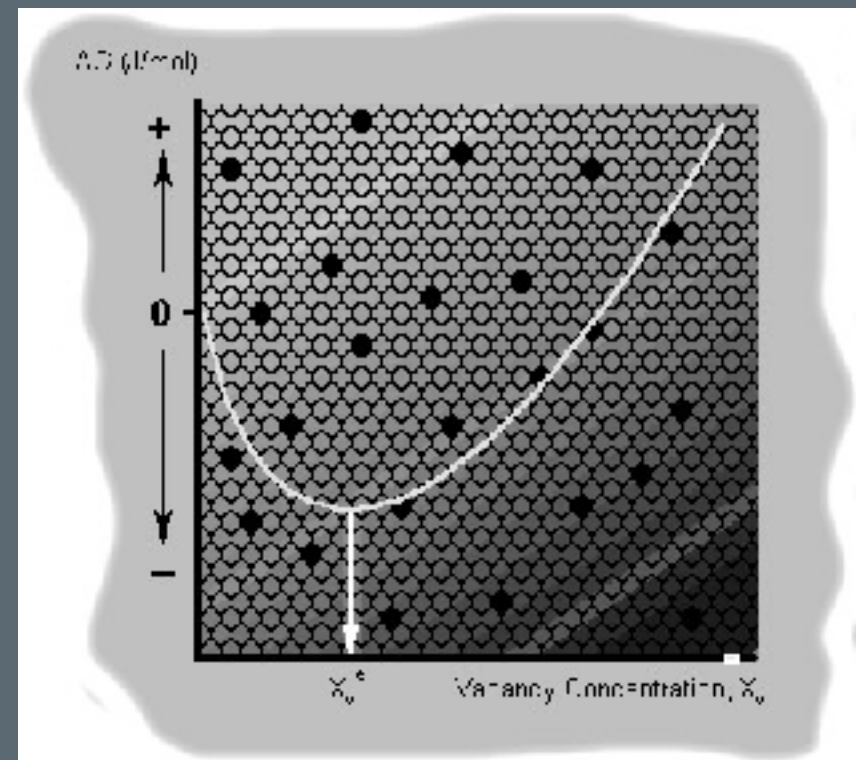
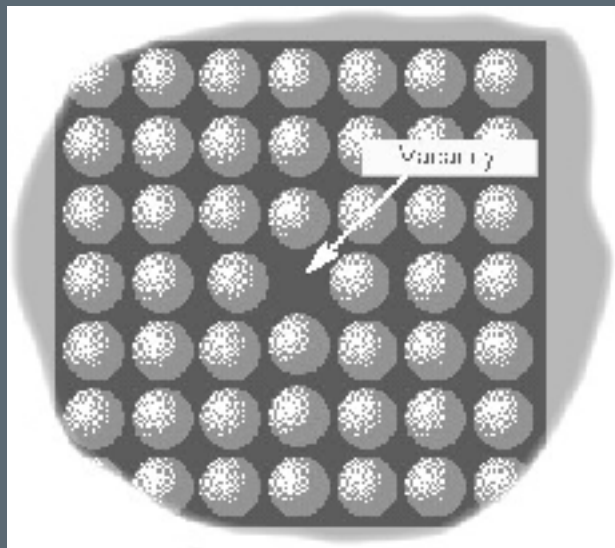
Conventional treatment (QT)  $\square \rightarrow Q \rightarrow T$

Cryotreatment (QCT)  $\square \rightarrow Q \rightarrow C \rightarrow T$



# SCIENCE

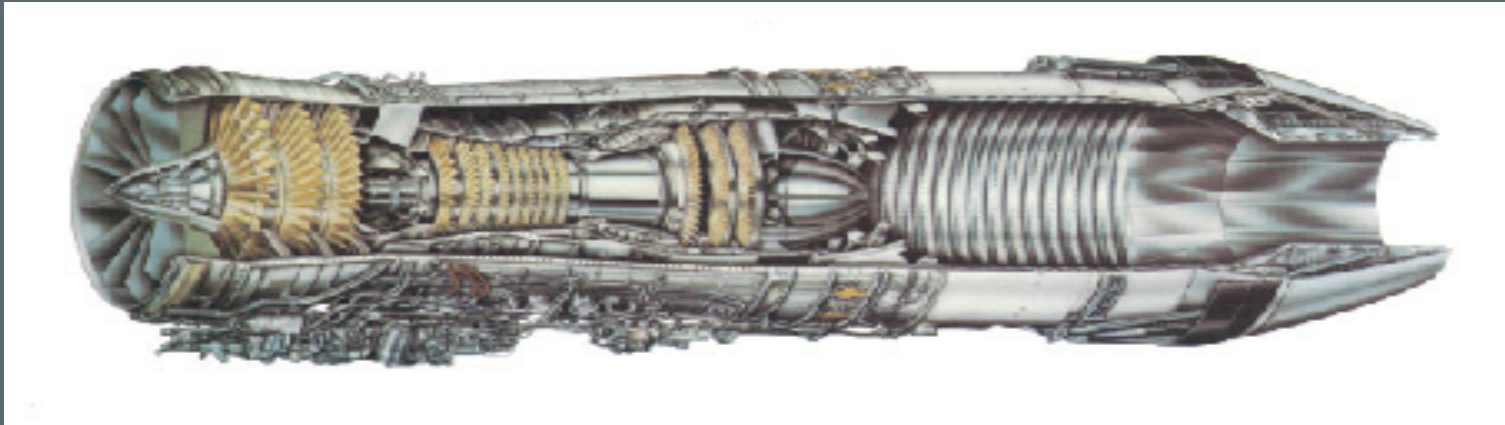
- The metallurgical changes resulting from cryo seen via SEM/TEM/EBSD:
  - Complete the austenite to martensite conversion without embrittlement
  - Precipitation of fine primary and secondary ( $\eta$ ) carbides
  - Lattice phase change from face centric to body centric tetragonal
  - Material grain refinement; edge and boundary diffusion





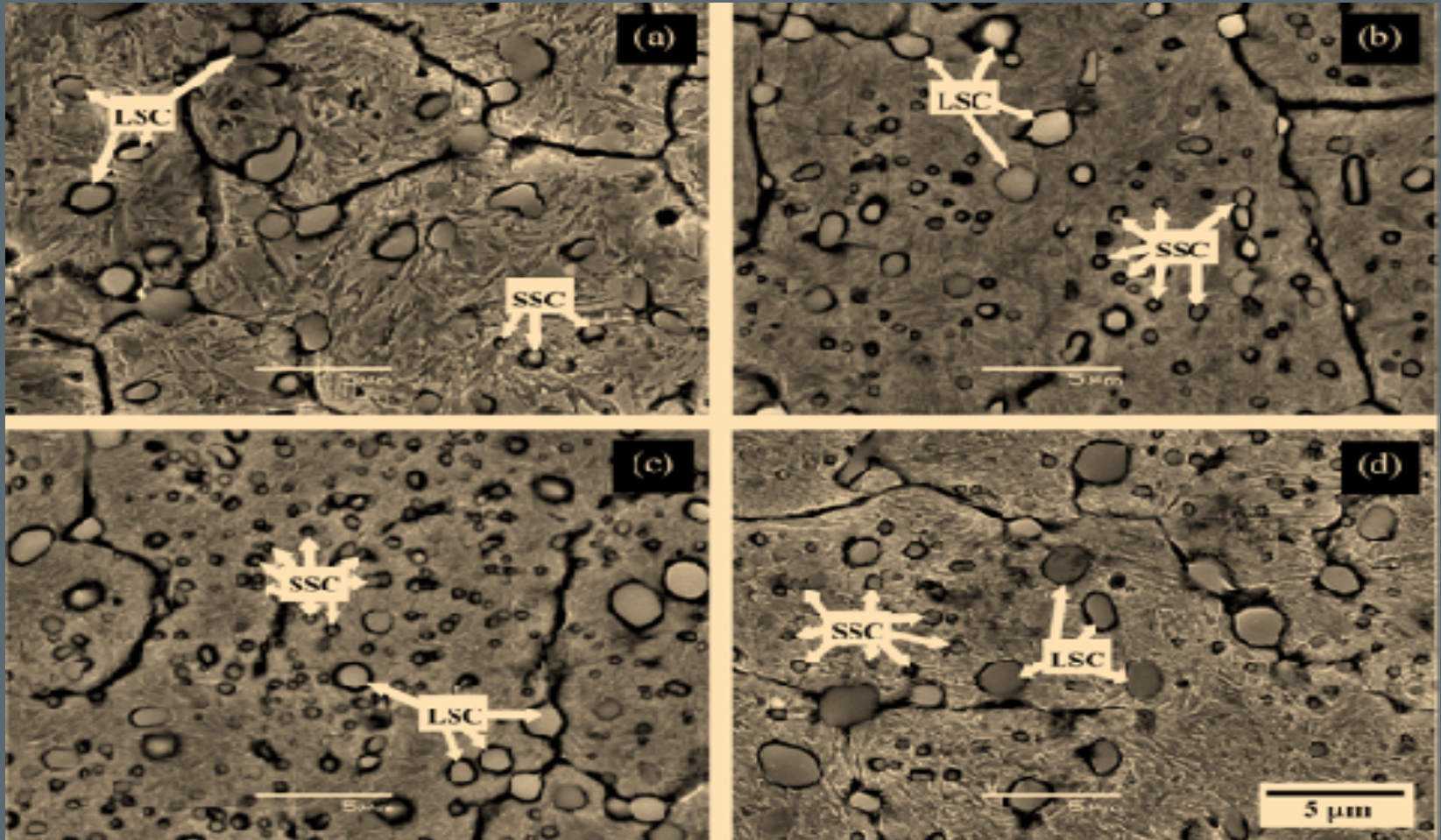
## THE “CONCRETE EFFECT”

