NISTIR 5837

Workshop on Characterizing Diamond Films IV

March 4 - 5, 1996
Gaithersburg, MD

Albert Feldman
National Institue of Standards and Technology

Ajay P. Malshe
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John E. Graebner
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U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards and Technology
Ceramics Division
Materials Science and Engineering Laboratory
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April 1996

U.S. DEPARTMENT OF COMMERCE
Michael Kantor, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

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Organizing Committee:

Chairman — Albert Feldman, NIST
Ajay P. Malshe, University of Arkansas
John E. Graebner, Lucent Technologies Inc.

Session Chairs

Michael Drory, Crystallume
Ajay P. Malshe, University of Arkansas
Aharon Inspektor, Kenametal Inc.
Jerry P. Zimmer, sp3 Inc.
John E. Graebner, Lucent Technologies Inc.

CONTENTS

WORKSHOP ON CHARACTERIZING DIAMOND FILMS IV

Abstract

The fourth in a series of workshops was held at NIST on March 4th and 5th, 1996 to discuss the characterization of diamond films and the need for standards in diamond technology. The audience targeted for this workshop were the producers and potential users of CVD diamond technology in the United States. Two technical topics that have relevance to applications of chemical vapor deposited (CVD) diamond were discussed: adhesion of CVD diamond to cutting tool materials and standardization of thermal conductivity measurement for heat spreading applications. A new round-robin of thermal conductivity measurements was planned which will utilize specimens having significantly better homogeneity than those used in a previous round robin.

Key Words: adhesion, chemical vapor deposition, cutting tools, CVD, diamond, round robin, standards, thermal conductivity, thermal diffusivity, tungsten carbide.

1. Introduction and Conclusions

The fourth in a series of workshops was held at NIST on March 4th and 5th 1996 to discuss, in depth, specific topics deemed important to the characterization of diamond films made by chemical vapor deposition (CVD diamond) and to address the need for standards in diamond technology. There were 28 registered attenders at the workshop.

We focussed on two technical topics for discussion: adhesion of CVD diamond to cutting tool materials and standardization of thermal conductivity measurement for heat spreading applications.

The principal conclusions of the workshop include:

- Considerable progress has been made in development of diamond coatings that adhere adequately to WC cutting tool substrates. Some expressed the view that the adhesion problem has been solved as attested to by the availability of off-the-shelf diamond coated cutting tool inserts.

- To promote the use of diamond coated cutting tools, protocols for their use will have to be developed. However, a considerable effort would be required due to the large range of machining conditions that can be encountered.

- The reasons for the relatively large laboratory-to-laboratory variations in our prior round robin on thermal conductivity measurement can be attributed to specimen inhomogeneity and specimen anisotropy. A new round robin based on specimens with significantly better homogeneity is being organized.

Appendix A contains the workshop program.

2. Adhesion of CVD Diamond to Cutting Tool Materials

This symposium consisted of eleven speakers distributed into four sessions. The abstracts are contained in the workshop program located in Appendix A.

The first session of the workshop consisted of three speakers. Evelio Sevillano of ASTeX gave a review of adhesion technology. He expressed the opinion that the adhesion problem had been solved citing as evidence the availability of carbide cutting tools coated with diamond. C. A. Klein of c.a.k. analytics described some aspects of mechanical modeling. C. H. Shen of General Motors provided considerable insight into the diamond coating issues of tooling. He expressed the view that adhesion of diamond coated cemented carbide inserts appears to be sufficient. However, our current understanding about the role of diamond microstructure on machining performance is limited. Additional effort is needed to optimize the diamond film properties in order to minimize abrasive wear.

In the second session, William Schmidt of Auburn University presented some preliminary results on measuring thickness and relating adhesion of the diamond film to x-ray measurements of stress at the diamond-carbide interface. The approach compares x-ray diffraction from an uncoated carbide substrate with diffraction from coated inserts with different thicknesses of the diamond films and as a function of position on the insert. Another technique, the ac-magnetic bridge, shows promise as a nondestructive method of measuring the thickness of a deposited diamond layer. Preliminary results show some correlation between the thicknesses measured by the magnetic bridge method and thickness measurements made with an SEM. Refinement of the magnetic bridge technique, such as reduction in the size of the magnetic probe, will enable the thickness to be measured over the surface of the insert.

