Superabrasives Industry Loses Another Pioneer & Icon

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The superabrasive industry has lost another great pioneer and icon. From 1967-1988 Dr. Philippe Douglas St. Pierre was the Manager of Engineering, GE Superabrasives. He was a brilliant scientist who made the development and production of superabrasive products like saw diamond, metal bond diamond, cubic boron nitride (cBN) and polycrystalline products economical to produce on a large scale in the early days of this industry. His work spearheaded the rapid improvement in all manufacturing operations and tremendous growth of all the businesses in the superabrasive industry.

**FINER POINTS** is the longest running publication devoted exclusively to the understanding, selection and application of diamond, cubic boron nitride and related materials. It is edited for recipients who are involved in some way with these “superabrasives”, either as providers of the materials, producers of products containing the materials or users of these products (e.g., grinding wheels, dressing tools, drill bits, saw blades, sawing wires, cutting tools, polishing compounds, CVD film products, etc.).
THE NEW ROARING TWENTIES, WILL THE 2020S REFLECT THE 1920S?

I have always been fascinated with history and truly believe those who ignore lessons of past mistakes are doomed to repeat them. In a similar manner, we learn to be successful when we look back on how our predecessors accomplished great deeds and technological advancements. There is a reason that most marketing and business courses dedicate a tremendous amount of time discussing the failure of the Ford Edsel and the success of the Ford Mustang. The Edsel was unattractive, overpriced and overhyped and The Ford Motor Company lost $250 million on Edsel development, manufacturing, and marketing. The very name “Edsel” became a popular symbol for a commercial failure. On the other hand, when the Ford Mustang was introduced in 1964 it was an instant success. Ford planned on selling 100,000 the first year but broke all records as 400,000 cars were sold! So goes history for success and failures in manufacturing and it can be useful and informative to turn back the clock a mere 10 decades to what life was like in America. In 1920, the U.S. had become an economic power, which is remarkable considering the “war to end all wars” had ended just two years earlier. America shifted attention from foreign affairs to economic growth … This is exactly what we are trying to do today!

The Roaring ‘20s featured women’s issues and advancements including the right to vote. In a like manner, women keep making tremendous strides as we begin the 2020’s. The Roaring 20’s also saw The Eighteenth Amendment of the United States Constitution establishing the prohibition of “intoxicating liquors” in the United States. Of course this led to the rise of popular speakeasies and criminal bootlegging success. The one thing that hasn’t changed over 100 years is human nature, the public can’t be dictated unpopular terms and conditions!

Due to The Industrial Revolution, more people moved to big cities the first time beginning in 1920. America’s total wealth more than doubled between 1920 and 1929. People across the USA wanted the same cars, clothes and music. For many Americans the 1920s brought more conflict than celebrations … sound familiar? Without getting into politics, aren’t things pretty much the same today? Opposing political and social views and religious beliefs have increased these past 100 years because of extensive and improving media devices and news coverage. Horrific and extreme news items flash across TV screens instantaneously and continuously to influence public opinion! Radio brought the nation together in the 1920’s and music created a new wave of entertainment! But this all made the youth of America excited and united by the culture and media. Pretty much the same as the 2020’s correct? In 1920, no one could have foreseen a Great Depression, or a Second World War, much less the prosperity and cultural changes that would come, or the threat of nuclear annihilation. The saying that “the more things change, the more they remain the same” has never seemed more accurate.

At this point we certainly have no idea what the 2020s hold for America and the rest of the world, but history in the 1920s has been recorded. We have seen the manufacturing world taken to new heights since mass production was popularized in the 1920s by Henry Ford’s Ford Motor Company, which introduced electric motors to the then-well-known technique of chain or sequential production. New machines and technology have been incorporated to take us into the 2020’s that even Henry Ford could never have imagined, with greater hope and exciting expectations … Over the next several years, economists tell us the economy will grow more slowly while unemployment is expected to remain low, as will inflation.

We are all looking forward to hearing William A. Strauss, Senior Economist and Economic Advisor of the Federal Reserve Bank of Chicago deliver his expert Economic Review and Update at the Industrial Diamond Associations Biennial Meeting this May!

Let’s hope the roaring 2020’s will bring all members even greater prosperity.

Best Regards,

Kevin Stiles, President
Industrial Diamond Association of America
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WHERE LAYS THE TRUTH? 
Searching for an Honest Person ...

In business, it is important we get the truth when hiring a person for a position in our company or obtaining a report internally on a particular project. We must rely on the truth and integrity of those around us. It goes without saying we need to be honest with our customers, particularly about costs and performance of our products. The same honesty in relationships with our vendors and suppliers built up over years can be destroyed by a simple exaggeration or untruth!

Diogenes (412 BC - 323 BC) was a Greek philosopher and famous cynic best known for holding a lantern to the faces of the citizens of Athens claiming he was searching for an honest man. Diogenes was a controversial figure who criticized Plato and sabotaged his lectures, sometimes distracting listeners by bringing food and eating during the discussions. We see the same lack of courtesy by many people today! However, we all must agree, searching for an honest man (or woman) has to be a dauntless task.

I am not going to risk talking in length about honesty in politics ... that is its own arena of shame. It appears most Politicians tell lies, stretch the truth and change their stories so often; I think that is a standard operating procedure!

In a recent Forbes survey, Medical professions were rated the most trustworthy and honest (Nurses topped the list!) while the lowest were Journalists, Car Salespersons and on the bottom, by an overwhelming margin, were members of Congress! Considering their impact on the entire country this is deplorable.

Since I am a Trade Journalist, I’d like to talk a little bit about honesty in the Media and I mean ALL types of Media! The news media or news industries are forms of mass media that focus on delivering news to the general public. These include print media (newspapers, news magazines), broadcast news (radio and television), and more recently the Internet (social media, live news streaming, blogs, other). Trade publications are a narrowly focused news media directed solely towards industrial and manufacturing topics. From all I have seen all these magazines and journals are very honest and truthful. That’s because what we report is based on fact and truth. There is no reason a Trade Journalist would go off the rails to make up a story about a new product, machine or manufacturing operation, so what we report can be taken at face value. This is not always true of the “fake news” and “social” media.