- Deep sub-zero processing causes precipitation of eta carbides
- Finish tempering ‘locks in’ the precipitate phase
- The formed eta carbide particles act as a matrix binder
- Stress concentration is minimized; reduction in crack initiation
- SSC particles provide barrier for crack propagation

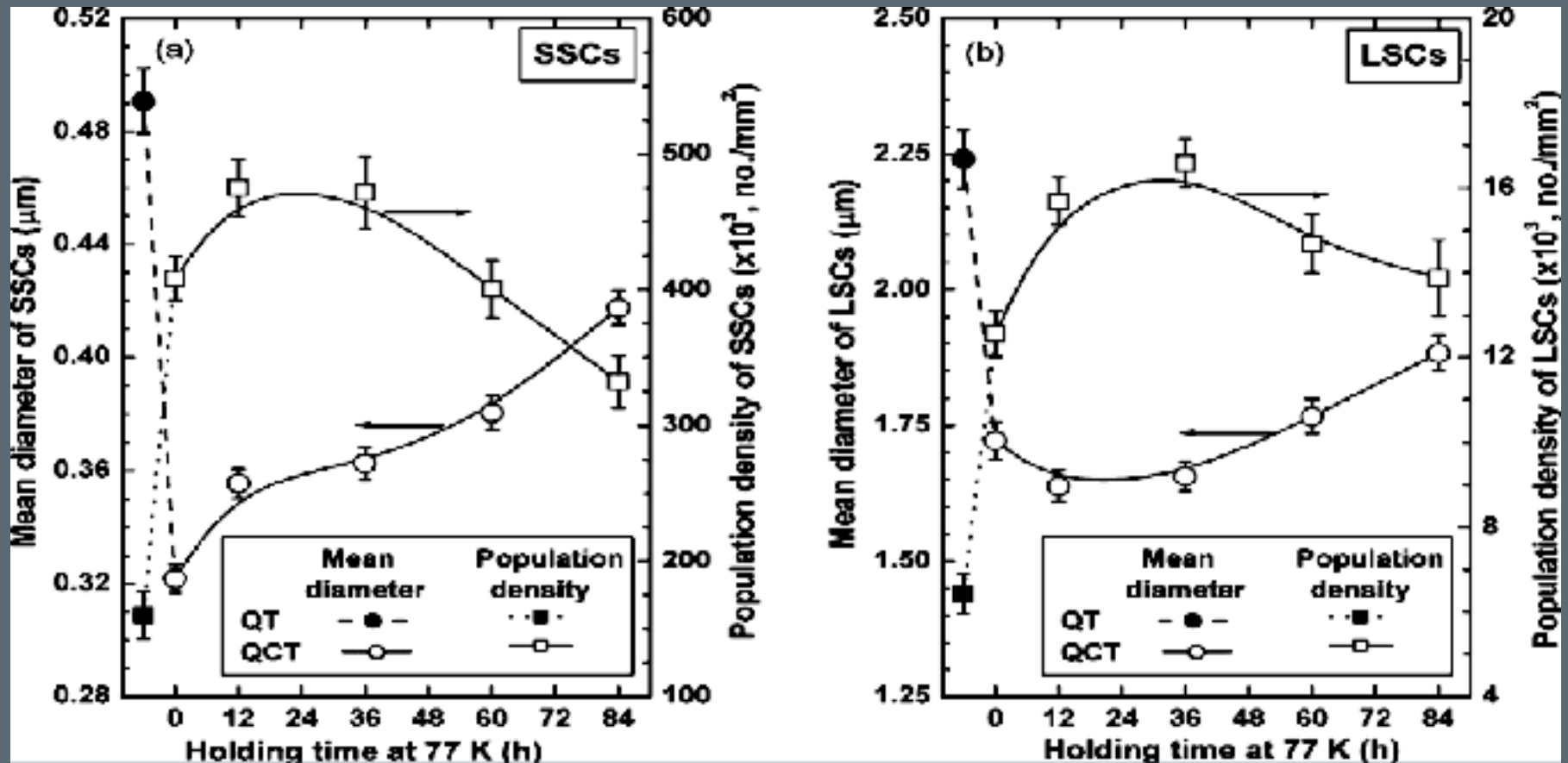


## SEM MICROGRAPHS

QT (non-cryo) and QCT (cryo treated) specimens exhibiting size, morphology and distribution of small secondary carbides (SSC) and large secondary carbides (LSC): (a) QT, (b) QCT12, (c) QCT36 and (d) QCT84 sample



