

# ***Development of Ultrananocrystalline Diamond (UNCD) Coatings***

*Presented by*

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*Argonne National Laboratory*

*Materials, Sensors & Automation, and Glass Project Review*

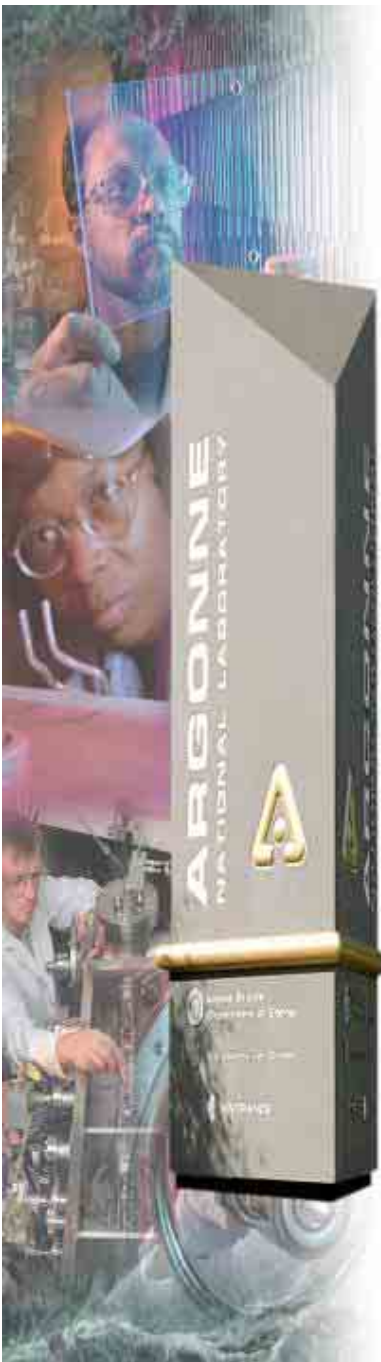
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*Arlington, Virginia*



Office of Science  
U.S. Department of Energy

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# Project Summary

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- **Goal:**
  - Use UNCD to achieve significant energy savings in IOF industries
    - *First application – SiC multipurpose mechanical pump seals*
- **Challenge:**
  - Develop technology to take UNCD from laboratory to market application
    - *Need to mass produce UNCD coated parts*
- **Benefits:**
  - Improved wear resistance and corrosion resistance of UNCD coated parts
  - 20% energy savings of 236 trillion Btu by 2020 in pump applications primarily due to reduced friction losses
- **FY05 Activities:**
  - Commission and optimize 11-inch IPLAS system (up from 6-inch system)
    - *Demonstrate UNCD coating of multiple seals simultaneously*
  - Demonstrate benefits of UNCD coatings on gas seals
  - Verify tribological benefits of UNCD coated seals
    - *Perform long-term pump tests*

# ***Project Participants – Laboratory-led project***

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- **Argonne National Laboratory**
  - Energy Systems:
    - *Jeff Elam, John Hryn (Project POC), Joe Libera*
  - Energy Technology:
    - *Ali Erdemir, Andriy Kovalchenko*
  - Materials Science:
    - *Orlando Auciello, John Carlisle, Dieter Gruen, Mike Pellin, Alex Zinovev*
- **Industry Partners:**
  - Advanced Diamond Technologies, Inc. (ADT)
    - *Neil Kane (Industry POC)*
  - John Crane, Inc.
  - IPLAS Innovative Plasma Systems
  - Morgan Advanced Ceramics
  - Northwestern University
  - University of Illinois at Chicago

# Barriers

# Pathways

# Critical Metrics

1) Uniform nucleation and growth of UNCD

- 1) *smooth films*
- 2) *good adhesion*

Understand plasma deposition and surface seeding requirements, UNCD characterization and testing