I recently read an article in the Washington Post where the reporter pointed out, “the media is wonderful for transmitting information, but in our hyper-connected world, information often travels faster than “facts” and “the truth.” Delve into the slippery world of mass media in the 21st century and consider the role of social media in blurring the lines between truth and lies.”

I cannot agree more ... Social media does not have a lot of restrictions and personal opinions and feelings far outweigh the search for truth. My friends and colleagues constantly send me negative and preposterous stories about celebrities, athletes and yes, politicians, trying to bolster their own views and opinions about these individuals. I have a habit of checking out these stories and most are not true at all or excerpts of a larger story or incident that intends to slant the reader’s viewpoint by eliminating certain facts!

Going forward in 2020 let’s all seek the truth in everything we do and unlike Diogenes we will not be required to hold a lantern to the faces of strangers, friends or colleagues in our search for an honest person.
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Fixed Abrasive Three-Dimensional Plate for LAPPING and POLISHING of ADVANCED MATERIALS

By: Benjamin Rosczyk, Clement Onyenemezu, Ph.D., Hyeseung Rachel Park, and Ion C. Benea, Ph.D. Engis Corporation, Wheeling, IL 60090, USA

ABSTRACT:
A fixed abrasive three-dimensional plate is provided, which comprises micron size diamond beads, ranging in size from a few microns to a few tens of microns, incorporated into a matrix consisting of one or more inorganic binders and fillers. Said composition is cast in a mold, followed by heat assisted hardening, or room temperature hardening, or hot pressed into a rigid plate blank. The fixed abrasive three-dimensional plate is then mounted on a base substrate to form a apping/polishing plate. Said fixed abrasive three-dimensional plate is capable of delivering high material removal rates coupled with reduced surface roughness when lapping and polishing of advanced materials, such as sapphire, titanium carbide reinforced alumina (AlTiC), silicon carbide (SiC), gallium nitride (GaN), aluminum nitride (AlN), zinc selenide (ZnSe), and other compound semiconductor materials.

The micron size diamond beads incorporated in the fixed abrasive three-dimensional plate, are composed of diamond particles ranging in size from a few nanometers to a few tens of microns, bonded with the aid of one or more inorganic binders and additives.

INTRODUCTION:
The most common industry practice used to polish materials such as AlTiC and other semi-conductor materials is chemical mechanical planarization (CMP) of the work piece on a pad or rigid plate. In the hard disk drive industry, the surface finishing process may also include rough lap and/or fine lap polishing. Rough lapping may use a free/fixed abrasive lapping method where some of the diamond grains are embedded into the plate to form a 2-body system while others roll between the plate and the work piece in a 3-body system. The use of both free and fixed abrasive diamond allows a higher material removal rate but at the cost of a higher surface roughness. After rough lapping, the surface roughness may be improved by adding fixed abrasive nanogrinding, using only pre-embedded fixed abrasives and lapping with a lubricant vehicle. Fixed abrasive nano-grinding gives lower lap rate but better surface finish. While the use of a free/fixed abrasive combination has resulted in improvements in surface finish of many hard materials,
there are some disadvantages of handling free diamond slurries such as charging efficiency and uniformity. It has been estimated that only about 10% of diamond particles embeds into the lap plate; the rest run off of the plate and are discarded. Most embedded (charged) diamond particles are simply pushed further into the lap plate as lapping continues, resulting in a decrease in the lap rate and necessitating plate reconditioning. Further, during lapping a small fraction of the charged diamonds may dislodge from the lap plate and scratch the work piece.

In order to improve the efficiency of lapping, fixed abrasives plates have been developed, but they have generally utilized thin films with limited life. In this study, development of a thick 3-dimensional fixed abrasive lap plate was designed to slowly wear and expose new abrasive, thereby avoiding frequent plate reconditioning while delivering the surface finish of fixed abrasive lapping.

**EXPERIMENTAL:**

**Preparation of Spherical Diamond Composites (SDC)**

Diamond powder was dispersed into metal oxide binders such as colloidal silica, ceria, zirconia, alumina, titania and mixtures thereof using a propeller mixer to produce a diamond/metal oxide slurry. The slurry was then spray dried using Yamato ADL311 spray dryer (Yamato Scientific America Inc., Santa Clara, California) equipped with a 1530 µ nozzle assembly. During spray drying, the slurry was under constant agitation and pumped into the spray chamber with a peristaltic pump with the inlet temperature of the spray dryer maintained at 170°C. The slurry was atomized as it entered the drying chamber and water stripped from each slurry droplet to form discrete particles. The discrete powder particles were separated by a cyclone into a collection jar while the steam was exhausted, while oversized particles and aggregates collected at the bottom of the drying chamber were discarded as waste. The collected particles were composed of diamond particles held together by a continuous metal oxide binder matrix. Analysis by scanning electron microscopy (SEM) showed the particles had a nearly spherical shape with distinct features of porosity and diamond grains finely dispersed within each composite particulate as shown in Figure 1. As spray dried spherical diamond composite particles exhibited a wide particle size distribution (Figure 2) and a median particle size of ~17µm when measured with a Multisizer 3 Coulter Counter (Beckman Coulter Inc.). Spherical diamond composite powders were subsequently graded to a median particle size of >20µm and used to make 3-dimensional fixed abrasive SDC lapping/polishing plates.

**Hot-pressing of 3-D Fixed Abrasive SDC Plate**

The 3-dimensional fixed abrasive plates were produced by dry mixing spherical diamond composites (SDC), phenolic binder, hollow glass spheres, and fillers. The mixture was then poured into a metallic mold and hot pressed for a specified time and temperature to fully cure the phenolic binder. After curing, the SDC abrasive plate was mounted atop an aluminum base plate and ground to

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**FIGURE 1:**
SEM micrographs of SDC particles with a (A) rounded particle shape and (B) hollow shell structure with well dispersed diamond particles.

**FIGURE 2:** Particle size distribution of as spray dried SDC particles (D50~17µm) and after sizing (D50>20µm).

**FIGURE 3:** Optical microscope picture of SDC plate at different magnifications.

Silicon carbide (SiC) is one of the fastest growing materials for semiconductor and optoelectronic applications due to its hardness, resistance to abrasion, relatively low thermal expansion, and high thermal conductivity.
flatness with a carbide grinding wheel. The new SDC plate as shown in Figure 3 was characterized by plurality of non-interconnected and irregular surface pores. During development testing, surface porosity was found to be essential for high lapping and polishing efficiency. Surface porosity was analyzed with a color thresholding of an image using a high resolution 3-D optical microscope. As represented in Figure 4, the percent surface area with pores were mapped and calculated to give the percent porosity of the plate surface at a given magnification (i.e. a measure of the pore area compared to total surface area of the plate). Surface porosity of the plates tested for this work ranged from 3% to 60% at 500X magnification.