# Mean Diameter and Population of Small (SSC) and Large (LSC) Secondary Carbides -

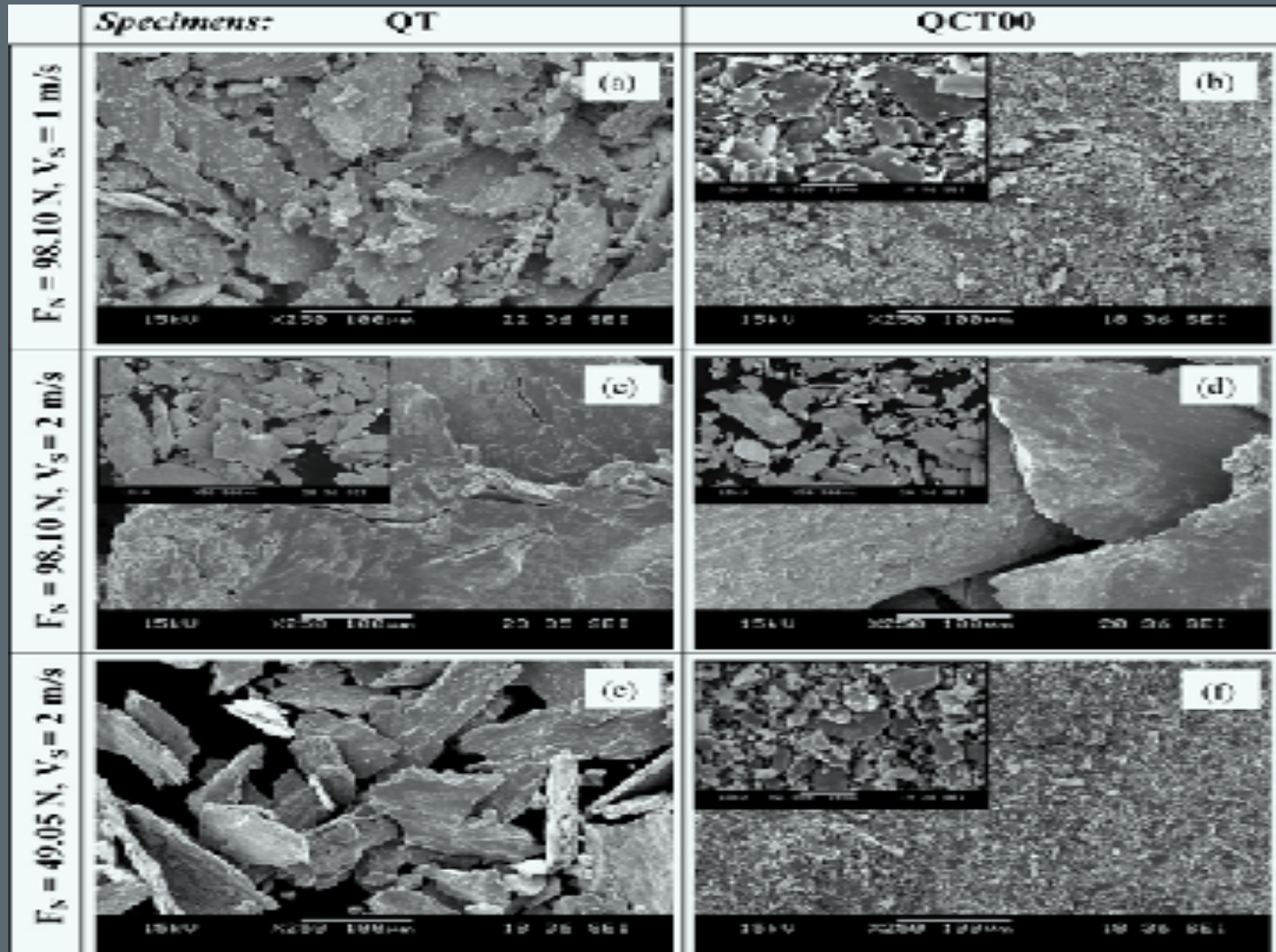




# Wear Debris Generated - Pin on Disc Testing

Non-Cryo material

Cryo treated material

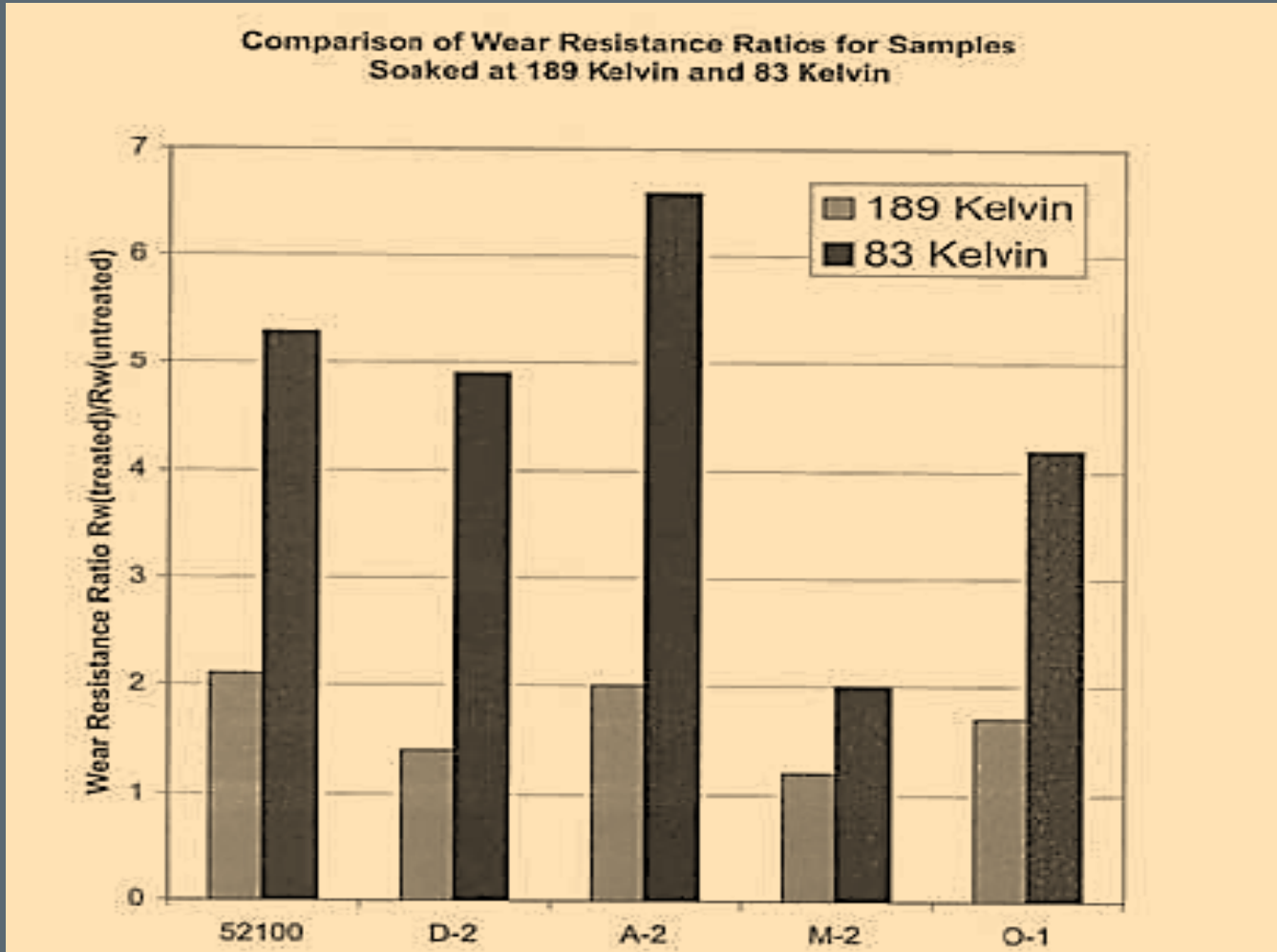


# CRYO SCIENCE AND MATERIAL SUMMARY

- Exposure of materials to deep-sub zero processing causes permanent and irreversible change to metal microstructure
- Increased mechanical properties due to temp induced order
- Atomic packing density is increased
- Abrasive wear and surface/sub-surface delamination retarded
- Crack initiation and formation substantially reduced
- Effect appears more dramatic in single versus dual phase alloys
- Cryo attributes responsible for transformation differ based on each alloy - different cryo processing recipes yield varying results
- The process works in billet, cast, sintered, HIP, forged and machined form
- New tools required to analyze, validate application of the cryo process
- Phenomenon being explored - scientific basis for improvements being analyzed

# Wear Resistance

1972 Barron Study



# November 2010 AMS 4340 STEEL CRYOGENICS STUDY

WEAR - SURFACE FINISH - CORROSION

SAMPLE SIZE - 30 ea; 15 control group, 15 deep cryo processed

- 1"dia x .040" round bar, HT per MIL-H-6875 at 44 Rockwell C
- Precision surface grind all samples to 125 Ra
- Cryo process 15 ea per 12/36/18; triple temper
- Precision surface grind all samples to 10 Ra

TEST AND MEASUREMENT PROCEDURES:

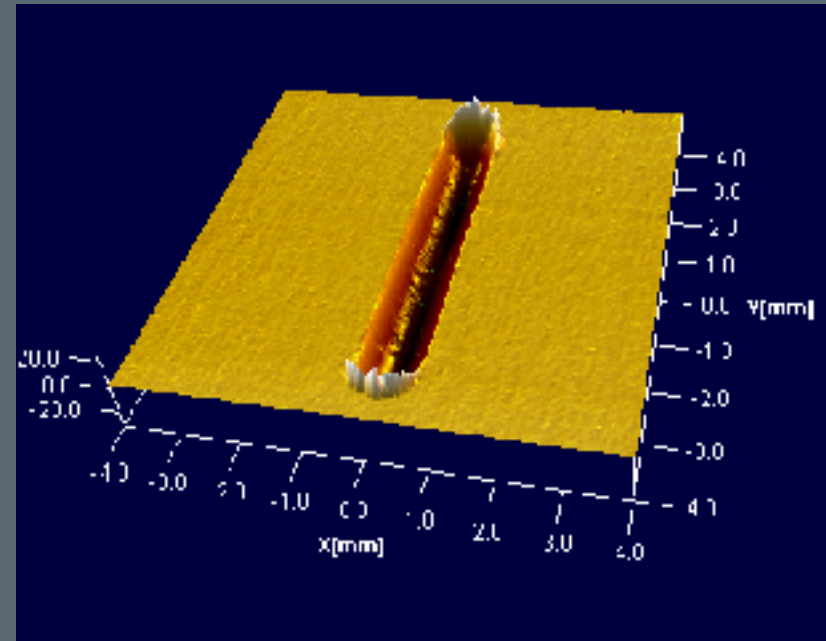
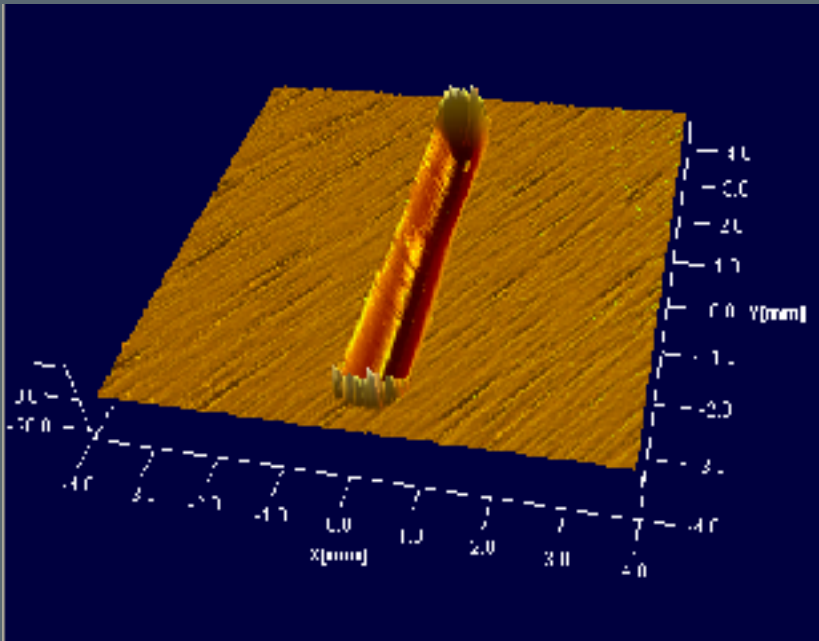
- Tribology - Pin on plate wear testing & 3D surface profiling
- Surface Morphology - 3D white light interferometry
- Corrosion - potentiodynamic and interferometry



# G133 Pin on Plate Abrasive Wear Comparison

Cryo Processed

Non Cryo Processed



Net Volume = 0.088[mm<sup>3</sup>]

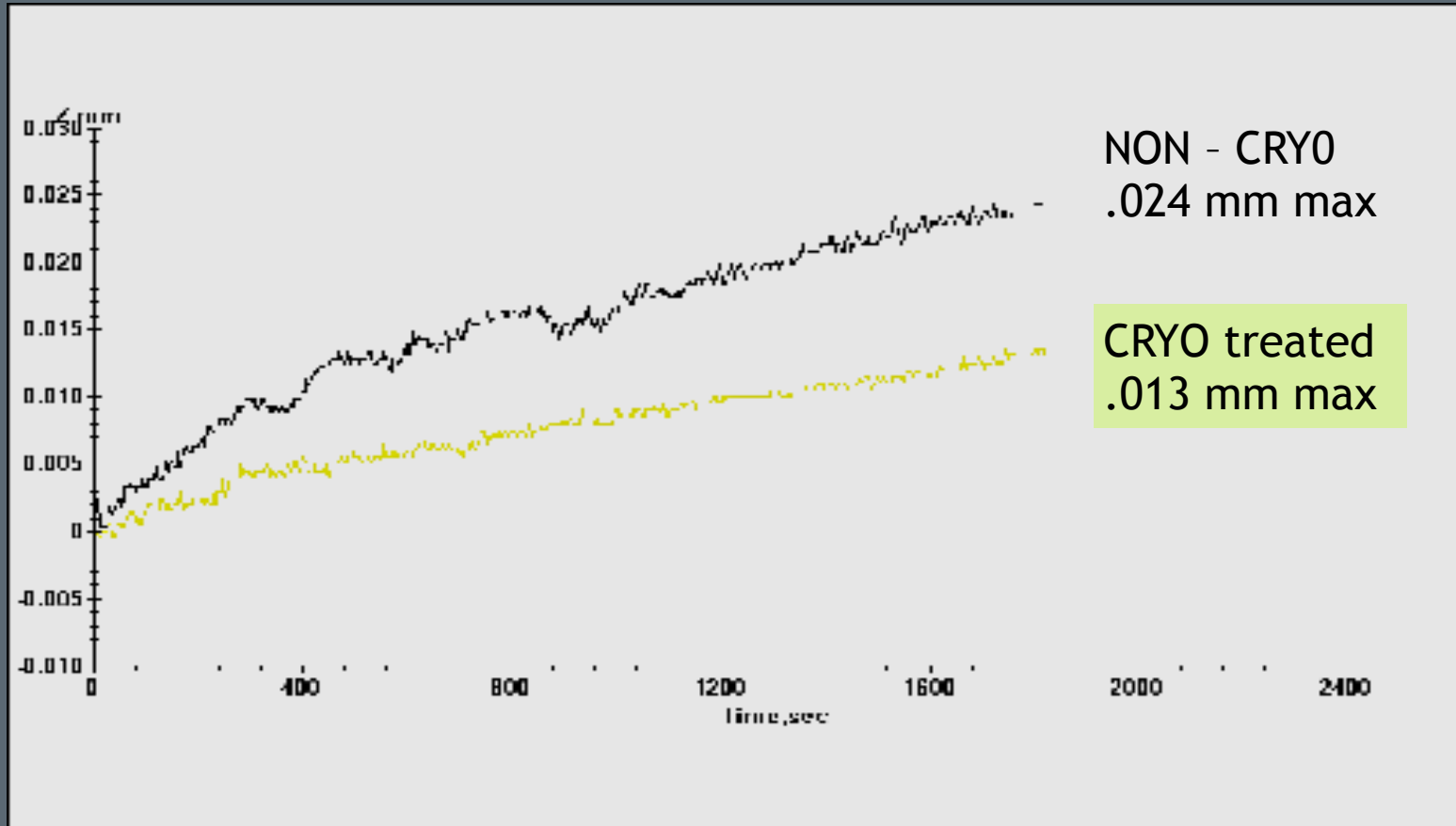
Width = 1062um

Net Volume = 0.133[mm<sup>3</sup>]