Develop seeding protocol to produce uniform UNCD films  
**(COMPLETED)**

2) Scale – up UNCD deposition process

Commission 11-inch plasma system

Multiple seals coated with UNCD simultaneously

3) Limited seal testing facilities

Use industrial facilities and expand lab capabilities

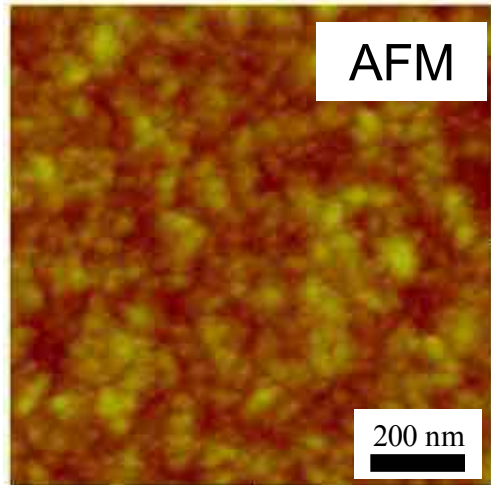
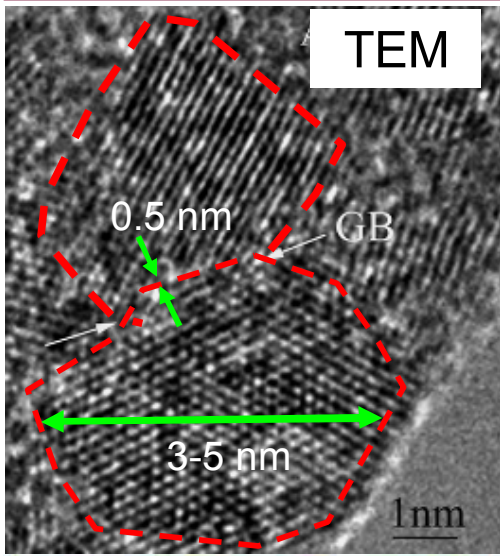
Perform successful pilot test (data indicate energy savings)

4) Commercialization of UNCD technology

Launch company (ADT)

Sign toll-processing agreement

# Ultrananocrystalline Diamond (UNCD) Properties



- Hardness - 97 GPa
- Elastic Modulus - 970 GPa
- Fracture Strength - 5 GPa

- Grain Size - 2 - 5 nm
- RMS Roughness - 0.5 - 1  $\mu$ -inch
- Friction Coefficient - 0.03

# UNCD for Multipurpose Mechanical Pump Seals



UNCD Coating



- UNCD to reduce friction and eliminate wear

# ***Technical Barrier #1: Uniform nucleation and growth of UNCD***

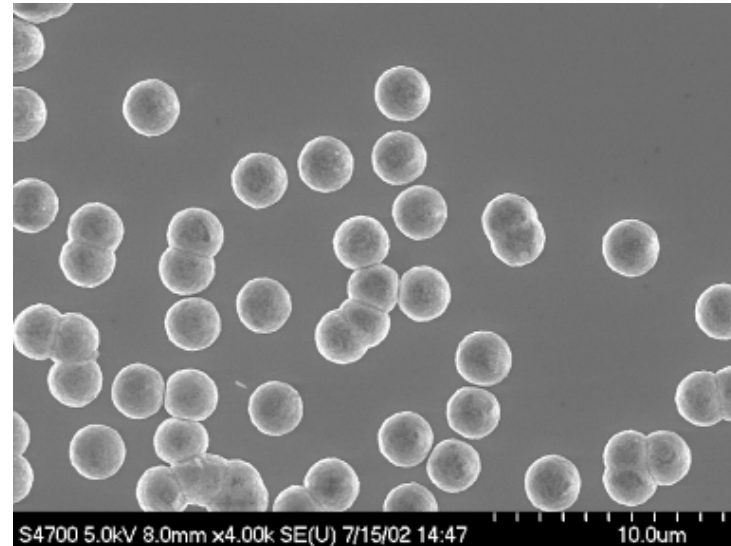
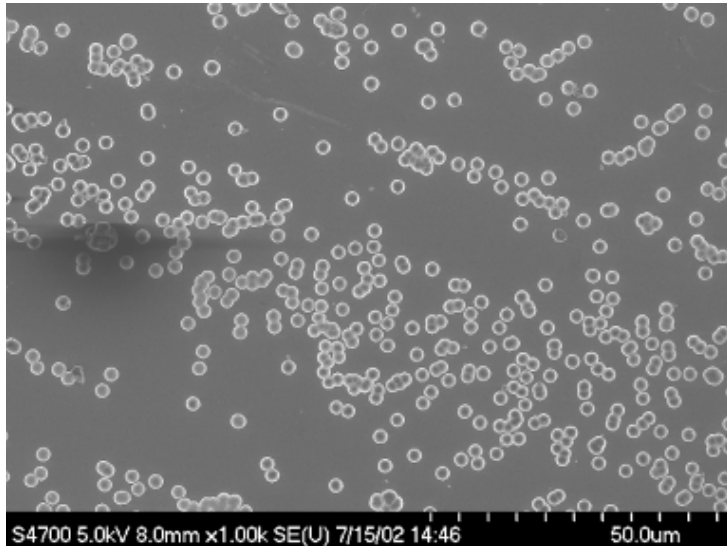
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- **Seeding to achieve smooth UNCD films**
- **Seeding to achieve excellent adhesion of UNCD to SiC Seal**