**Lapping/Polishing Tests**

The planarized SDC plate was tested on a modified 15-inch Engis Corporation Fast Lap machine, FL-15. An 8-inch diameter SDC plate was centered atop a 15-inch diameter platen and held in place with three studs. The workpiece consisted of samples of AlTiC or SiC bonded to a 4-inch diameter ceramic block in a triangular pattern (Figure 5). The workpiece was positioned 2 inches from the center of the SDC plate and both SDC plate and workpiece rotated counterclockwise at a set RPM. Lapping of the workpiece was performed in six successive runs of 10 minutes each with a constant application of lubricant (Glycol lubricant from Engis Corporation, Wheeling, IL). After each run, the difference in thickness of each workpiece was measured with a Fowler Sylvac Ultra-Digit Mark IV drop indicator with accuracy +2µm in order to determine the average material removal rate (MRR) over 6 runs. Surface roughness was measured using Zygo NewView 6K Optical Profilometer.

**TEM Subsurface Damage Analysis**

Samples of lapped SiC were prepared and analyzed by TEM by the staff of the Nuance Center at Northwestern University. TEM samples were prepared using SEM/FIB according to Figure 6. After sample preparation, TEM images near the lapped surface of the SiC were collected to characterize subsurface damage.

**RESULTS and DISCUSSION:**

**Lapping of Titanium Carbide Reinforced Alumina (AlTiC) Bars**

Three (3) AlTiC bars were bonded to a 4-inch diameter...
AMERICAN SUPERABRASIVES CORP. (ASC) SARASOTA, FLORIDA is pleased to announce that Mr. John Chalvadakis has joined our team. John brings 15 years of superabrasives experience in operations, manufacturing, sales and marketing, as well as international trade. John’s knowledge of the industrial diamond and cBN global market will be a great addition to ASC. Prior to joining ASC, John was Worldwide Superabrasives’ (WWSA) Operation and Sales Manager for 14 years. American Superabrasives Corp. is focused on providing the finest quality assured superabrasives products and services.

HYPERION MATERIALS & TECHNOLOGIES COMPLETES ACQUISITION OF AFC HARTMETALL ... Partnership creates large independent global producer of cemented carbide tool blanks.

WORTHINGTON, Ohio – Hyperion Materials & Technologies, a global leader in developing hard and super-hard materials for a wide range of demanding applications, on Jan. 1 completed the acquisition of AFC Hartmetall, a premium cemented carbide tool blank manufacturer, marking the close of the highly complementary agreement announced in September. "Bringing AFC into the Hyperion group creates an extensive product portfolio and expands our manufacturing capabilities, boosting our already robust offering and further positioning us as the first choice for toolmakers in need of high-precision, high-performance solutions for drilling and milling applications," said Ron Voigt, CEO of Hyperion. "AFC has an extremely talented workforce and a tremendous reputation for supporting customers. Together, we will work toward the shared goal of becoming the world’s top independent supplier of cemented carbide tool blanks." AFC is highly regarded for its innovative technologies in solid, preformed and coolant channel tool blanks, the latter of which is a rapidly growing market because of the ability to offer increased cutting performance and precision. Customers will continue to buy from their current contacts at each company as they gain access to the combined product portfolio. To learn more, visit HyperionMT.com or AFCarbide.de.

CSDA ANNOUNCES NEW EXECUTIVE DIRECTOR

ST. PETERSBURG, Florida – The Concrete Sawing and Drilling Association (CSDA) is pleased to announce the appointment of Erin O’Brien as Executive Director, following the retirement of Pat O’Brien who served as Executive Director since 1992. “Erin has been intimately involved with the operations of CSDA for the past 10 years, so the opportunity to have her as the new Executive Director creates a transition that will be as seamless as can be imagined. This new chapter creates the opportunity for innovation as well as the growth of programs for our members and the industry as we enter a new decade,” according to Matthew Finnigan, CSDA’s President.

BOEING CEO STATEMENT ON US-CHINA TRADE DEAL

CHICAGO, Boeing President and CEO Dave Calhoun issued the following statement regarding the announcement of a US-China trade deal: “Boeing has a long-standing partnership with China that spans nearly 50 years. We’re proud that Boeing airplanes will continue to be a part of this valued relationship, one that has fueled aerospace innovation and sustained manufacturing jobs. Boeing applauds Presidents Trump and Xi as well as Vice Premier Liu, Secretary Mnuchin and Ambassador Lighthizer for their leadership in building a fair and mutually-beneficial trading relationship between the United States and China. Contact: Boeing Communications, media@boeing.com.
ceramic block as shown in Figure 5. Four lap plates made with SDC of diamond sizes 1.25µm, 3µm, 4.5µm and 6µm were tested under the process conditions shown in Table 1. The work piece was lapped under constant pressure of 14psi and constant plate speed of 60rpm. Results of lap rate and surface roughness of the work piece are shown in Figure 7. As expected, the material removal rate (MRR) of the AlTiC and surface roughness as measured by Ra decrease with smaller diamond size in the SDC lap plates.

SDC fixed abrasive process may increase the efficiency of the AlTiC HDD head lapping by eliminating the need for facing, grooving, shaving, and charging of diamond during plate preparation frequently encountered in the current free abrasive lapping process. With SDC plates, plate preparation only involves planarization and plate cleaning which may result in improvement of production, without the loss of surface quality. Figure 8 compares the lap rate and surface finish of free abrasive lapping to fixed abrasive lapping. SDC plate with 0.25µm diamond grains gave lap rates similar to 0.25µm free abrasive lapping with comparable surface finish. For faster removal, 1.25µm diamond SDC plate was used to give lap rate of 0.5µm/min about double that from free abrasive without compromising the surface finish as indicated by the Ra of 2.9nm.