Width = 1108um

**TEST RESULT: 51% REDUCED EROSIVE WEAR VOLUME IN 30 MINUTES OF TESTING**

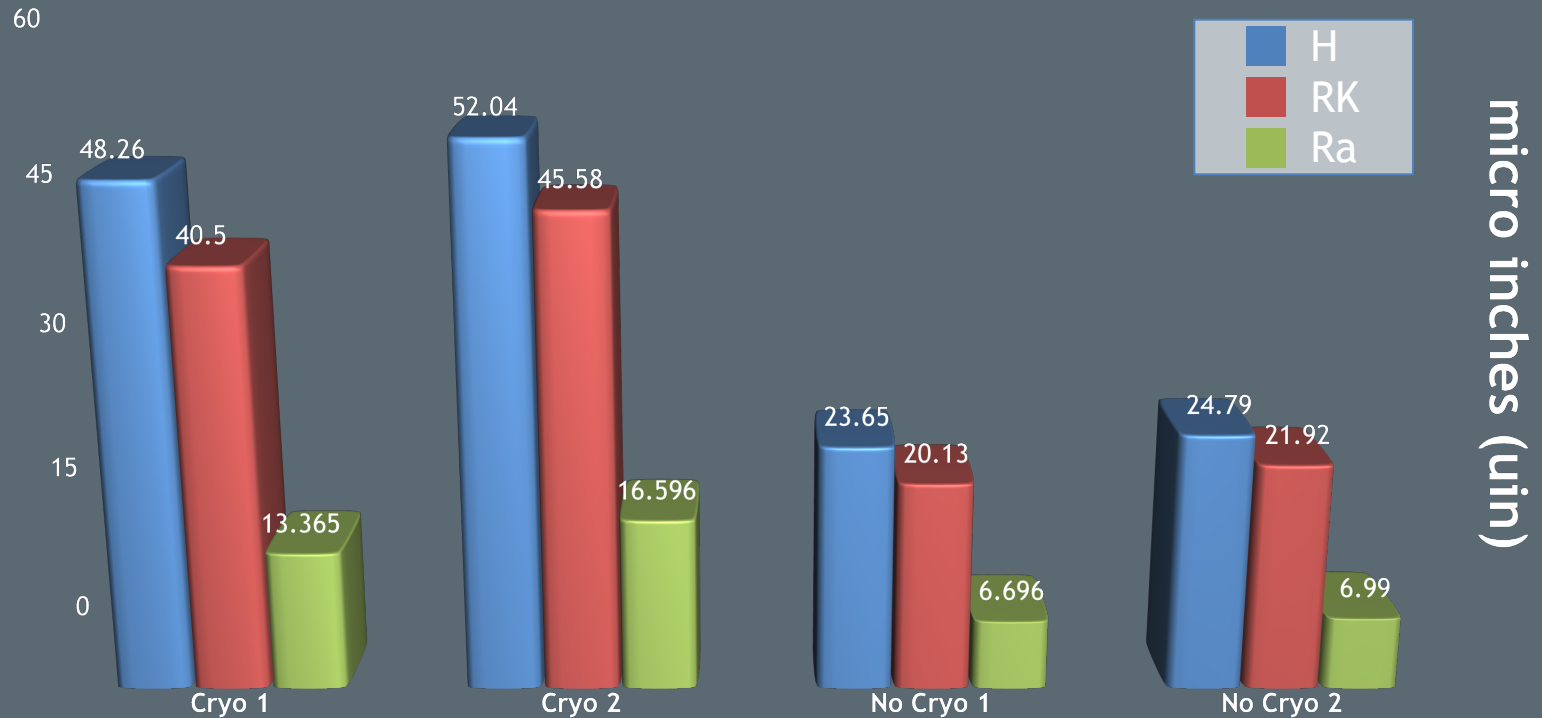
# Pin on Plate Wear Depth Comparison



**TEST RESULT: 84% REDUCTION IN WEAR DEPTH DURING 30 MINUTE SELF-MATING TEST**

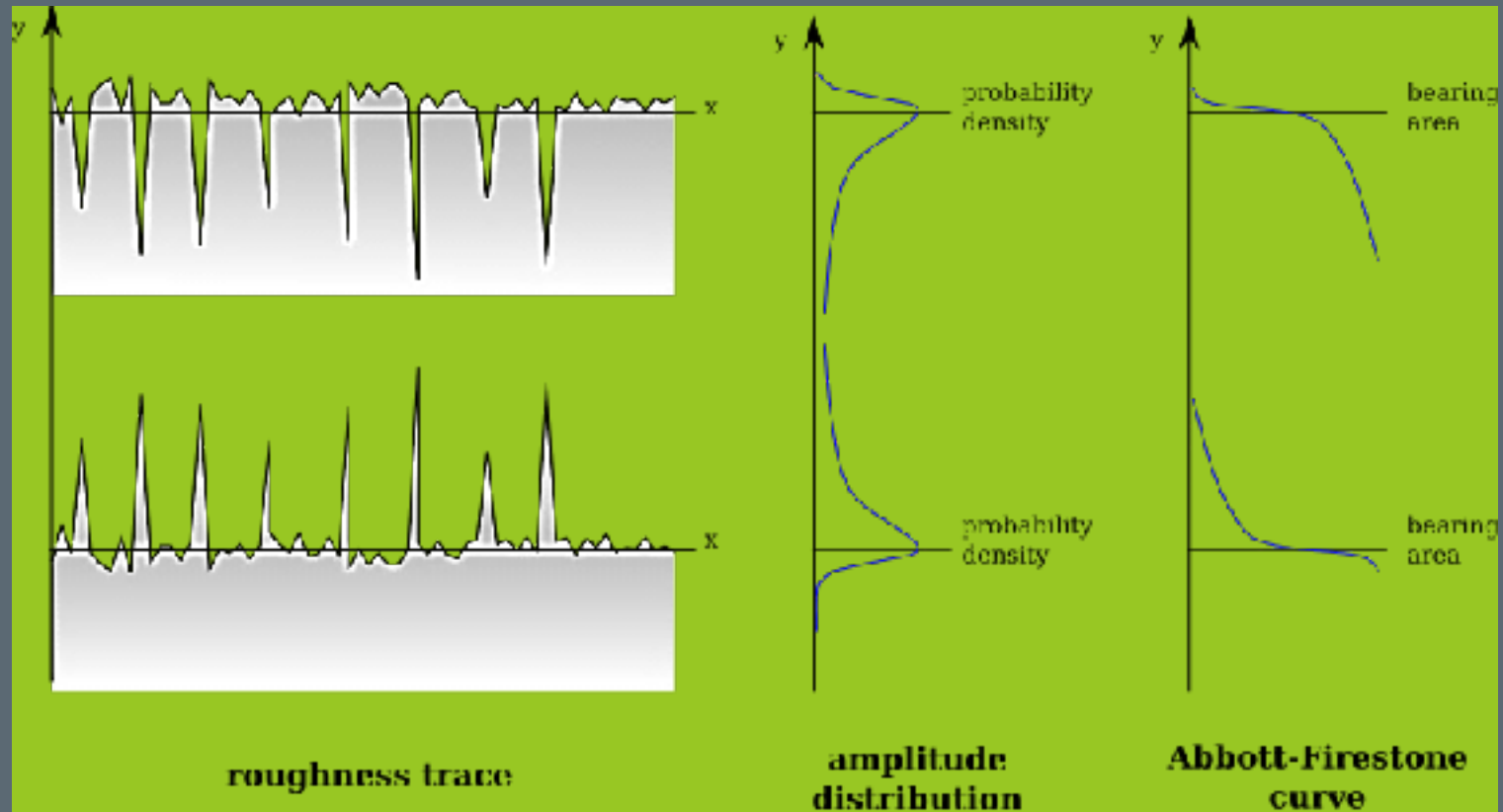
# Surface Bearing Load - Rk and Asperity Height Contrasted to Ra