# UNCD – Example of Poor Seeding

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Sample: Unseeded CVD SiC



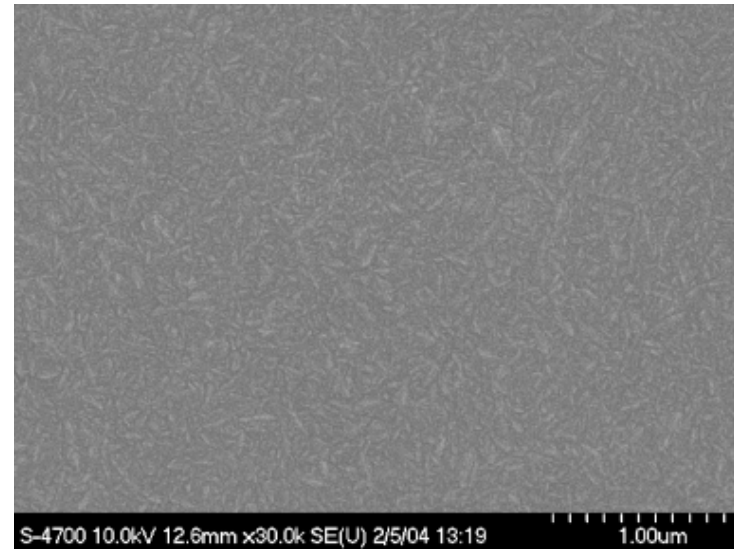
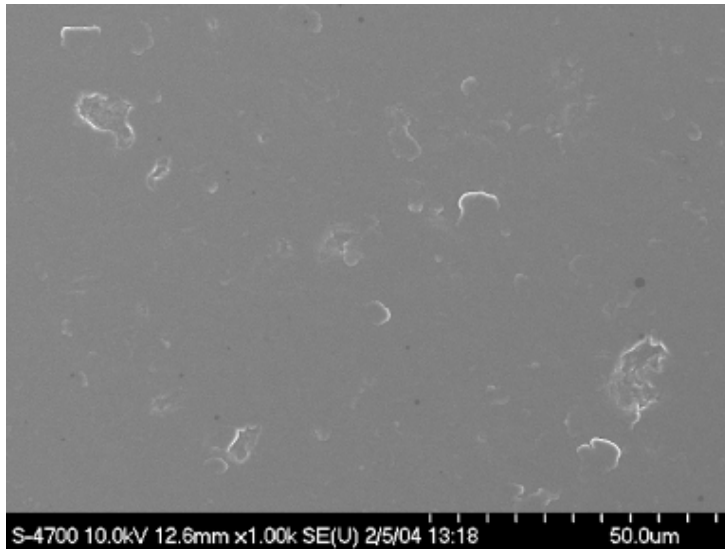
- Low nucleation density – discontinuous coating



# ***UNCD – Example of Excellent Seeding***

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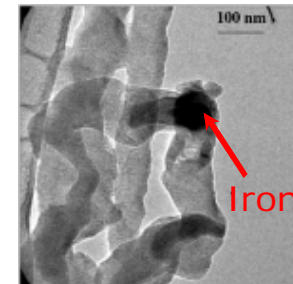
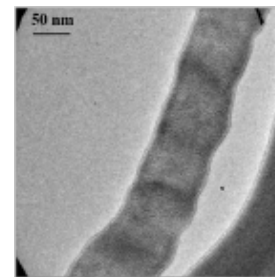
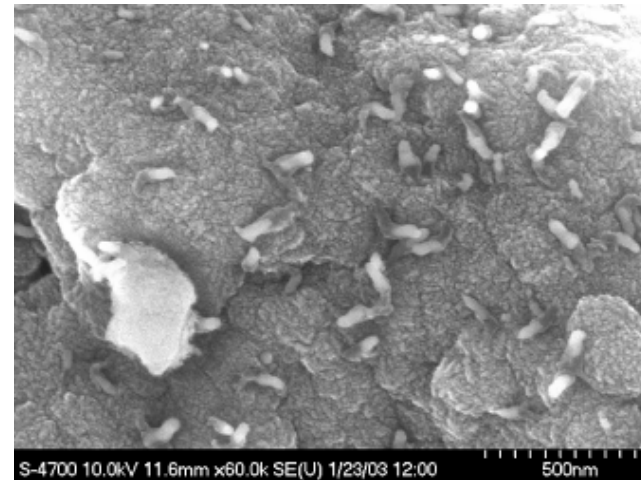
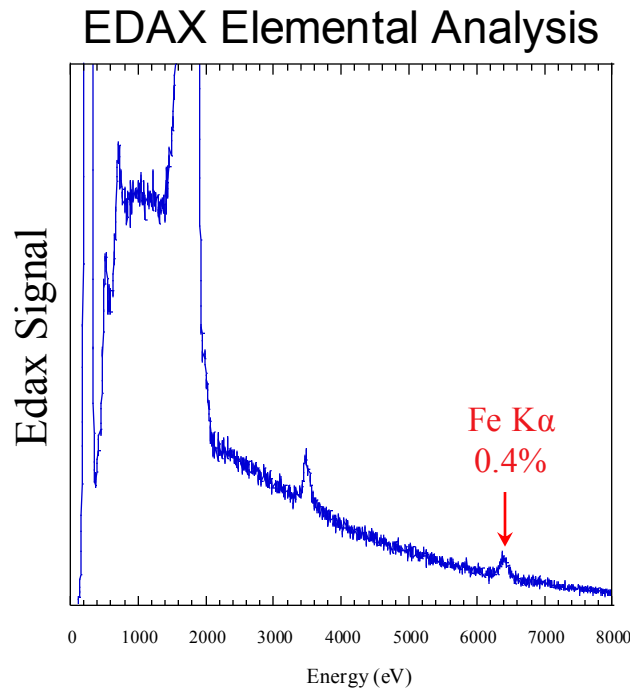
Sample: Mechanically Seeded  $\alpha$ -SiC