A common issue with diamond charged lapping plates used in hard disc industries was the reduction in lap rate over time. Embedded diamonds become dull or further imbedded into the plate and become ineffective for lapping. With SDC fixed abrasive lapping plate, the lap rate remains consistent over longer times. As shown in Figure 9, lapping AlTiC bars with 4.5µm SDC plate provided consistent removal rate over time. Because SDC plates do not need to be reconditioned at the end of each run, plates last much longer than Sn-Bi plate and lowers processing cost. The breaks in the data from Figure 9 indicated when the plate was stopped and cleaned of swarf. The removal tended to increase due to plate wear and exposure of new diamond cutting points. Compared to a competitor fixed abrasive lap plates and a diamond lapping film (Figure 10), SDC plates gave superior surface finish and comparative lap rates. Competitor A is a 1µm diamond in a resin bonded fixed abrasive film used for rough lapping, while Competitor B is a 4.5micron diamond in a vitrified bonded fixed abrasive plate. Lower lap rate of Competitor A may be due to lower diamond size, but Ra as a measure of surface finish is high. The superior surface finish of the SDC plate may be due to a better distribution of diamond grains within each SDC particle; thereby avoiding diamond aggregates that cause deep scratches.

For efficient removal of the swarf and maintenance of constant cutting rate, the new 3-dimensional fixed abrasive plate was designed with plurality of pores in the plate. Pores on the surface of the plate, as shown in Figure 3 and 4, form discontinuous lapping lands and
provide channels for lapping lubricant to flow and prevent hydroplaning of the workpiece. The resultant debris/swarf is washed into the pores to avoid scratching of the workpiece. Control of the surface porosity of the plate is critical to SDC plate performance, and hollow glass spheres were added into the formulation of the 3-dimensional fixed abrasive plate to increase and control the porosity of the plate. SDC particles also had a hollow sphere structure with a non-sintered, porous silica/diamond shell. The SDC particulates are friable and easy to abrade to expose diamond. There were also pores due to entrapped air during mixing and/or curing that was controlled by the molding pressure. Excessive molding pressure squeezed out entrapped air and decreased the interstices and overall porosity of the SDC plate. Stock removal rate efficiency of the workpiece depended on total porosity of the plate as shown in Figure 11. 3-dimensional fixed abrasive SDC plates with little to no porosity did not provide a good stock removal. Figure 11 summarizes the effect of surface porosity on the AlTiC lapping removal rate. It was found that with a surface porosity of about 30% or greater, high lap rates were achieved.

**Lapping of Silicon Carbide Wafer**

Silicon carbide (SiC) is one of the fastest growing materials for semiconductor and optoelectronic applications due to its hardness, resistance to abrasion, relatively low thermal expansion, and high thermal conductivity. These good physical characteristics also make it very difficult to process. Processing single crystal SiC often involves, grinding, lapping with free abrasive slurries and chemical mechanical planarization (CMP) on a pad as illustrated in Figure 12. The lapping step often involves two or three diamond lapping steps before CMP. Often the CMP step is notoriously long and uses harsh chemical to achieve the desired surface finish. Also, due to the long CMP step, edge rounding is often a problem in processing SiC. The present work presents an alternative lapping process with fixed abrasive spherical diamond composite plates. The wafers are lapped with 3µm and 0.25µm fixed abrasive SDC plates to Ra of about 0.4nm without edge rounding problem and then cleaned up with CMP step for a short time if necessary, as illustrated in Figure 12.

In this work, planarized SDC plate was used to lap a

| **Table 2: PROCESSING PARAMETERS for SDC PLATE LAPPING of SiC** |
|-----------------|-----------------|-----------------|-----------------|-----------------|
| **PROCESS CONDITIONS** | **Plate Conditioning** | Engis Diaflex Diamond Pad |
| **Workpiece** | 4 Pieces 1” Square SiC Wafer Attached to a 4” Glass Plate |
| **Diamond Size** | 3 µm | 1.25 µm | 0.4 µm | 0.25 µm |
| **Total Process Time (min)** | 30 | 60 | 90 | 60 or 90 |
| **Plate Speed (rpm)** | 90 |
| **Pressure (psi)** | 5.2 |
| **Lube** | Hydrogen Peroxide Lube |
| **Lube Flow Rate (ml/min)** | 1.2 |
The silicon carbide workpiece shown in Figure 13 under the lapping conditions of Table 2. The workpiece consists of 4-pieces of 1 square inch 4H- N type SiC wafers bonded to a 4-inch diameter glass block. The fixed abrasive plate was mounted atop a 15-inch platen in the center and held in place in three places with studs. The workpiece was brought into contact with the fixed abrasive plate at a pressure of 5.2 psi. The SDC plate rotates counterclockwise at 90 rpm, while the workpiece which is off center from the plate by 2 inches rotates counterclockwise at about 30 rpm. Lapping of the workpiece was done in three successive runs of 10 minutes each, at a constant flow rate of 2.4 ml/min of 20 wt.% L6037 lubricant (glycol lubricant from Engis Corporation, Wheeling, IL) in deionized water. After each run, the difference in thickness of each silicon carbide workpiece was measured with a Fowler Sylvac Ultra-Digit Mark IV drop indicator with an accuracy of ± 2µm in to determine the average material removal rate (MRR). Surface roughness was measured using Zygo NewView 6K Optical Profilometer.

The results from lapping with different sizes of diamond grains in SDC plates are shown in Figure 14. Removal rate and surface finish varied proportionally with diamond size with submicron diamond SDC plates producing Ra less than 0.5nm. A surface Ra of <0.5nm may shorten the CMP step to achieve an Epi-ready surface. One major concern of diamond abrasive lapping of SiC wafers subsurface damage which may deteriorate the epitaxial layer. The CMP process is supposed to remove any subsurface damage remaining from the lapping steps, but due to very low material removal rates, the damaged layer may require a very long process time. It is preferred to minimize subsurface damage before the CMP step.

SiC wafer lapped with a 0.25µm SDC plate was analyzed by TEM to characterize any subsurface damage after lapping. During the sample preparation (Figure 6), the surface was coated with carbon/Pt to protect the surface from any damage from subsequent sample preparation steps. Fast Ion Bombardment (FIB) was used etch out a small sample from the surface. The sample was removed and attached to the TEM sample holder where it was thinned to electron transparency. TEM micrographs of the SiC wafer surface lapped with 0.25µm SDC plate showed only about 4-5nm thick damaged layer on the wafer surface (Figure 15). This thin subsurface damage layer could significantly reduce or eliminate the CMP step.