Jerry Zimmer of sp3 Inc. opened his presentation with an analysis of factors affecting the deposition of diamond on cemented carbide and adhesion of the diamond film. He emphasized that both bulk and surface properties of the carbide and the diamond will affect adhesion, as well as the interface between the two. Of particular importance are stress levels, surface topography, nucleation density, carbide substrate properties, such as surface composition, bulk expansion coefficient, stiffness, surface roughness and contamination with cobalt, tungsten oxide, brittle ETA-phases and loose carbide grains. He pointed out the need for improved quantitative accelerated tests of the coated tools in a laboratory environment.

Pravin Misty of QQC Inc. presented recent advances in multiplexed laser technology for surface property modification and diamond deposition. The multiplexed laser system is based on a combination of 3 to 4 pulsed laser beams of widely different wavelengths swept over the entire surface of the object to be coated. Substrate materials used were carbides, cermets, ceramics, and high speed steels. Diamond coatings $\sim$40 μm thick were deposited on inserts at a rate of 45 seconds per insert. Finally, he discussed current and potential capabilities for the diamond coatings market, emphasizing diamond coated cemented carbide inserts and tools.

In the third session, Aharon Inspektor of Kenametal discussed his views on adhesion of diamond to WC-Co cutting tools. His presentation was directed primarily toward the substrate surface structure and the need to produce a rough, cobalt free surface to achieve good adhesion. The technique presented was based on restructuring the surface of the carbide tool by selective regrowth of carbide grains at the surface to obtain large anchor points for the diamond film. At the same time the cobalt concentration at the surface was reduced so that the potential for graphite formation between the diamond and the substrate was much lower. His conclusion was that this system completely solves the adhesion problem and that other film properties could now be optimized.

Testing for adequate adhesion of diamond to carbide inserts is always problematic and

Michael Drory of Crystallume presented data to illustrate the technique of brale indentation to measure adhesion of diamond to titanium alloy substrates. His results clearly showed the value of the technique for use on brittle films over relatively ductile substrates. In these cases consistent quantitative data was obtained. Results of indents on tungsten carbide substrates were not presented but Dr. Drory stated that correlation between indent measurements and tool machining performance was not straightforward in this case and would require more analysis and testing.

Dr. Drory's presentation and several others discussed the difficulty of measuring adhesion using contact type techniques such as indentation or scratch tests. Vinod Sarin of Boston University offered additional evidence concerning the difficulty of using contact type measurement techniques and as to why these techniques are not applicable to brittle films on brittle substrates. He then discussed a noncontact measurement technique using a compression test to measure adhesion of diamond films to silicon nitride substrates. Using a three dimensional numerical model he was able to correlate the load force to the direct characteristics of adhesion such as debond shear stress and elastic energy of delamination. This test would require specific substrate shapes but appeared to be directly applicable to WC-Co substrates.

The fourth session contained presentations from NIST research. Said Jahanmir gave a detailed discussion of a round robin set of measurements on ceramic grinding with diamond wheels. He demonstrated how statistical design of the experiment conducted prior to the measurements can allow a systematic evaluation of the data. Albert Feldman reviewed the results of an ATP funded project for developing NDE techniques to evaluate diamond film adhesion. He showed that a thermal wave image of an indent in a diamond coated WC specimen may reveal large areas of delamination not observable by SEM or optical inspection alone. Indentation testing is frequently used to evaluate diamond film adhesion.

The symposium concluded with a discussion of the need for standards and guides for users of diamond coated cutting tools, lead by A.P. Malshe. While protocols for machining with diamond coated cutting tools would be useful, the large range of machining conditions that exist would make this a formidable task.