- High nucleation density - dense, continuous, smooth film

# Simultaneous UNCD and Carbon Nanofiber (CNF) Growth

SEM and TEM Following UNCD Treatment



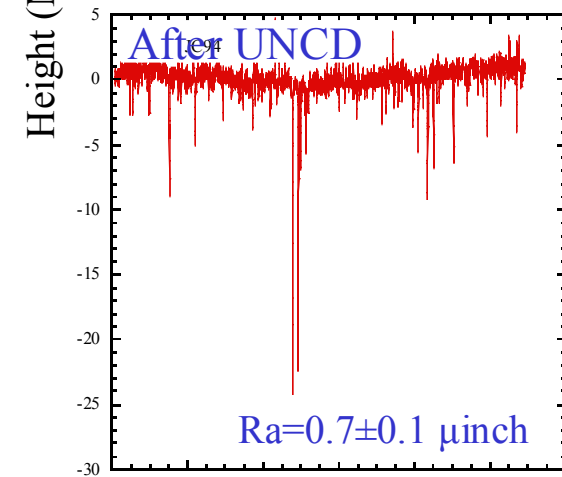
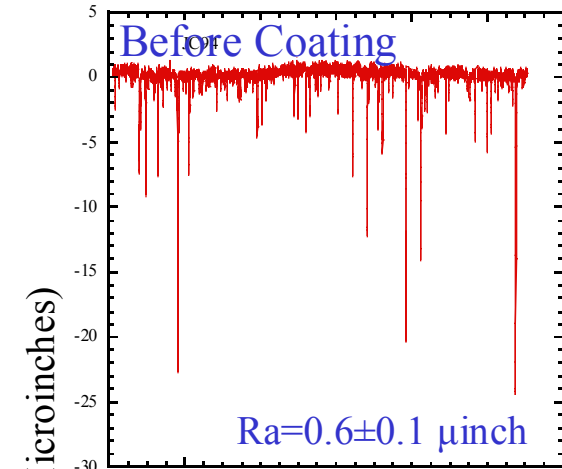
SEM, TEM shows CNF with iron particle catalyst

- Trace Fe Contamination Catalyzes CNF Growth
- Developed Screening Process for Iron

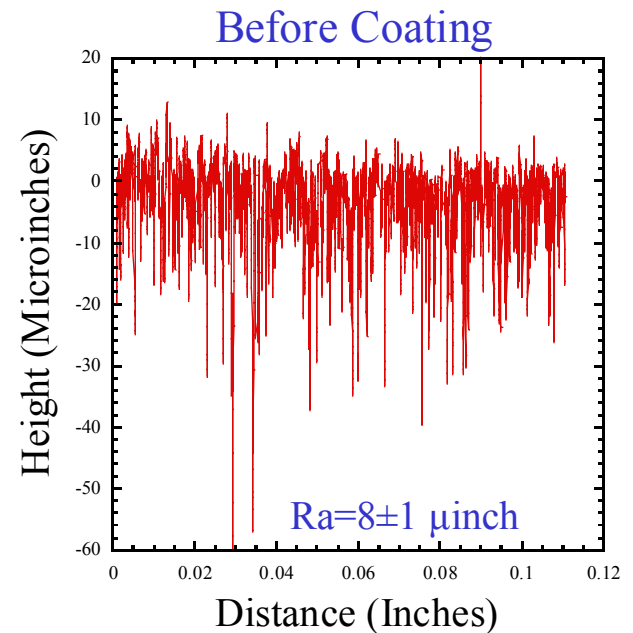
# UNCD Coating of Smooth 2" OD $\alpha$ -SiC Seals