ACKNOWLEDGEMENTS:
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ILJIN Diamond
Grinding of Aerospace Materials Used for HIGH-TEMPERATURE APPLICATIONS

By: K. PHILIP VARGHESE, PhD, Group Leader, JOHN HAGAN, Sr. Application Engineer, ANDREW BIRD, Application Engineer, Saint-Gobain Abrasives

ABSTRACT
Many aerospace components used in the high and low pressure sections of a turbine engine are made from materials such as high nickel-based superalloys, intermetallics such as gamma titanium aluminide and ceramic matrix composites. These components that may be cast, forged or sintered using powder metallurgy techniques, are notorious for being difficult to machine due to their high strength, toughness, corrosion and fatigue resistance, and low thermal conductivity. Many of these parts have as much as two-thirds of their original weight removed to produce the finished component. Traditionally turning, milling and broaching processes are employed to remove most or all of the material from these components. However, with the use of modern grinding wheels composed of engineered grains and high strength bond technology, grinding can now remove the material more quickly and economically than turning or milling. This paper will show data/results of surface grinding and creep feed grinding tests conducted on advanced aerospace engine materials specifically gamma titanium aluminide, IN718 using both conventional and superabrasive grinding wheels. It will also compare the grinding of ceramic matrix composites with that of monolithic ceramics.

BACKGROUND
Nickel-based superalloys, which constitute greater than 40% of the total weight of an aircraft engine, find applications in the combustor and turbine sections of the engine [1]. Their use is primarily due to the unique combination of high temperature strength, toughness and resistance to degradation in extreme environment. These properties also make them difficult to machine, and render high-speed machining of components made from these superalloys very costly. Only abrasive wheels designed specifically for these types of materials can survive the conflicting goals of higher productivity, higher wheel life and can grind at low threshold power/forces and low specific energy.

Titanium aluminides are an intermetallic compound consisting of nearly equal parts titanium and aluminum. Their high specific strength, high-temperature stability and oxidation resistance compared to conventional titanium and nickel alloys make them attractive for use in aerospace engines. Both the new Pratt and Whitney GTF engines and CFM Leap engines make use of \(\gamma\)-TiAl blades in their low pressure turbines [2]. The heat resistance typically associated with heavy nickel alloys, alongside the lightweight characteristics of titanium, make \(\gamma\)-TiAl attractive for these engine makers who are driving increased fuel efficiency and lower emissions. Titanium aluminide components are generally very expensive to process and can be 2 to 3 times the cost of nickel-based superalloys [3].
Neargamma alloys are processed through ingot metallurgy, powder metallurgy or casting. Obtaining the final net shape in the component is usually done through some form of machining, grinding or by non-traditional methods such as electro-discharge machining, electro chemical machining or some combination of these processes. When compared with single-point machining, like with any intermetallic grinding, is often recognized as the best method for achieving final part dimensions and surface characteristics on γ-TiAl components [3].

CMCs are generally a system of materials made up of ceramic fibers or particles that lie in a ceramic matrix phase. They have many unique characteristics that lend them for use in high-temperature applications. They weigh a third of the weight of traditional aircraft engine metal alloys and can withstand temperatures of up to 2,400°F, much higher than the metal alloys. CMCs can be silicon carbide based or aluminum oxide based and their properties differ based on the degree of densification achieved after sintering and polymer or metal melt infiltration.

At the state-of-the-art Higgins Grinding Technology Center in Northboro, Massachusetts, engineers from Norton/Saint-Gobain have been studying the manufacturing of these innovative materials using grinding technology for many years. This has resulted in the development of grinding products and processes which enable more effective manufacture of components made from these materials. This article shows a sampling of data and results from grinding of high nickel-based super alloys, gamma titanium aluminide and ceramic matrix composites.

**GRINDING of HIGH-NI BASED SUPERALLOYS**

Testing consisted of creep-feed grinding of IN-718 and Waspaloy bars with two grades of TG2 wheels. Bars of IN-718 and Waspaloy measuring approximately 12.7 mm width by 76.2 mm thickness by 279 mm length were used. Instead of grinding slots into the material, the full width of the bars was ground. In this configuration coolant was not trapped by the sidewalls of the slot, but was able to escape along the sides of the part. A Campbell model 930 five Axis Grinder with straight oil coolant (Castrol Variocut B27) was used for this test. The large 30 kW. spindle is suitable for wheels up to 400mm in diameter and a 75,000 rpm high speed spindle is available for using with mounted points. A Field Instrumentation System (FIS) developed by Saint-Gobain Engineers was used to collect grinding power. Just two wheels (TG280-H20VTX2, TG280-G20VTX2) were tested and they were dressed with a BPR dress roll (BPR-2227). Truing was done at a wheel speed of 33 m/sec, at a speed ratio of 0.85. Depth per pass was 0.0254 mm and a feed of 0.508 mm per wheel revolution used for an overlap ratio of two. Figure 1 shows a picture of the test set-up used. Each wheel was tested at 2.54 mm depth per pass and at four feed rates. Under each condition approximately 17.8 mm of material was removed from the 279 mm long parts. Wheel speed was 43 m/sec and using a graphite coupon, wheel wear was determined by the step left between the used and unused portions of the wheel.

Figure 2 is a graph of the specific power for each condition tested. As expected for both materials the softer G grade wheel had a lower specific cutting energy (slope of the specific power) than the harder H grade wheel. The material type did not appear to have a significant influence on power. Although the specific power increased with increasing removal rate the specific grinding energy dropped as seen in the graph on Figure 3. As expected wheel wear increased as removal rate increased. This time separation due to the material type was more pronounced as seen in the G-Ratio graph on Figure 4. Figure 5 and 6 show the variation of surface finish at increasing removal rates in IN-718 and Waspaloy respectively. The harder H grade wheels show consistent surface finish values even at significantly high removal rates in case of both workpiece materials.