Cryo vs. Non-Cryo Treated 4340 Steel 11/1/2010



**TEST RESULT: 200% IMPROVEMENT IN SURFACE FINISH (Rk) IN 30 MINUTES**

# Abbott-Firestone (RK) Curve



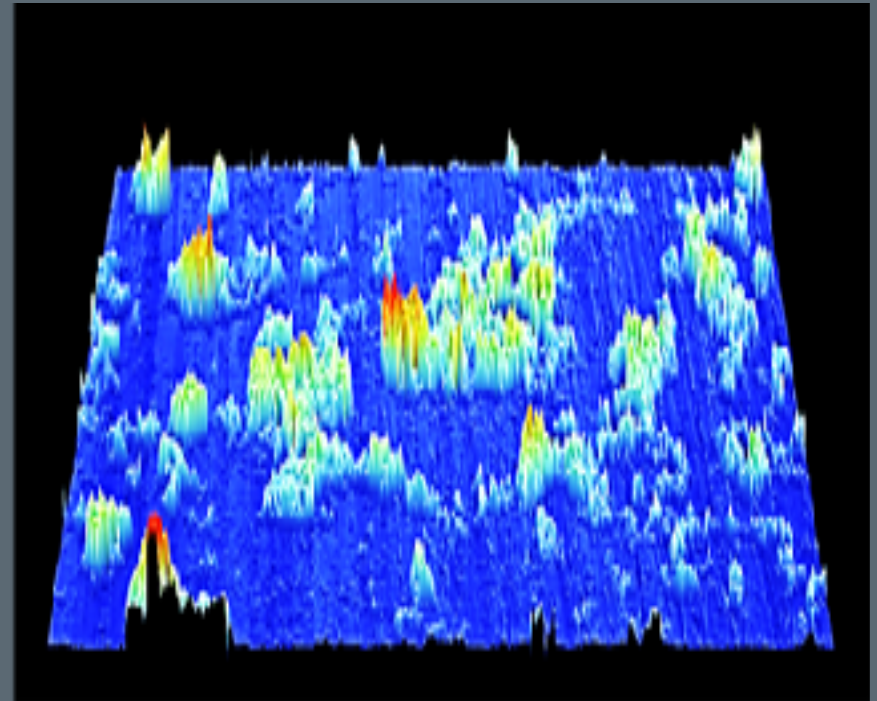
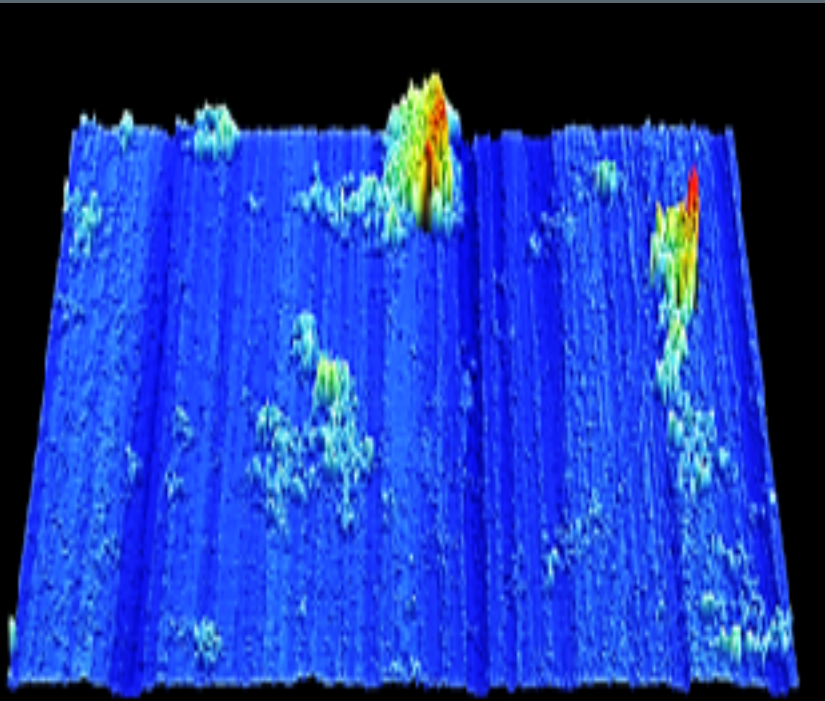


# 4340 Steel Corrosion Test # 1

Uniform Surface Corrosion

Corrosion-18 hrs in 3.5% NaCl Cryo treated coupon 100  $\mu\text{m}$

Corrosion-18 hrs in 3.5% NaCl Non-cryo treated coupon 100  $\mu\text{m}$



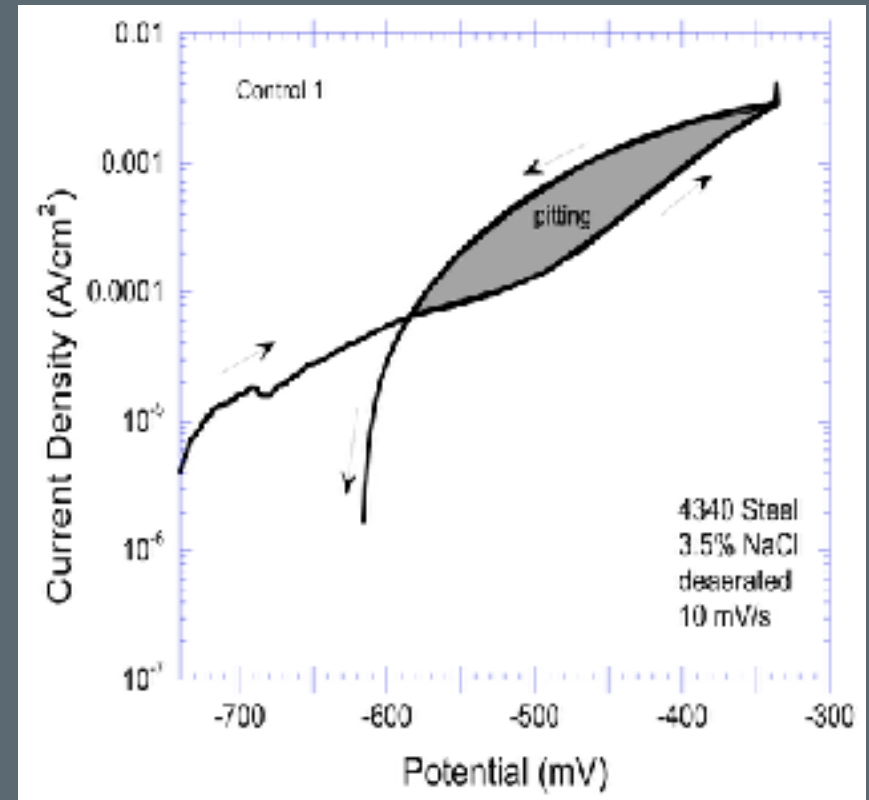
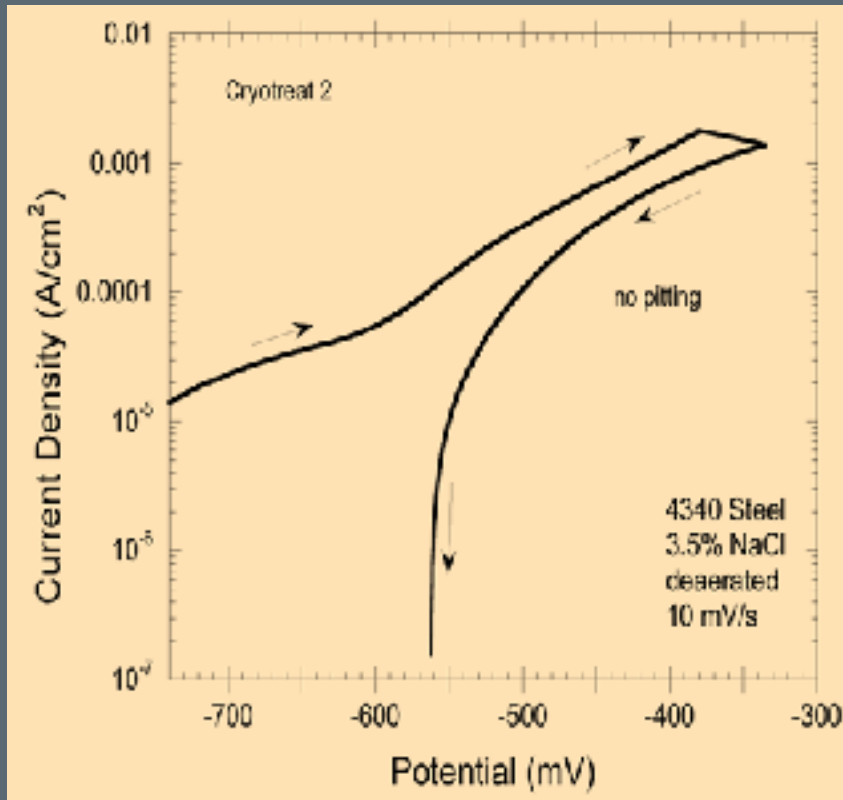
**TEST RESULT: 84% REDUCTION IN GENERAL CORROSION (VOLUMETRIC)**