3. Working Group on Standardizing Thermal Conductivity Measurement

A new round robin for measuring the thermal conductivity is to be held by consensus of the participants. A new round robin is to be conducted because of the relatively large variations in measured values reported in the previous round robin.[1] The specimens had large inhomogeneities. It is believed that the different ways the various measurement methods averaged over the inhomogeneities contributed to the variations observed. Discussions were held to finalize the details of the second round-robin. Many suggestions that had been faxed or e-mailed prior to the meeting were presented for discussion resulting in the present plan, which is presented in Appendix B.

4. Acknowledgments

The assistance of the Michael Drory of Crystallume, Jerry Zimmer of sp3 Inc., and Aharon Inspektor of Kenametal in preparing this report is appreciated.

5. References

[1] A. Feldman, "Round Robin Thermal Conductivity Measurements on CVD Diamond", in NIST Special Publication 885, *Applications of Diamond Films and Related Materials: Third International Conference*, A. Feldman, M. Yoshikawa, Y Tzeng, and M. Murakawa, editors (USGPO, 1995) p. 627-630.

Appendix A

Workshop Program including Abstracts

WORKSHOP on CHARACTERIZING DIAMOND FILMS IV
National Institute of Standards and Technology
Gaithersburg, MD 20899
March 4, 5, 1996

PROGRAM

Monday Morning, March 4, 1996
Administration Building, Lecture Room A

8am *Registration and Refreshments*

8:50am Opening Comments
Albert Feldman, *NIST*

ADHESION OF CVD DIAMOND TO CUTTING TOOL MATERIALS
Chair, A.P. Malshe, MRL, ME, *University of Arkansas*

SESSION I
Chair, Michael Drory, *Crystallume*

9am Status of WC Adhesion Technology Worldwide: A Review of Published Results
E. Sevillano, *Applied Science and Technology, Inc.*

9:30am Shearing and Peeling Stresses in Diamond-Coated Slabs
Claude A. Klein, *c.a.k. analytics, inc.*

10am CVD Diamond Film Tooling Evaluation and Standardization
Chi-Hung Shen, *General Motors Technical Center*

10:30pm *Refreshments*

SESSION II
Chair, Aharon Inspektor, *Kenametal, Inc.*

10:45am Diamond Adhesion and Residual Stress in Coated Tools
William F. Schmidt, *University of Arkansas*

11:15am Physical Factors Affecting Adhesion of Diamond Films to Tungsten Carbide Cutting Tools
Jerry W. Zimmer, *sp3 Inc.*

11:45am QQC's Laser-based Technology for Depositing Adherent Diamond Coating on Cutting Tools
Pravin Mistry, *QQC Inc.*

12:15pm Discussion

12:30pm *Lunch*

WORKSHOP on CHARACTERIZING DIAMOND FILMS IV

Monday Afternoon, March 4, 1996
Administration Building, Lecture Room A

ADHESION OF CVD DIAMOND TO CUTTING TOOL MATERIALS
(continued)

SESSION III
Chair, Jerry W. Zimmer, *sp3 Inc.*

1:30pm	Adhesion of Diamond Films on WC-Co Cutting Tools Aharon Inspektor, *Kenametal, Inc.*
2pm	Adhesion Testing of Diamond Coated Tools Dave Kropfl and Michael Drory, *Crystallume*
2:30pm	Adhesion Evaluation of Diamond Coatings Vinod Sarin, *Boston University*
3pm	*Refreshments*

SESSION IV
Chair, A.P. Malshe, *University of Arkansas*

3:15pm	Machining of Advanced Ceramics Said Jahanmir, *NIST*
3:45pm	NDE Techniques for Evaluating Diamond Adhesion A. Feldman, E. Drescher-Krasicka, and D. Hurly, *NIST*
4:15pm	Round table discussion of diamond adhesion and the need for standards/guide rules for users of CVD diamond Coordinator, A.P. Malshe, *University of Arkansas*
5:45pm	Discussion about future workshops Coordinator, A. Feldman, *NIST*
6:00pm	*Adjourn for dinner*
6:30pm	*Dutch Treat Dinner*