At the completion of the testing, samples of the bars ground at the highest removal rates were cut on an abrasive water jet machine and sent for metallurgical analysis. The analysis done found no occurrence of white layer on any of the samples and surface stress and damage never exceeded 0.05 mm depth. Table 1 shows a summary of the results of metallurgical analysis on the ground IN-718 and Waspaloy workpieces. The results show that both Ni-based superalloys were capable of being ground at very high removal rates without significant impact on surface finish or any white layer formation on the tested materials. Waspaloy was not much more difficult to grind than the IN-718 material from a grinding power perspective, however it did cause higher wheel wear rates particularly with...
the softer grade wheel where the G-Ratio dropped below five at a removal rate of 150 mm³/sec/mm. These conditions being representative of a roughing process any surface damage caused during this process could easily be removed with a quick finish pass.

**GRINDING of GAMMA TITANIUM ALUMINIDE**

Recent product testing on gamma titanium aluminide focused around evaluating three abrasive types: diamond, cubic boron nitride (cBN) and conventional silicon carbide (SiC). The diamond and cBN products included electroplated and dressable multi-layer metal bonded wheels, while the SiC abrasives were used with a conventional vitrified bond system. For each superabrasive type (i.e. diamond & cBN), two grit sizes were evaluated to better understand the effect of grit size on force, power, and surface roughness. A summary of the wheels is shown in Table 2. In order to evaluate the products in both mild and aggressive grinding conditions, slots were ground, as shown in Figure 7, into blocks of gamma titanium aluminide at two material removal rates. Only one set of grinding conditions was used per slot in order to preserve the ground surfaces for subsequent analysis. Testing was all in non-continuous dress mode and wheels were dressed in between slots (i.e. for each new set of grinding conditions). A summary of the grinding conditions is shown in Table 3. Products were evaluated based on power, normal force, part surface finish, and part damage (cracking and/or burn). The results of this study showed that for the conventional SiC abrasives, the power

---

**Table 1: SUMMARY of METALLURGICAL ANALYSIS of GROUND IN-718 and WASPALLOY**

<table>
<thead>
<tr>
<th>Sample</th>
<th>(Q^2) (mm³/sec/mm)</th>
<th>Maximum Depth of Longitudinal Surface Distortion (µm)</th>
<th>Maximum Depth of Transverse Surface Distortion (µm)</th>
<th>Maximum Depth of Longitudinal Strain Lines (µm)</th>
<th>Maximum Depth of Transverse Strain Lines (µm)</th>
<th>Longitudinal Imperfections (µm)</th>
<th>Transverse Imperfections (µm)</th>
<th>White Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>IN718-3</td>
<td>193.5</td>
<td>15.2</td>
<td>5.1</td>
<td>30.5</td>
<td>15.2</td>
<td>Tearing 10.2</td>
<td>Tearing 5.1</td>
<td>None</td>
</tr>
<tr>
<td>IM171-6</td>
<td>193.5</td>
<td>10.2</td>
<td>10.2</td>
<td>25.4</td>
<td>45.7</td>
<td>Tearing 10.2</td>
<td>Tearing 7.6</td>
<td>None</td>
</tr>
<tr>
<td>Waspaloy</td>
<td>161.3</td>
<td>5.1</td>
<td>5.1</td>
<td>15.2</td>
<td>20.3</td>
<td>None</td>
<td>Tearing 15.2</td>
<td>None</td>
</tr>
<tr>
<td>Waspaloy</td>
<td>161.3</td>
<td>10.2</td>
<td>5.1</td>
<td>15.2</td>
<td>25.4</td>
<td>None</td>
<td>Gouge 15.2</td>
<td>None</td>
</tr>
</tbody>
</table>

**Table 2: SUMMARY of WHEELS & CORRESPONDING ABRASIVE GRIT SIZES TESTED**

<table>
<thead>
<tr>
<th>Wheel Type</th>
<th>Abrasive Grit Size (Average Diameter)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vitrified-Bonded SiC (Hardness L)</td>
<td>60 grit (250 µm)</td>
</tr>
<tr>
<td>Electroplated (EP) cBN</td>
<td>60/80 (250 µm)</td>
</tr>
<tr>
<td>Dressable Metal-Bonded cBN</td>
<td>100/120 (151 µm)</td>
</tr>
<tr>
<td>Electroplated Diamond</td>
<td>60/80 &amp; 100/120 (250 &amp; 151 µm)</td>
</tr>
<tr>
<td>Dressable Metal-Bonded Diamond</td>
<td>100/120 (151 µm)</td>
</tr>
</tbody>
</table>

**Table 3: SUMMARY of TESTING CONDITIONS**

<table>
<thead>
<tr>
<th>MACHINE TOOL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Machine</td>
</tr>
<tr>
<td>Grinding Mode</td>
</tr>
<tr>
<td>Coolant</td>
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</table>

<table>
<thead>
<tr>
<th>DRESSING CONDITIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dress Roll</td>
</tr>
<tr>
<td>Dress Speed Ratio</td>
</tr>
<tr>
<td>Dress Overlap Ratio</td>
</tr>
<tr>
<td>Dress Depth per Pass</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>WORKPIECE MATERIAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
</tr>
<tr>
<td>Geometry</td>
</tr>
<tr>
<td>Grinding Length</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>OPERATIONAL CONDITIONS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheel Speed</td>
</tr>
<tr>
<td>Material Removal Rates (MRR)</td>
</tr>
<tr>
<td>Depth of Cut</td>
</tr>
<tr>
<td>Table Speed</td>
</tr>
</tbody>
</table>
Contract inspection and reverse engineering capacity has doubled in terms of throughput, while the size of the component that can be measured has increased more than five-fold at Laser Scanning Ltd. It follows the installation of a larger coordinate measuring machine (CMM) built by LK Metrology. The latest LK 20.12.10 ceramic-bridge CMM with 2,000 x 1,200 x 1,000 mm working volume has joined a smaller 8.7.6 model. Most data acquisition is by laser scanning on both LK CMMs as well as on two articulated arms supplied by Nikon Metrology. Visit: https://www.lkmetrology.com/