SEM of Uncoated Seal

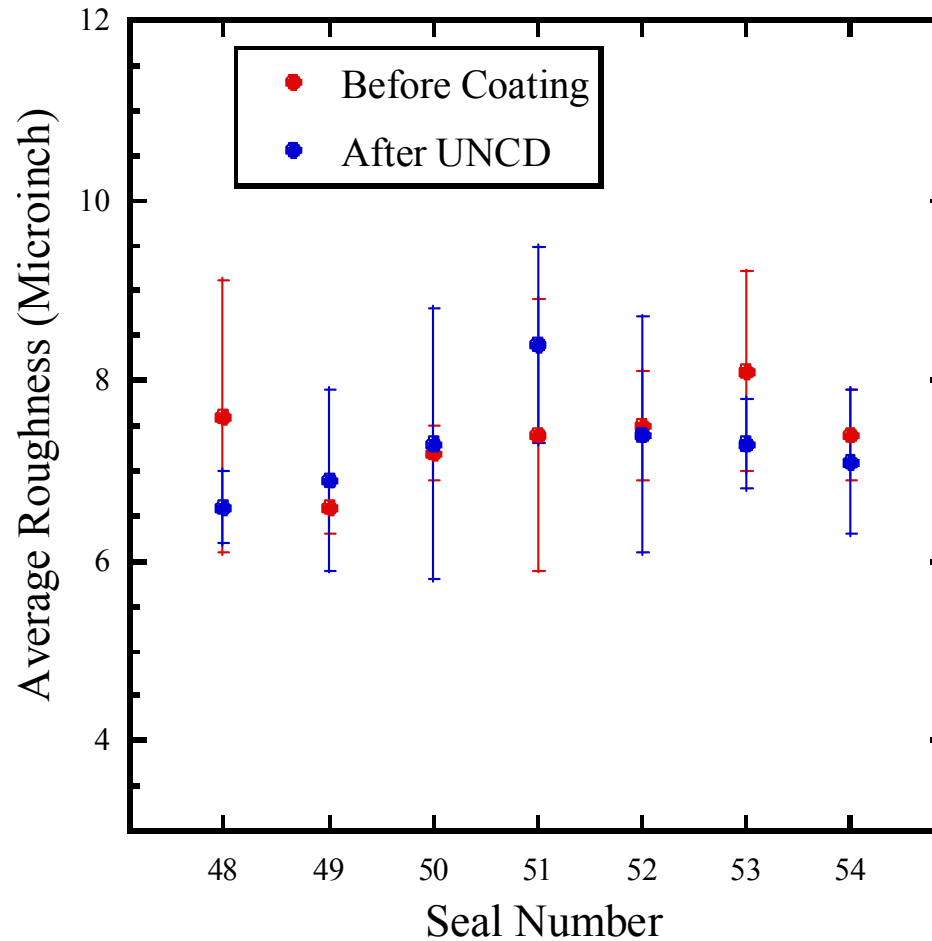


# UNCD Coating of Rough 1" OD $\alpha$ -SiC Seals



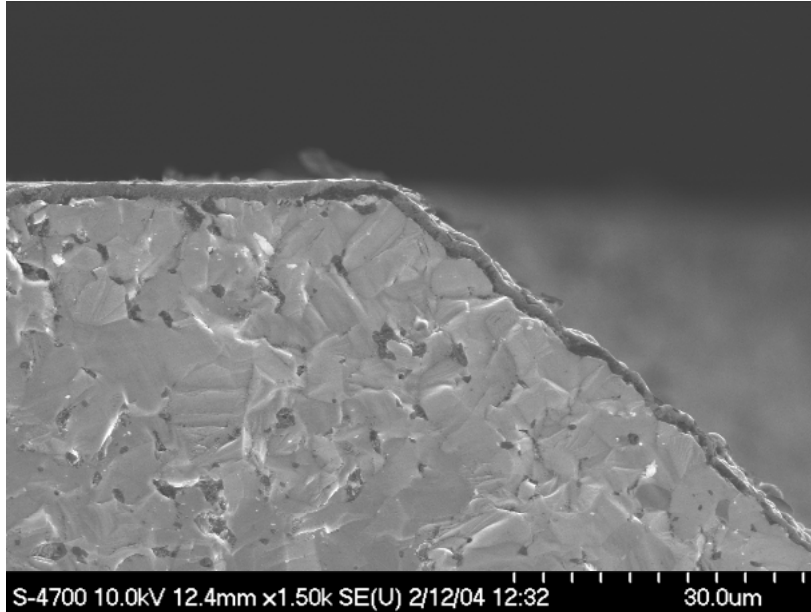
- Rough initial surface for 1" seals

# Roughness Measurements of Rough 1" OD Seals Following UNCD Coating

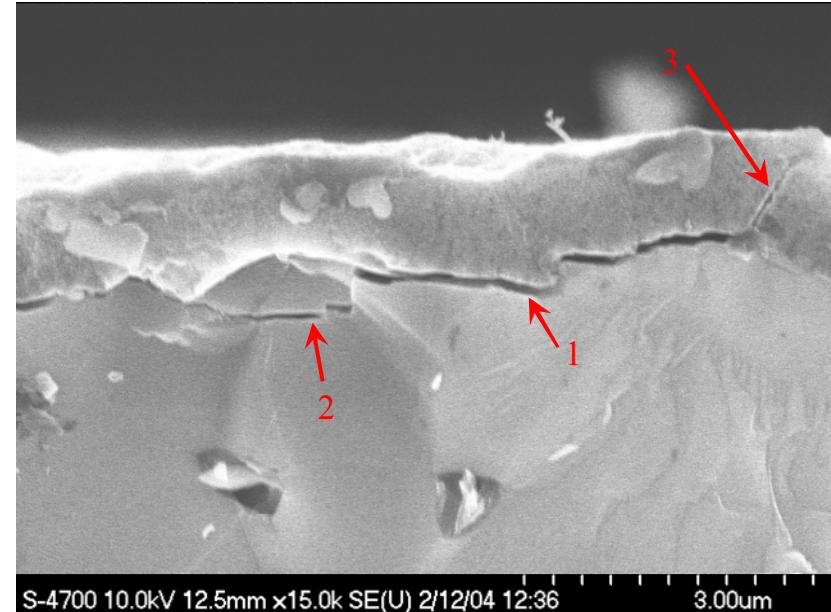


- No roughness change from UNCD Coatings

# Adhesion Measurements Using Fracture Analysis



Conformal UNCD coating over Seal face and beveled edge

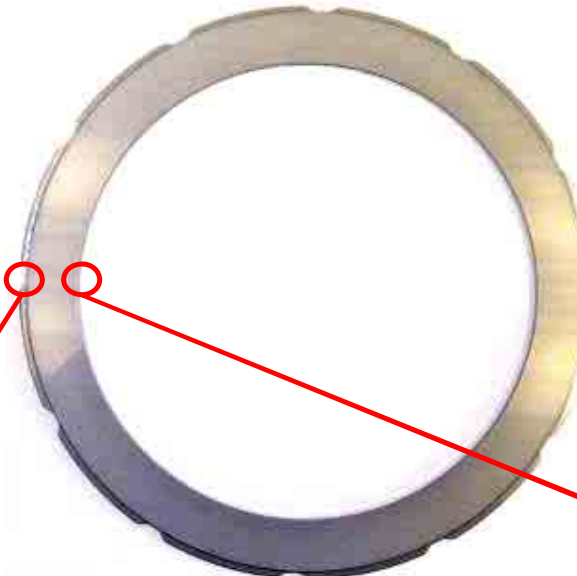


Fracture of UNCD-SiC interface observed after diamond saw cutting (1). However, fracture also observed along SiC grains (2) and UNCD film (3)

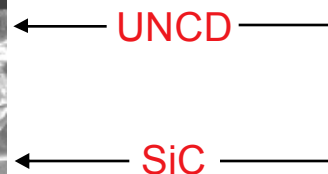
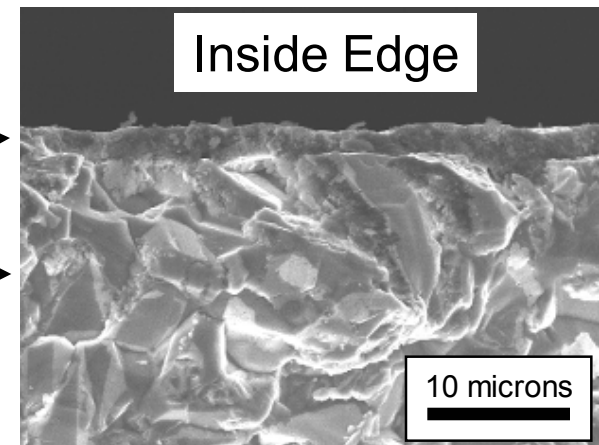
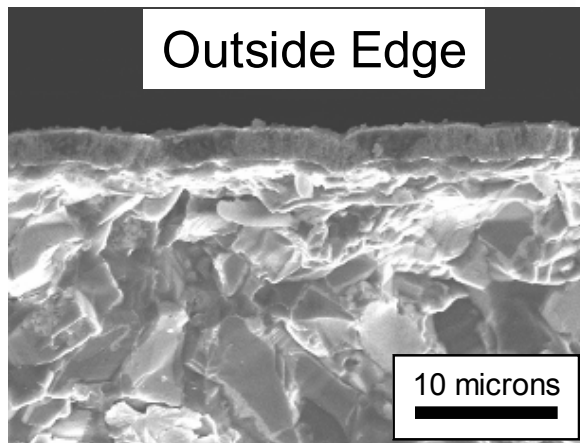
# UNCD Coating of 5" Gas Seals

- Improved coating technology required for 5" gas seals

- Coating must preserve precisely engineered taper



- Segment of 5" seal was coated in existing small-area plasma system



- UNCD Maintains Critical Tolerance Across Face of 5" Gas Seal



# Technical Barrier #2: Scale – up UNCD deposition process

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## *IPLAS 11" Microwave Plasma CVD System*



- First unit of its kind in the world
- Will enable batch-coating of multiple 2" seals
- Will enable coating of intact 5" seals



# ***Technical Barrier #3: Limited seal testing facilities***

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- **Building Test Pump Loop at Argonne for Measuring Seal Friction and Wear**
- **Installed Surface Profiler at Argonne**
- **Friction and Wear Analysis at John Crane Testing Facilities**