WORKSHOP on CHARACTERIZING DIAMOND FILMS IV

Tuesday Morning, March 5, 1996
Administration Building, Lecture Room A

8:30am	*Refreshments*	
9am	Working Group on Standardization of Thermal Conductivity Measurement	
		Chair, John Graebner, *AT&T Bell Laboratories*
	Requirements for a Standard Reference Material	
		Robert Gettings, *NIST*
	ASTM requirements for Interlaboratory Round Robin Measurements	
		Ronald P. Tye, *ULVAC Sinku Riko*
	Planning a new round robin	
		John Graebner, *AT&T Bell Laboratories*
10:30am	*Refreshments*	
10:45am	Discussion	
12:30pm	*Lunch*	

Adjourn Workshop

INSTRUCTIONS TO SPEAKERS

Speakers are allotted a total of 30 minutes for their presentations and for questions. Please allow at least 5 minutes for questions.

ABSTRACTS

STATUS OF WC ADHESION TECHNOLOGY WORLDWIDE; A REVIEW OF PUBLISHED RESULTS

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CVD diamond adhesion to WC substrates used in tooling applications has proven to be a major challenge to the commercialization of this technology. To date, tool performance under the best adhesion treatments is not as good as PCD, especially in milling applications. Efforts are still ongoing at many companies and universities to further improve adhesion. Many treatments have been used ranging from simple acid treatment for cobalt removal to surface roughening techniques. A review of the techniques and treatments available from the patent and published literature will be presented. An evaluation of the best treatments based on tool performance will be given.

Shearing and Peeling Stresses in Diamond-Coated Solid Slabs

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Diamond has unusual properties in terms of thermal expansion and elastic modulus. For this reason CVD-diamond coated solid slabs experience substantial thermoelastic stresses that may affect the coating's adhesion and may induce unacceptable bowing. Current investigations of diamond-film induced stresses focus on evaluating the normal stresses, *i.e.*, the stresses that act in the film layers and in the thick substrate; they do not address issues relating to the interfacial stresses that cause film delamination. It is the purpose of this contribution to provide user's friendly formulas that can throw light on the magnitude and the distribution of longitudinal and transverse interfacial stresses (shearing and peeling stresses), thus providing guidelines for designing multilayered structures that minimize the shear at the critical first interface.

Our approach is based on a model proposed by Suhir,[1] which postulates isotropic elastic properties and assumes "thin-film" conditions as in conventional stress theory but makes use of the one-dimensional "beam approximation" to derive formulas for the stress magnitude and the stress distribution that takes into account the finite size of the structure. The main advantage of Suhir's model resides in the pivotal role he assigns to the interfacial force concept, which enables one to obtain expressions for the normal as well as the interfacial stresses in a straightforward and consistent manner. Whereas normal stresses are almost uniformly distributed but at the edges, shearing and peeling stresses concentrate near the edges, where delamination will be initiated; a key result in this regard is that the shear stress at the substrate/filmstack interface reflects an algebraic sum of the contributions stemming from each layer.

The case of a diamond-coated ZnS window with a single intermediate layer clearly illustrates how these results can be taken advantage of to implement proper "buffering" action. Since the thermal expansion coefficient of the substrate is relatively large, the CVD-diamond film will be in compression; to achieve zero-shear conditions at the first interface then requires a buffer in tension whose thickness must be adjusted so as to minimize the interfacial force at the first interface, $F_0 = h_1\sigma_1 + h_2\sigma_2$, where h refers to the layer thickness and σ is the normal stress. Guidelines for identifying appropriate buffer materials thus include a positive thermal expansion mismatch with regard to the substrate.

1. E. Suhir, *ASME J. Appl. Mech.* 55, 143 (1988).

CVD Diamond Film Tooling Evaluation & Standardization

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Workshop on Characterizing Diamond Films IV
March 4-5, 1996
NIST, Gaithersburg, Maryland 20899.

