

ADMINISTRATIVE INFORMATION

1. **Project Name:** Development of Ultrananocrystalline Diamond Coatings
2. **Lead Organization:** Argonne National Laboratory
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Argonne, Illinois, 60439
3. **Principal Investigator:** John N. Hryn, Ph.D.
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4. **Project Partners:** ADT – Advanced Diamond Technologies, Inc.
www.thindiamond.com
POC: Mr. Neil Kane 217-265-9824
John Crane, Inc.
IPLAS Innovative Plasma Systems
5. **Date Project Initiated:** October 1, 2001
6. **Expected Completion Date:** September 30, 2008

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:** (Please provide 1-2 sentences describing the objective of this project.)

The objectives of this six-year R&D project, which falls under the “knowledge base or core activities R&D” in the *Industrial Materials for the Future* (IMF) program plan, are to (a) understand the fundamental processes involved in the growth of ultrananocrystalline diamond (UNCD) coatings, (b) develop a technological base for UNCD applications, and (c) demonstrate the applicability of UNCD coatings in industrial applications, such as in multipurpose mechanical pump seals.

8. **Technical Barrier(s) Being Addressed:** (Please provide a brief paragraph describing the barrier to industrial energy efficiency that is being addressed by this project and the associated technical challenges.)

Increased corrosion resistance and improved wear resistance of industrial materials could substantially improve industrial energy efficiency. Ultrananocrystalline diamond (UNCD) is diamond: the hardest, most-wear-resistant, and most corrosion-resistant material. This project will strengthen the fundamental understanding and processing methods of UNCD coatings that, when applied to industrial materials, will increase their corrosion resistance and improve their wear resistance. Sliding applications of UNCD-coated materials, such as in multipurpose mechanical pump seals, offer the greatest potential for improved energy efficiency in industry. Four technical barriers being addressed to allow wide-spread use of UNCD coatings in industrial applications are:

- Nucleation of UNCD during the coating process
- Effect of initial surface roughness of the substrate on the friction coefficient of UNCD coatings
- Adhesion of the UNCD coating to the substrate in service
- Scale-up of UNCD deposition process to allow cost-effective coating of materials

9. **Project Pathway:** (Please provide a one-paragraph summary of the approach, or pathway, being used to address the barrier(s). Emphasize the overall strategic approach for the project, not individual R&D tasks.)

The approach taken in this project is to first understand the UNCD plasma deposition process, including the seeding requirements to promote the initial nucleation needed to grow uniform UNCD coatings. This is followed by understanding and developing UNCD processing methods to insure the effects of surface roughness and adhesion are well understood. Finally, feasibility of scaling up the technology needs to be demonstrated by coating sufficient UNCD-coated seals to allow for field testing. As fundamental understanding and processing methods of UNCD coatings mature to the point at which an industry-specific or cross-cutting technology application appears viable (such as application in rotating pump seals), further development of that application will be more appropriate in the industry-specific or crosscutting technology portions of the IMF subprogram. A company, Advanced Diamond Technologies, Inc., has been formed to further develop, commercialize, and market the technology.

10. **Critical Metrics:** (Please describe the application-specific metric(s) (i.e., metrics in the end-use industrial process) that will be used to determine the success of the project.)

1-3 metrics in application/end use, including baseline, in sentence or bullet form

Success or failure of this project will be measured by our ability to meet our 3 project objectives. Specifically, we have made substantial progress in understanding how UNCD coatings nucleate and grow, and have completed the work on plasma R&D on the 6-inch IPLAS reactor, and on UNCD seeding and nucleation. We have developed a solid technological base for UNCD applications that we are now extending to the 11-inch IPLAS reactor. Scaling up the UNCD coating technology is critical to our ability to coat materials for industrial applications in a cost-effective manner. Finally, and most importantly, we need to demonstrate the energy and value-added benefits of UNCD coating in industrial applications. To this end, we need to conclude this project with a successful field test of UNCD-coated seals in multipurpose mechanical pumps. The critical metric for industry will be the ability of UNCD-coated seals to perform at an overall lower cost compared to current practice.

PROJECT PLANS AND PROGRESS

11. **Past Accomplishments:** (Please summarize the major accomplishments and key milestones achieved to date.)

Plasma R&D

- New 6-inch IPLAS microwave plasma system installed and commissioned
- Completed R&D on 6-inch unit, optimized process, demonstrated production proof-of-concept
- New 11-inch IPLAS unit (50% co-funded by DOE-OBER) was installed
- 11-inch unit was commissioned and modified to deposit acceptable UNCD coatings

Surface Seeding Study

- Completed surface seeding study, investigated CDC – found to be not beneficial when used in conjunction with UNCD
- Identified effect of Fe contaminants in SiC to UNCD growth
- Developed new seeding methodology

UNCD Materials and Processing Development

- Demonstrated growth of UNCD on a variety of seal substrate materials (alpha SiC, reaction-bonded SiC, WC, CDC – includes design and commissioning of CDC facility)