# ***Hot Water Test of UNCD-Coated Seals at John Crane***

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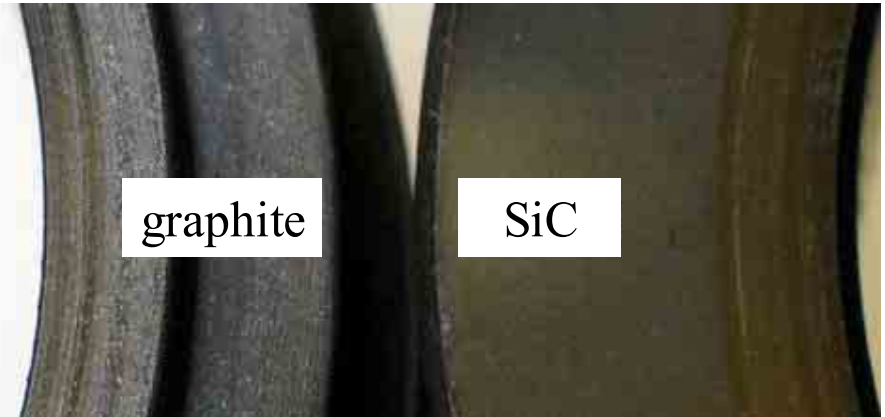
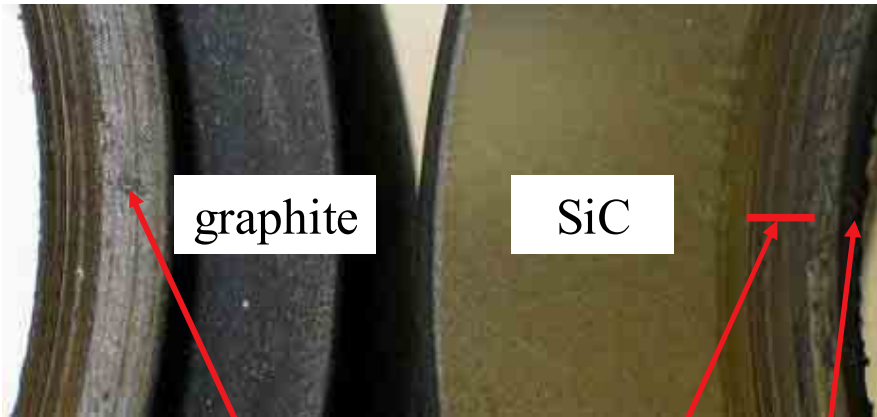
- 2" OD Seal
- 100 Hours

- Very aggressive test of materials properties
- Mimics harsh conditions in chemical process pump
- 100 hour test simulates 2 years of extreme use

# Hot Water Test of UNCD-Coated Seals

Uncoated

UNCD-Coated

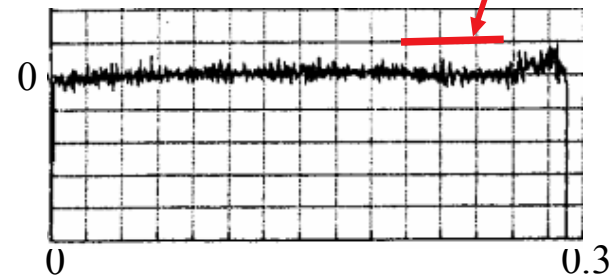
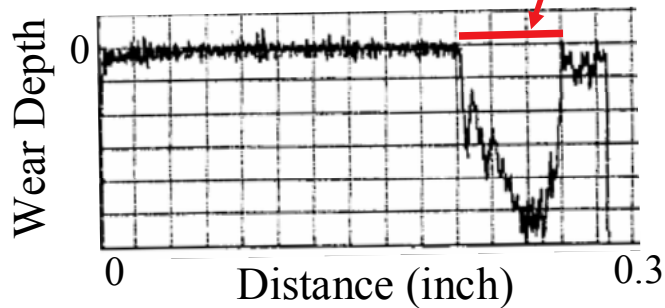