Abstract

This paper will present the most recent machining test results with CVD diamond coated tools in turning, milling and drilling. The performance of these coated tools will be compared with the traditional uncoated carbide and polycrystalline (PCD) tooling. In the failure mode analysis of the coating wear and expected tool life, various plausible causes (e.g., inconsistencies in the 390 aluminum castings, the nature of the CVD diamond coatings, and the adhesion strengths to the substrates) which may contribute to the data scatter will be discussed. The need to investigate and optimize the coating thickness, diamond grain size, crystal orientation, surface morphology, cutting edge sharpness, etc. will also be illustrated and highlighted. The establishment of certain long term acceptable industrial standards among different suppliers can be realized only after such a vast wealth of knowledge and database has been developed. The availability of this basic understanding and common codes will then enable both the users and the suppliers to apply the optimal diamond coated tooling to the appropriate machining operations with high confidence and minimal efforts.

DIAMOND ADHESION AND RESIDUAL STRESS IN COATED TOOLS

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Diamond coatings on machine tool inserts are available based on several processes such as chemical vapor deposition, microwave reactors, etc. Unfortunately, there are difficulties in using these tools in a production environment, in particular the lack of reproducible machining performance. The variations in machining performance are due to many factors, adhesion, coating thickness, grain structure, etc. This paper presents a discussion of techniques that can be used to detect the thickness of the diamond coating and some approaches for assessing the adhesion of the diamond layer to the substrate. The thickness measurement, whether nondestructive or destructive, is relatively straightforward. One nondestructive technique that shows some promise is the use of an AC magnetic bridge [1,2]. This technique is easy to use and preliminary results indicate reasonable agreement when compared to destructive measurements of thickness. Adhesion is a more difficult issue. Several destructive techniques have been presented such as the scraper test [2] or using a hardness indenter to fracture the coating and then correlating the area of diamond removal to adhesion. Here, discussion will focus on a new destructive approach similar to the hardness indenter process but possessing some advantages. This approach uses the energy of a falling sphere to induce the fracture surface. The energy of the sphere at impact can then be related to the adhesion of the coating. There are no direct nondestructive techniques for finding the adhesion. Several papers have been published which utilize X-ray or Raman techniques to determine the residual stress in the diamond layer. The residual stresses are then used to infer adhesion. An approach using x-ray diffraction to determine the interfacial stresses at the bond for a cemented tungsten carbide tool insert with 6% cobalt which has been diamond coated using hot filament chemical vapor deposition will be discussed. The approach uses X-ray diffraction from the WC-Co layer for a coated insert with the residual stress in the diamond to give an estimate of the bond stress. Preliminary results will be presented, however this approach again does not actually give adhesion strength only a better estimate of the stress at the diamond - WC interface.

1. W. F. Schmidt and O. H. Zinke, "Reluctance Variation as a Result of Lift Off for an ac Magnetic Bridge," Review of Progress in Quantitative Nondestructive Evaluation, Vol. 12, pp 1885-1890, Plenum Press, N. Y., 1993.
2. O. H. Zinke and W. F. Schmidt, "Linear ac Magnetic Circuit Theory," IEEE Transactions on Magnetics, Vol 29, pp 2207-2212, 1993.
3. M. Murakawa and S. Takeuchi, "Quantitative Adhesion Strength Measurements of Diamond Coatings," Thin Solid Films, Vol. 181, pp 443-450, 1989.

Physical Factors Affecting Adhesion of Diamond Films to Tungsten Carbide Cutting Tools

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CVD diamond coated carbide tools would seem to be the ideal solution to many machining applications but many factors conspire to reduce to viability of the combination in real life applications. Both bulk and surface properties of the substrate and the diamond film must be addressed as well as the interface between the two in order to optimize the adhesion of the diamond to the carbide. These factors include stress levels, surface topography, surface composition, expansion coefficients, and substrate stiffness. Each of these has a role to play in the adhesion of the diamond film however the relative importance of each varies with the application. The performance of the system must therefore be tested under conditions which will simulate real world application problems such as substrate fatigue during milling operations. This can be accomplished with real world testing or accelerated testing in a laboratory environment. However the results of these tests tend to be more qualitative then quantitative in nature. Because of limitations in accelerated testing and the cost and time factors in real world testing there exists a need for much better measurement, modeling and characterization of the entire tool system. Quantitative measurements of stress, substrate hardness at the surface, cobalt depletion depths and other film properties as well as modeling of stresses and impact force distributions in a multilayer film system would provide a means for predicting tool performance and identifying the most critical parameters affecting adhesion.