- Demonstrated UNCD growth on multiple 1-inch seals simultaneously, with required flatness and roughness, uniform thickness, and minimal waviness
- Demonstrated UNCD growth in 11-inch plasma system on multiple 3-inch seals simultaneously, with required flatness and roughness, uniform thickness, and minimal waviness

Pump Seal Application

- Solved delamination issue, adhesion of UNCD-to substrate determined
- Identified testing protocol
- Launched Advanced Diamond Technologies, Inc., to be commercialization vehicle for UNCD
- Demonstrated acceptable performance of UNCD coated seals in accelerated pump tests by industrial partner

12. **Future Plans:** (Please describe the specific dates, milestones, deliverables, and activities of all partners (i.e., who is doing what) for the life of this project, with particular emphasis on FY06. PLEASE NOTE: This information is used by DOE throughout the year for funding actions and to respond to Congressional inquiries. Failure to provide the requested information may delay funding actions.)

Date	Milestone/Deliverable	Partner Activities
9/06 6/07	Plasma R&D Optimize 11-inch IPLAS system Design and construct automated system	Argonne Argonne/ADT
9/05	UNCD Materials and Processing Development Demonstrate tribological benefit of UNCD	Argonne
3/06 12/07	Pump Seal Application Complete qualification tests for pump application Complete field tests and analyses	Crane/ADT Crane/ADT/Argonne
9/05 3/06	Other applications Demonstrate UNCD coatings on gas seals Evaluate UNCD coated gas seals (field tests)	Argonne Crane/Argonne/ADT
9/08	Final Report	Argonne/ADT/Crane

13. **Project Changes:** (Please describe changes in scope, approach, or schedule during the past year in response to any unforeseen problems/issues or successes.)

The major change that occurred over the last year is in the approach toward commercialization. Accelerated wear tests by John Crane, Inc., indicated that UNCD as a seal coating material has superior wear resistance compared to all other coating materials previously tested. As a result the project approach was changed so as to demonstrate commercial viability of the UNCD deposition process at the earliest opportunity. That milestone was reached when multiple seals were successfully coated with UNCD in the 11-inch plasma system. Although the 11-inch system is not yet optimized for commercial treatment of seals, plans for evaluating UNCD-coated pump seals in two field tests were accelerated. This accelerated approach uncovered new qualification tests that are required before UNCD-coated seals can be commercialized. Plans for these qualification tests are underway.

14. **Commercialization Potential, Plans, and Activities:** (Describe the end-use application and market potential for the project, and the plans, progress, and partners for commercial application/adoption, where appropriate; identify the product of the project and how this product will be introduced and disseminated to industry.)

A company, Advanced Diamond Technologies, was formed by Argonne National Laboratory to be the commercialization vehicle for UNCD. ADT has, over the past year, developed partnerships and entered into collaborations with leading pump seal companies who, like our project partner John Crane, Inc., will be marketing partners of ADT. Through ADT, a mechanism exists for rapid, and broad, deployment of UNCD technology to a variety of IOF (and non-IOF) industries.

In the case of pump seals, ADT will be a toll-coating provider to John Crane, Inc. It is envisioned that Crane will provide engineered seals to ADT and ADT will add the UNCD coating. With this business arrangement, ADT is free to focus on its competency which is the production of large area UNCD coatings, while leveraging the unmatched marketing and distribution capabilities of Crane worldwide. This arrangement was optimized around speed to market.

ADT has already done the financial modeling to provide justification to the assertion that the UNCD platform can be used to provide cost-effective and market-ready coated seals. Advanced Diamond Technologies was judged "Most Promising Company" at the Nanotechnology Venture Fair in La Jolla, California in September 2002. An R&D 100 award, entitled "Large-Area Deposition of Ultrananocrystalline Diamond Films" was won in 2003.

15. **Patents, Publications, Presentations:** (Please list number and reference, if applicable. If more than 10, please list only 10 most recent.)

Publications:

A. V. Zinovev, J.W. Elam, J.F. Moore, J.N. Hryn, O. Auciello, J.A. Carlisle and M.J. Pellin, "Coating of SiC surface by thin carbon films using the carbide-derived carbon process", Thin Solid Films, vol. 469-470, pp.135-141 (2004).

Presentations:

J.A. Carlisle*, O. Auciello, J.W. Elam, J.A. Libera, M. J. Pellin, and J.N. Hryn (Argonne National Laboratory, Argonne National Laboratory, Argonne, IL 60439), A.V. Sumant, R.W. Carpick (Engineering Physics Department, University of Wisconsin-Madison, Madison, WI 53706), James Birrell (Advanced Diamond Technologies, Inc., Champaign, IL 61820), Ultrananocrystalline diamond as functional coatings for mechanical pump seals, MEMS, and biodevices, [presented at the at the 107th Annual Meeting & Exposition of the American Ceramic Society, Baltimore, MD, April 10-13, 2005 \(Invited\)](#).

J. Libera, Development of Ultrananocrystalline Diamond (UNCD) Coatings, presented at Materials for Energy Efficiency in Industrial Processes and Manufacturing, 2004 ASM International Meeting, Columbus, OH, October 18-20, 2004,.