Custom engineered clad metal wire composites that can achieve specific properties for use in harsh environments, inside or outside of the human body, has been introduced by Anomet Products of Shrewsbury, Massachusetts. Anomet Composite Clad Metal Wire combines up to three metals or alloys to achieve specific properties such as corrosion-resistance, biocompatibility, or operation at temperatures from cryogenic to 1200°C. Featuring 2% or more cladding thickness to produce wire that meets design requirements and cost criteria, the clad wire is ideal for use in sensors, switches, connectors, implantable medical devices, and other applications in harsh environments. Metallurgically bonded together to provide high reliability, flexibility, and optimum formability without breaking, flaking, or blistering, Anomet Composite Clad Metal Wire is available in sizes from 0.002” to 0.125” O.D. Incorporating a gold, silver, palladium or platinum exterior layer, the core and second layer can include copper, stainless steel, Kovar®, niobium, Nitinol®, nickel-iron, molybdenum, tantalum, and titanium. For more information visit: www.anometproducts.com

A line of low absorption CO2 laser lenses that are designed to run cooler than standard lenses to provide focal length stability for consistent cuts has been introduced by Laser Research Optics of Providence, Rhode Island. Laser Research Cool-Cut® CO2 Laser Lenses feature a proprietary coating that absorbs less than 0.15% of laser energy to run cooler than standard lenses and protect against thermal damage. Direct replacements for most standard OEM laser lenses, these cool-running lenses provide focal length stability which results in consistent quality cuts. Available in 1.5” to 2” dia. sizes from 0.236” to 0.380” thick with focal lengths from 5.0” to 8.5” in 0.5” increments, Laser Research Cool-Cut® CO2 Laser Lenses have a 40-20 scratch-dig surface quality and transmission is greater than 99.0% at 10.6 micron. Longer lasting than standard A/R coated lenses, they are offered unmounted or in customer supplied mounts. For more information contact visit: www.laserresearch.net

FANUC, one of the world’s leading supplier of factory automation, robotics, ROBOMACHINES and Industrial IoT solutions has introduced the new CRX-10iA and CRX-10iA/L (long arm version) collaborative robots that set new standards in terms of ease of use, reliability and safety. FANUC offers the widest range of collaborative robots that can handle products from 4-35kg. The new 10kg payload CRX-10iA and CRX-10iA/L provide a reach of 1249mm and 1418mm respectively. Like the entire family of collaborative robots, the CRX-10iA and CRX-10iA/L are designed with FANUC’s world-renowned technology, proven reliability and sensitive contact detection that allows them to work safely alongside people in a variety of industrial and manufacturing jobs. For more information visit fanucamerica.com/CRX

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(and forces) increased as a function of the amount of stock. Similar trends were also observed for cBN wheels; however, the power increase was much lower than for SiC. The diamond wheels consistently drew the lowest power, and had the most stable power as a function of the stock removed. The same trends were observed with the coarser superabrasive wheels (250 um) and the multi-layer superabrasive products.

The increase in power (and forces) with the SiC wheels resulted in cracking and mild burn on the bottom of the ground TiAl slots (shown in Error! Reference source not found.). No cracking was observed with the cBN or the diamond wheels after removing 1800 mm$^3$ of material. In fact, the diamond wheels removed ~48000 mm$^3$ of material without damaging the parts. The cBN and SiC wheels reached only about 6 & 10 % (respectively) of that before damage was observed.

The damage to the ground parts was attributed to metal adhesion on the tips of the grains. This adhesion is shown in Error! Reference source not found., and was most prevalent in the SiC wheels. Metal adhesion was also observed on cBN grains, but to a much lower degree while very little metal adhesion was observed on the tips of the diamond grains. Adhesion of metal to the abrasive grains is known to lead to lower grinding efficiency (i.e. higher frictional contributions and a reduced ability for the grains to remove material).

While the diamond products were clearly superior in terms of cumulative stock removal without component damage, individual grinding processes may still dictate the use of SiC grinding wheels. Processes that require frequent wheel dressing in order to maintain strict form requirements could benefit from using SiC, as the cost/wheel is substantially lower and the form can be easily maintained through dressing. Electroplated diamond wheels, while capable of removing substantial amounts of material, are limited in their tolerance capability. Once used to the point where the parts lose tolerance, no redressing can be done and the wheel must be replaced. With Saint-Gobain Abrasives’s recent developments in new, multi-layer dressable metal bond diamond products, new possibilities have opened up to use these products in applications where form and finish have very strict tolerances that must be maintained over time [5].

**GRINDING of CERAMIC MATRIX COMPOSITES (CMC)**

Higgins Grinding Technology Center carried out a grinding study where a Ceramic Matrix Composite material (CMC) and a Monolithic Ceramic material (MC) were evaluated in a plunge surface grinding mode. Figure 11 shows the test setup. Polymer infiltrated CMC plates (silicon carbide-in-silicon carbide matrix) were used to compare with monolithic ceramic (silicon carbide) made by Saint-Gobain Ceramic Materials. Table 4 shows a brief comparison of the properties of the tested materials. The study involved grinding multiple slots in each material at different Specific Material Removal Rates (MRR'). Each slot was 0.89mm wide and was completed by making 20 passes at 0.254mm depth/pass, resulting in an overall slot depth of 5.08mm. Both materials were evaluated at five MRR' ranging from 5.4 to 32.4 mm$^3$/mm/sec (see Table 5). The range of MRR' was achieved by adjusting only the feedrate. Both the width of cut and the depth of cut/pass were maintained constant throughout the study at values of 0.89mm (wheel width) and at 0.254mm/pass, respectively.

The test wheel was a resin bonded, 150 grit, diamond, slicing wheel (1A1R shape). The test machine was an Elb Brilliant surface-creefeed grinder. The grinding wheel was trued and dressed prior to starting each condition. The workpieces were fixtured by waxing them onto a substrate plate. After securing the workpiece to the sub-plate, the assembly was bolted to a raider block that was held in a machinist’s vice. The wheel speed was 30m/s and the coolant was water-soluble oil. Table 6 shows the characteristics of the test wheel and some of the grinding conditions. Both materials were evaluated in terms of Specific Grinding Power, Specific Grinding Energy.
In this, the 74th year of the Industrial Diamond Association of America (IDA) we are once again having a Biennial Business Meeting in a Non-INTERTECH year. As a reminder, the IDA Board of Directors with the approval of the General Membership have opted to change from a yearly Annual Business Meeting to a Biennial Business Meeting on alternating years with INTERTECH. This year’s meeting has been planned at the Holiday Inn Ft. Myers Airport @ Town Center, Fort Myers, Florida USA May 11-13, 2020. We have negotiated an outstanding room rate of just $94.00 USD, which will be honored 3 days prior and 3 days after our scheduled meeting dates! So don’t miss this tremendous opportunity to spend some extra time with family and business colleagues!