QQC's Laser-based Technology for Depositing Adherent Diamond Coating on Cutting Tools

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Abstract

Advances in laser technology has enabled industry to incorporate lasers as manufacturing tools in production. Laser cutting, drilling, welding and sterolithography applications are widely known and accepted methods for product manufacturing. The QQC technology core program includes use of laser technology which enables adherent coatings for applications driven, material synthesis and cost effective flexible product manufacturing. The presentation will highlight some of the potential and current capabilities for surface enhancement for the Diamond Coatings market.

Adhesion of Diamond Films on WC-Co Cutting Tools

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Low pressure deposition of continuous polycrystalline diamond films has enabled the economic usage of diamond-protective-coatings on cutting tools, on machine parts, on glass lenses and in other industrial applications. Still, *the very best coating will do very little if it flakes;* Implementation of the diamond technology greatly depends on the ability to ensure good bonding between the film and the substrate. This paper will deal with basic and applied aspects of adhesion of diamond films on WC-Co cutting tools.

Good diamond-carbide bonding is a key factor in the development of diamond coated tools and the most sensitive criterion in their metal cutting applications. Without strong bonding to the substrate the coating will nor endure the shearing force in machining process and will flake; leading to unpredictable and inconsistent tool lifetimes. Adhesion and other properties of the diamond film have been shown to be greatly affected by the substrate material and by the substrate preparation prior to diamond deposition. This paper will discuss properties of the diamond - WC/Co interface, review the primary causes of poor adhesion of diamond to WC-Co and present techniques used to improve adhesion of diamond film on carbides. These will be classified as techniques designed to reduce internal stresses at the diamond-carbide interface and as substrate modifications aimed to increase the adhesion force between the coating and the base material. Of particular importance is a surface treatment of the carbide to produce a rough cobalt free surface that has enabled the deposition of firmly adherent diamond films on WC-Co inserts, by any deposition technique. The developed surface modification allows consistent tool behaviour to be achieved and predicted; step that is necessary in the commercialization of diamond coated tools.

ADHESION TESTING OF DIAMOND-COATED CUTTING TOOLS

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Abstract

Adhesion testing of diamond coated substrates has been of considerable interest in the development of useful coatings and for quality assurance testing during tool-coating production. However, there is a general lack of (mechanical) diagnostic tools which predict performance without application-specific machining tests. This is due to the extreme hardness and stiffness of diamond, the microstructure of cemented carbide substrates, and the complex failure processes which occur during machining. Recent progress has been made in quantifying the interface toughness, G_c, of diamond coated materials for relative ductile substrates. A brale indentation test is now available for measuring G_c, as a function of substrate hardness, film elastic properties and thickness, and the film residual stress. Considerable work is needed in obtaining useful information on diamond-coated brittle substrates. In addition, the relationship between film adhesion and tool performance requires additional development of mechanical modeling, as well as failure analysis (and capture) of diamond-coated tools during machining.

Adhesion Evaluation of Diamond Coatings

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A key requirement of an effective coating is its adequate adhesion to the substrate. Thus, reliable test methods to evaluate coating adhesion and to characterize the deposition parameters affecting it are necessary for the systematic development of such coatings. The conventional technique for measuring diamond coating adhesion, the scratch test, is unreliable because of wear of the stylus and influences of the substrate. Thus, a noncontact technique (compression test) of evaluating the adhesion of diamond coatings on brittle substrates was modelled and developed. This method utilizes the differences in Young's Modulus between the coating and the substrate via application of an external load in order to generate interfacial stresses and debond the coating. An innovative three-dimensional numerical model, based on combining the variational and boundary integral approaches, was utilized to link the indirect (i.e. load) to the direct (i.e. debond shear stress or elastic energy of delamination) characteristics of adhesion. Factors affecting adhesion strength of the diamond coatings are discussed in relation to process parameters. This test offers an excellent alternative to conventional techniques for measuring the adhesion strength of diamond coatings on brittle substrates.