# 4340 Steel Corrosion Test # 2

Potentiodynamic Test for Pitting Resistance

Corrosion 36 hrs in 3.5% NaCl  
3 Cryo treated coupon samples

Corrosion 36 hrs in 3.5% NaCl  
3 Non-cryo treated coupon samples



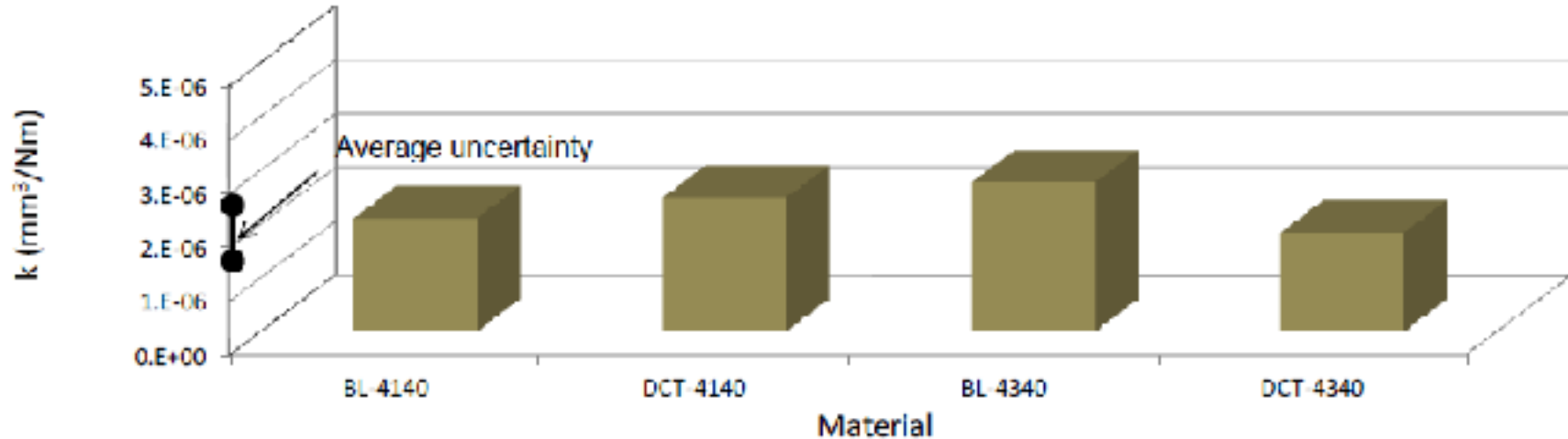
**TEST RESULT: 0% PITTING CORROSION DETECTED IN DEEP CRYO COUPONS**

# Results of Deep Cryogenic Treatment on Selected Steel Alloys

## 2009 DCI-ARDEC CRADA - 52100 and 4340 Steels

SUMMARY RESULTS: Break	Yield KSI	Peak Strength KSI	% %	Strain Stress %	Reduction Elong. @ %
<b>20% INCREASE IN YIELD STRENGTH</b>					
No Cryo 52100, Baseline	268	359	2.5	3.9	6
Cryo 52100, Cryo -Tempered	<b>317</b>	382	1.6	3.5	1
Cryo 52100, Tempered - Cryo	<b>320</b>	376	1.8	3.8	4.5
No Cryo 4340, Baseline	221	295	15.3	12.5	51.7
Cryo 4340, Cryo -Tempered	<b>240</b>	300	14.2	11.6	51.3
Cryo 4340, Tempered - Cryo	221	287	15.9	12.3	51.6

- Wear rate (k) of the WC/Co ball is minimal while that of plates are relatively consistent across sample sets. For 4140, the baseline performed slightly better but within the average uncertainty range while for 4340, the wear rate is reduced by an average of 30-35%, which is just outside the average uncertainty range of  $\pm 20\%$ .



- Similar trends are observed for average wear depth as expected.

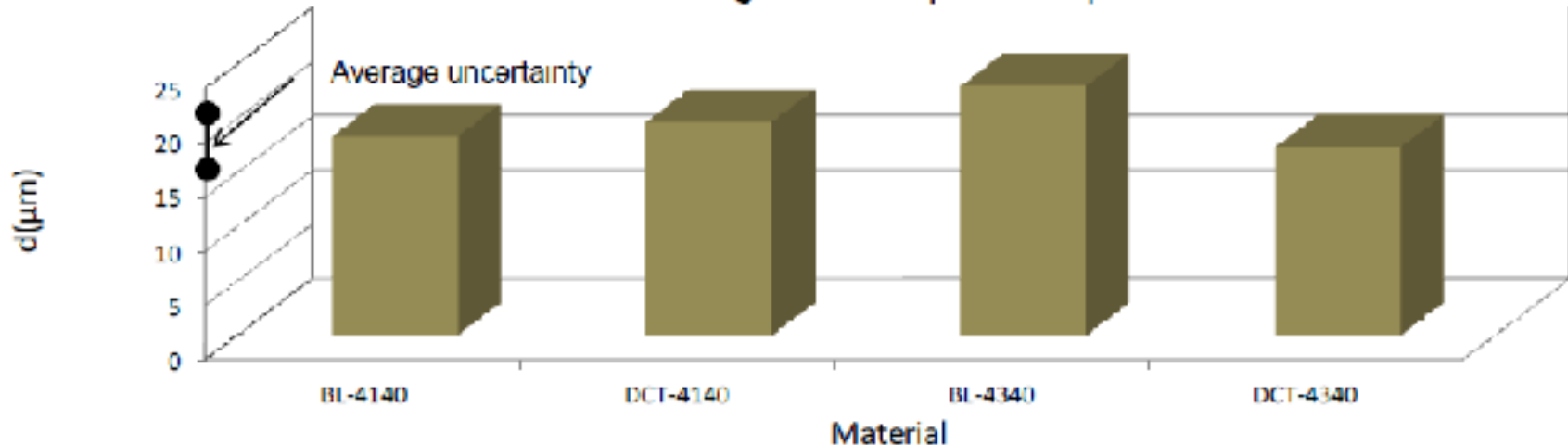




Plate Surface Post-test  
Baseline 4340

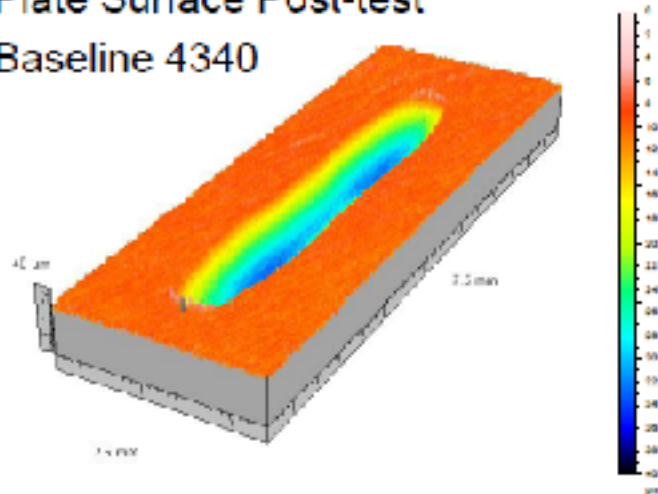
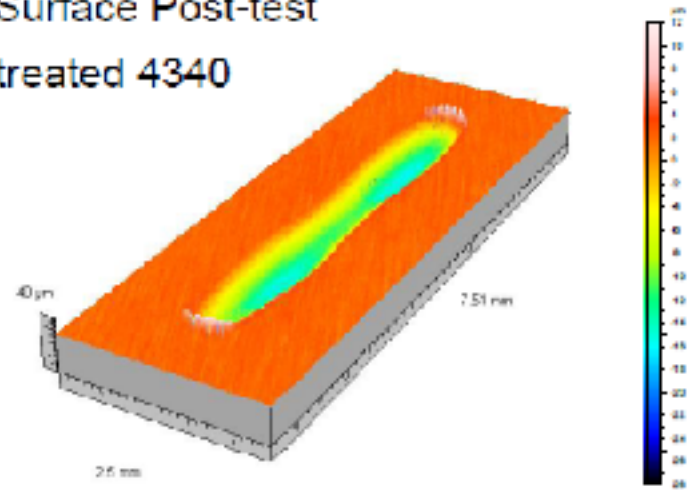
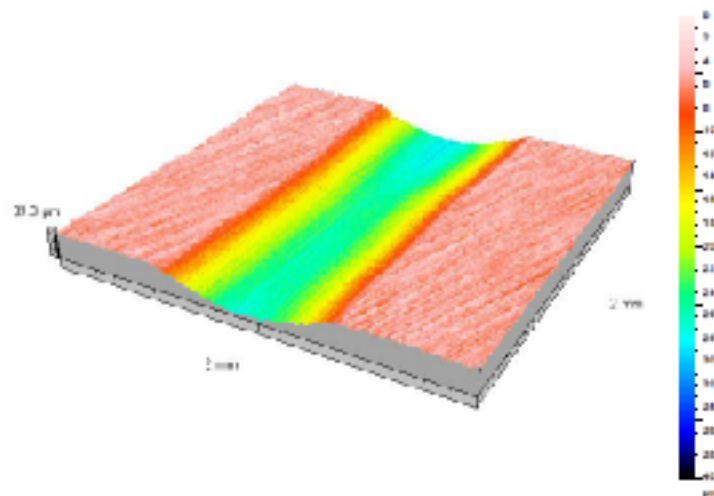