blistering

wear

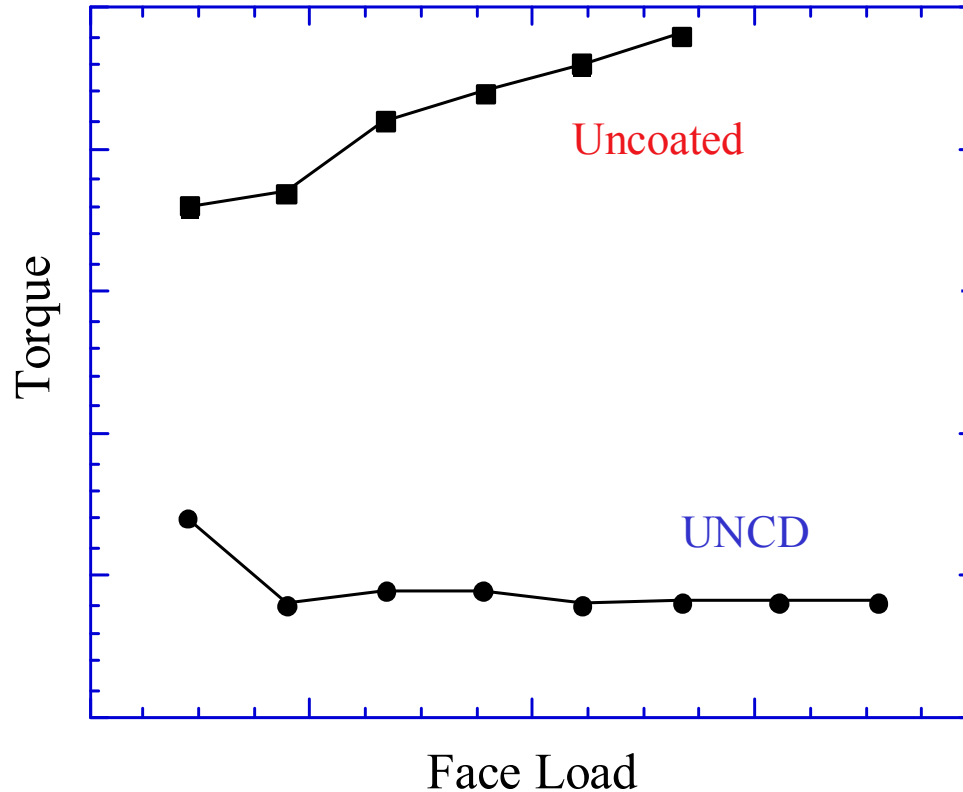
debris

No wear!



# Friction Measurements of UNCD Coated 2" SiC Seal

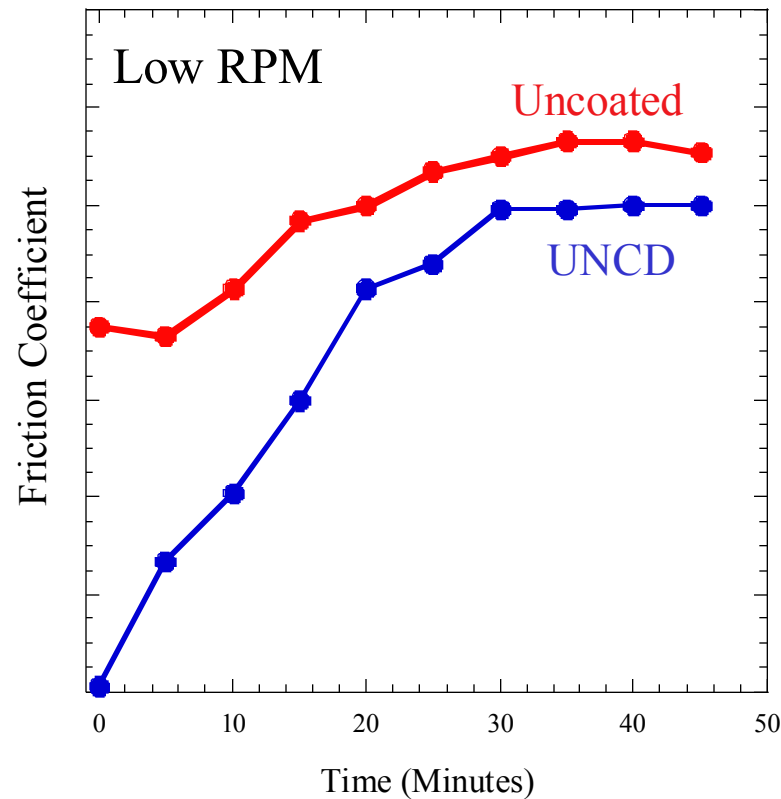
Low Initial Roughness



- Significantly Reduced Friction for Smooth Seal Surface

# Friction Measurements of UNCD Coated 1" SiC Seal

High Initial Roughness



- Marginally Reduced Friction for Rough Seal Surface

# ***Technical Barrier #4: Commercialization of UNCD technology***

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***Advanced  
Diamond  
Technologies***

- **Advanced Diamond Technologies**
  - Argonne-initiated start-up company
    - *Exclusive license in UNCD application*
  - Company officially launched in 2003
  - Business plan established
  - Toll processor for seal manufacturers
    - *Agreements in principle reached with partners*

# ***Future Work Leading to Commercialization***

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- **FY05**

- Understand effect of initial substrate roughness on UNCD friction
- Commission and optimize 11-inch IPLAS system (up from 6-inch system)
  - *Demonstrate UNCD coating of multiple 2" seals simultaneously*
- Demonstrate benefits of UNCD coatings on gas seals
- Verify tribological benefits of UNCD coated seals
  - *Perform long-term pump tests*

- **FY06-07**

- Pilot tests (field tests): 2" seals, 5" seals
- Automation of UNCD deposition process

- **FY07**

- Commercialization (sign toll-processing agreements)
- Final report