Machining of Advanced Ceramics

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Following a comprehensive survey of U. S. industry, which confirmed that the high cost of machining is a primary impediment to the widespread use of advanced ceramics, a research program on ceramic machining was initiated by the National Institute of Standards and Technology (NIST) in July 1992. The goal of this program is to provide measurement methods, data, and mechanistic information needed by industry to develop innovative, cost-effective methods for machining of advanced ceramics. To make sure that industrial needs were properly addressed and to take advantage of the expertise existing at academic institutions, a consortium including industry, academic, and government members was established as an integral part of the program. The current members of the NIST Ceramic Machining Consortium are: Ceradyne, Inc.; Cercom, Inc.; Cincinnati Milacron, Inc.; Dow Chemical Company; Eaton Corporation; Ford Motor Company; General Electric Company; General Motors Corporation; Georgia Institute of Technology; Landis/Western Atlas Corporation; Lehigh University; St. Gobain Norton Company; Stevens Institute of Technology; Torrington Company; University of Connecticut; University of Delaware; University of Maryland; University of Massachusetts; University of Rochester; and West Advanced Ceramics, Inc. Consortium members participate in several research projects by providing materials, testing, advice, and other in-kind contributions.

A primary goal of the consortium is to produce and collect data on the effects of grinding on properties and performance of ceramics. Of particular interest is how the strength and surface integrity vary with the grinding parameters, with emphasis on grinding conditions that result in high material removal rates. These data and other evaluated data gathered from literature and consortium members are being assembled in a computerized database for use with personal computers. The *Ceramic Machinability Database* will provide access to data for different types of ceramics and will help users such as manufacturing engineers, tooling managers, and machinists develop machining plans for the cost-effective production of ceramic parts.

In one project, fundamental mechanisms involved in the material removal process during ceramic grinding are being investigated. The influence of grinding parameters such as down feed, wheel grit size, grinding fluid, and the role of ceramic microstructure and properties, for example, grain size, distribution of second phases, hardness, and fracture toughness are being explored. In addition to generating data and fundamental information on grinding, the feasibility of several non-destructive evaluation methods, such as ultrasonics and thermal wave measurement methods, for detecting and characterizing the machining damage in ceramics is being evaluated. Since generation of reliable surfaces is an important issue in applications using ceramics, in another project, the effects of grinding conditions in the "ductile regime" grinding process are being identified to establish the role of microstructure and grinding parameters in obtaining damage-free silicon nitride surfaces.

NDE Techniques for Evaluating Diamond Adhesion

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Several nondestructive techniques are being evaluated for detecting delamination and for determining whether they are able to predect premature failure of the diamond/substrate bond. The techniques under study include thermal wave imaging, scanning acoustic microscopy, and nonlinear ultrasonics

Thermal wave imaging utilizes thermal waves to probe a subsurface interface. The reflection of a thermal wave from an interface depends on the thermal mismatch across the boudary which should be affected by the bonding. The results of model calculations will be shown to that evaluate the sensitivity of the technique to debonding. Preliminary experimental thermal images in the vicinity of an indent in a diamond coated WC substrate will be shown.

Scanning acoustic microscopy has been shown to be a sensitive technique for detecting subsurface debonding and for detecting stresses in diamond coating/substrate systems. The indent studied above has also been examined. Features of the indent are to be seen including upwelling around the indent rim and stress fields extending well beyond the indent.

Nonlinear Ultrasonics. Under investigation is the applicability of nonlinear ultrasonic methods to understand relationships between microstructure and mechanical properties. Since they probe a material's third-order elastic moduli, nonlinear ultrasonic techniques may prove more sensitive to microstructural properties than standard ultrasonics. Unique instrumentation for enhanced nonlinear measurements has been developed and characterized. Using this instrumentation, experiments to measure the nonlinear properties of bulk materials are being performed. Such measurements lay the groundwork for future quantitative evaluation of surfaces and films.