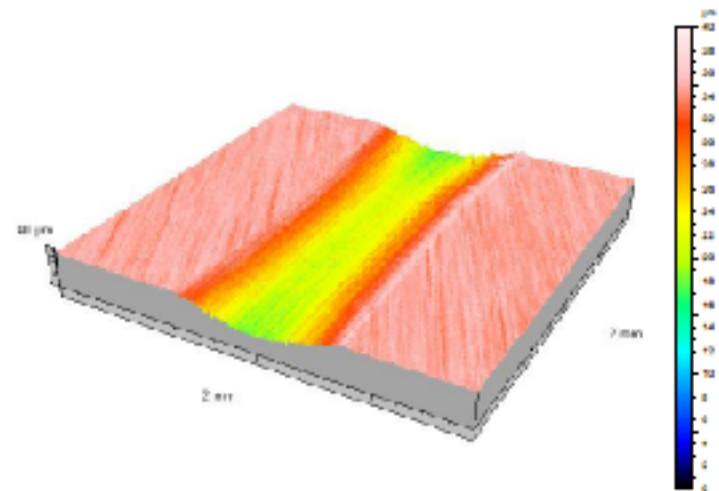
Plate Surface Post-test  
Cryo treated 4340



High Resolution

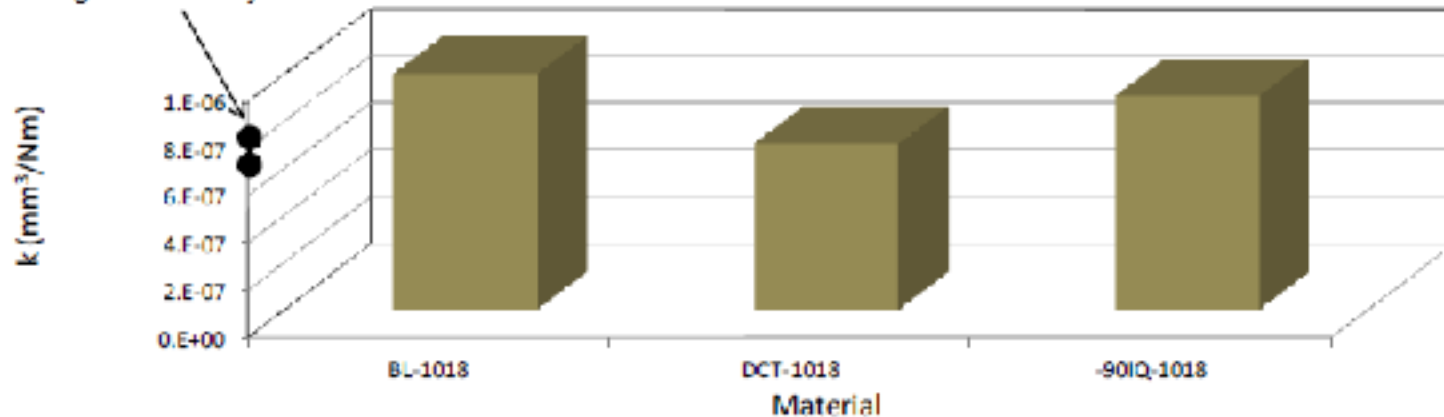


High Resolution



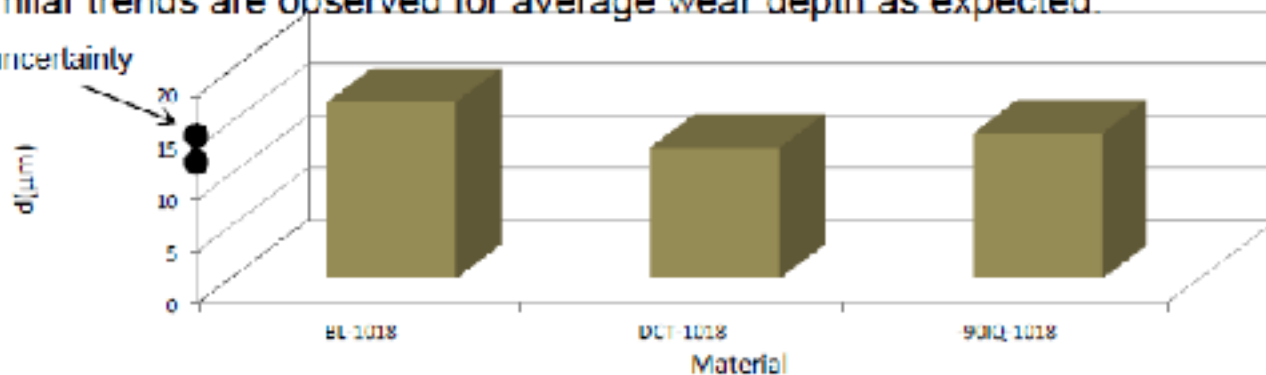
- For 1018, the DCT specimens the wear rate is reduced by an average of 30% compared to non-cryo treated specimens, which is well outside the average uncertainty range of  $\pm 0.07 \times 10^6 \text{ mm}^3/\text{Nm}$ .

Average uncertainty



- Similar trends are observed for average wear depth as expected.

Average uncertainty



# CURRENT DEEP CRYO MILITARY APPLICATIONS

## Project Descriptions

- Increase wear life on 4340 steel weapon platforms
- Reduce corrosion on inconel, nickel and HSLA hull materials
- Increase coating bond to substrate on laser guided munitions
- Improve thermal management on Be, AlBe, CuNi guidance housings
- Increase ballistic defeat mechanism on tank tiling
- Reduce fatigue and corrosion on JSF components
- Reduce use of DU by increasing lethality of WHA KE penetrators
- Increase penetration defeat on B4C body armor

# CURRENT DEEP CRYO INDUSTRIAL APPLICATIONS

## Project Descriptions

- Reduce tooling and consumable cost on machined items
- Increase casting and forging die life
- Increase wear and corrosion life of turbine bearings
- Reduce corrosion in nuclear waste storage casks and containment vessels
- Reduce high temperature fatigue of Ni single crystal turbine blades
- Extend wear life on deep-hole oilfield components
- Decrease corrosion on marine sensors and navigation instruments



# RESEARCH CREDITS

- RF Barron, “Cryogenic Treatment of Metals to Improve Wear Resistance”, Cryogenics, Volume 22, No 5, August 1982, pp 409-413
- J. Cahn, “Cryogenic Processing of Weapon Components”, Cooperative Research Agreement between ARDEC and DCI, April 2009
- J. Cahn, “The Effect of DC Treatment on the Wear Rate of High and Low Carbon Steel Alloys”, Cooperative Research Agreement between ARDEC and DCI, April 2009
- J. Cahn, “Tribological and Corrosion Properties of Deep Cryogenically Treated 4340 Steel”, Cooperative Research Agreement between ARDEC and DCI, April 2009
- D.Das, “Influence of Varied Cryotreatment on the Wear Behavior of AISI D2 Steel”, Elsevier B.V., 2008, pp 297-309