Appendix B

Plan for Second CVD Diamond Thermal Conductivity Round Robin

1. Purpose: To test the precision and accuracy of various methods for measuring the thermal conductivity/diffusivity of high-conductivity samples of CVD diamond.

2. Sample types: CVD diamond, possibly single-crystal diamond, and several non-diamond unidentified standard materials of fairly high conductivity.

2.1 CVD Diamond Specifications

Number and quality: 4 long bars, each of which has a thermal conductivity that falls in the range 5-20 W $cm^{-1}K^{-1}$. We are seeking the highest possible uniformity within each sample.

Dimensions:

Length: 40.0±0.5 mm, laser cut. The 40 mm long bars will be cut into 20 mm long bars after measurements that require the 40 mm length are completed.

Width: 7.0±0.5 mm, laser cut. Sides to be parallel to within ±0.05 mm.

Thicknesses: in the range 300 to 500 μm, with thickness variations over each sample less than 2% of the sample thickness.

Finishing:

Edges: to be relatively free of graphitic residue from laser cutting. However, any etching used should not be so aggressive as to remove material from grain boundaries (i.e., no reactive ion etching, no hot $KCrO_4$ etching, etc.).

Growth surface: Remove rough growth facets by not-too-aggressive abrasion.

Substrate surface: Remove ~150 μm of CVD diamond by not-too-aggressive abrasion.

Roughness: R_A ~50 nm on *both* large faces.

Identifying mark: A mark identifying each specimen is to be laser-inscribed on the *growth* surface near a corner.

2.2 Specifications for non-diamond samples: Approximately the same size and shape as the diamond samples, with emphasis on uniformity within each sample.

3. Schedule: Because Round Robin 2 is expected to involve nearly 20 labs around the world, time is a matter of some concern. To accelerate the procedure, the samples will be divided into two groups that will be circulated independently. Each group of samples will visit all labs. Thus, at any one time, two labs will be making measurements. We will request that each lab keep a group of samples no longer than 7 days and send them on to the next lab by express mail, resulting in a total of 10 days per lab. While this may seem to be a rather aggressive schedule, we feel that with proper advance notice about when the samples are to arrive, it should be an acceptable hardship. We expect to be in close touch with the measuring labs by e-mail or fax, to help with any emergency changes in schedule, for example. A log (paper record) will accompany each set of samples to document progress. We hope to have all results completed within six months.

4. Coatings and attachments: Any thin-film coatings or attachment of thermocouples, etc., that are required for the measurement are the responsibility of the individual measuring lab. The laboratory is to both install and remove them before sending the samples on to the next lab. Avoidance of damage to the specimens is imperative.

5. Temperature of measurement: Because of the temperature dependence of the thermal conductivity of CVD diamond (and especially of the thermal diffusivity), the measurement labs will be requested to give their best estimate of the effective temperature at which the conductivity/diffusivity was being measured. How to determine the temperature is not obvious for techniques that use very local heating and distant thermometry along a rather steep temperature gradient.

6. Conversion of diffusivity to conductivity: Recent measurements of mass density ρ and specific heat per unit mass C performed at Bell Labs on CVD diamond samples from round robin one indicate that within the limits of the measurement accuracy ($\sim 1\%$ for C and $\sim 0.1\%$ for ρ) C and ρ have the same values as the corresponding quantities for bulk single-crystal diamond. Therefore, single-crystal values can be used to convert measured values of diffusivity to values of conductivity, for comparison with the results of methods that measure conductivity directly.

7. Starting date: We hope to have samples collected and ready to send out to the first measuring labs by some time in May, 1996. The exact date depends on how soon the suppliers can find time in their busy schedules to process samples that are not for immediate profit.

8. Acknowledgement: We would like to thank in advance all the suppliers and measuring labs who have volunteered their time and effort. We recognize that this is a major undertaking, involving hundreds of hours of highly skilled labor. We are confident that with close cooperation, a great deal can be learned about the reliability of thermal measurements n CVD diamond.

Appendix C

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