This site was selected by our Board of Directors because of the excellent venue, amenities and exceptional room rates! Average high temperature in May is 89.4°F and average low temperature is 68.7°F, which also makes this an attractive location. The Holiday Inn Ft. Myers is two miles from Southwest Florida International Airport. The Gulf Coast Town Center is within walking distance and attendees can enjoy easy access to Ft. Myers’ attractions like shopping and dining at the upscale Gulf Coast Town Center or the nearby Miromar Outlet Mall. Historic Edison-Ford Winter Estates and other exceptional tourist attractions are all within 10 miles, while the Sanibel and Ft. Myers beaches are just a short distance from the hotel. You will also enjoy complimentary high-speed Internet access and airport shuttle service during your stay. An outdoor pool, a well-equipped fitness center, an indoor/outdoor poolside "Oasis" lounge and a full-service restaurant with room service are some deluxe amenities.

The IDA has made great strides in the past few years and it is important that we continue this momentum with the active participation of our membership. This is the only time when we can all meet as suppliers, customers, competitors and colleagues to share in marketing goals and address issues that affect us all and all the industries we serve. Here is your chance to have a say in your Associations activities and share in that feeling of accomplishment for a successful Association!

Invited speakers already confirmed will be William Strauss, Senior Economist and Economic Advisor of the Federal Reserve Bank of Chicago who will present an “Economic Overview and Forecast” and Daniel Pickard, Esq., Partner - Wiley Rein LLP an attorney who is an expert on the governments trade and tariff issues. These featured speakers will highlight an outstanding program that will also have an expanded Business Meeting covering ongoing projects and planning for events like the Superabrasives Education Course and INTERTECH 2021!

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and Grinding Ratio. The grinding power was measured using a power monitoring system developed by Saint-Gobain engineers and built into the test machine. The Specific Grinding Energy (SGE) was calculated using the power measurements and the material removal rate (SGE = Power / MRR). The wheel diameter was measured before and after each grind. Using the wheel wear and the stock removed, the Grinding Ratio was calculated (G-Ratio = Volume of Work Material Removed / Volume of Wheel Wear).

Figure 12 shows the Specific Power (W/mm) as a function of the cumulative number of grinds. The results show that the grinding power was significantly lower for the CMC material when compared to the MC material. Also the grinding power level was relatively flat throughout the test for the CMC material while the power for the MC material increased steadily as the number of cuts increased. Figure 13 shows the Specific Grinding Energy (J/mm³) as a function of the material removal rate. The results showed that the SGE for the CMC material was significantly lower when compared to the MC material. Figure 14 shows the Grinding Ratio as a function of Material Removal Rate. The results showed that the Grinding Ratio is similar for both materials at lower MRR’s. However, as the MRR’ was increased, the G-Ratio for the MC material started to decrease while the CMC material remained relatively steady. This showed that at higher Material Removal Rates, the wheel wear will increase when grinding the MC material. Whereas, regardless of the MRR’ the wheel wear remained relatively steady when grinding the CMC material. The results of this study showed that the CMC material, when compared to the MC material, required less Grinding Power, less Grinding Energy and the G-Ratio was higher (less wheel wear). Therefore, based on the results of this study, when comparing these materials, the CMC material is less difficult to grind than the MC material.

**CONCLUSION**

Materials such as CMCs, γ-TiAl, and high Ni-based superalloys used in newer aerospace engines are expanding in the aerospace sector primarily due to their unique combination of high temperature strength, toughness and resistance to degradation in extreme environment. These properties also make them difficult to machine and expensive. Higher productivity in machining is hampered by reduction in tool life and quality of machined components. However, grinding these materials with modern engineered conventional abrasive wheels and superabrasive wheels show that higher stock removal rates are achievable without part damage. Only highly engineered abrasive wheels that can remove stock at low threshold power/forces and low specific energy can achieve higher productivity as well higher wheel life compared to conventional milling / turning tools. The data presented here shows that newly developed abrasive products can meet the challenge to grind these difficult-to-machine materials economically.

<table>
<thead>
<tr>
<th>Table 4: Summary of Wheels &amp; Corresponding Abrasive Grit Sizes Tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheel Type</td>
</tr>
<tr>
<td>------------</td>
</tr>
<tr>
<td>Vitrified-Bonded SiC (Hardness L)</td>
</tr>
<tr>
<td>Electroplated (EP) cBN</td>
</tr>
<tr>
<td>Dressable Metal-Bonded cBN</td>
</tr>
<tr>
<td>Electroplated Diamond</td>
</tr>
<tr>
<td>Dressable Metal-Bonded Diamond</td>
</tr>
</tbody>
</table>

**Table 5: MATERIAL REMOVAL RATE TESTED for the TWO MATERIALS**

<table>
<thead>
<tr>
<th>Specific Material Removal Rate (mm²/mm/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.4</td>
</tr>
<tr>
<td>Monolithic Ceramic (MC)</td>
</tr>
<tr>
<td>SiC-SiC CMC (CMC)</td>
</tr>
</tbody>
</table>

**REFERENCES**


**FINER POINTS**

Featuring Superabrasive Applications & Technologies
In Memoriam – DR. PHILIPPE DOUGLAS ST. PIERRE

Dr. St. Pierre aged 94, passed away peacefully on January 1, 2020 at Kemper House Worthington, Ohio. He was predeceased by his beloved wife of 69 years, Sylvia Hill St. Pierre. From his youth, Douglas was analytical and studious and showed great promise in science and mathematics. Douglas entered the Royal School of Mines in 1943, graduated with honors in 1946, and was then employed at Murex Ltd. From 1943 to 1945 he also served in the Civil Defense Service, which provided critical assistance protecting the civilian population from German air raids, and spotting fires caused by incendiary bombs dropped on London.

Following WWII, in 1947, Douglas was very proud of receiving a Nuffield Traveling Scholarship, a prestigious award that encouraged the pursuit of new knowledge in the sciences. He was thus able to spend a year traveling in Canada and the U.S. where he studied mining and processing of gold, copper, nickel, iron, and other minerals. It was an opportunity that shaped his future.

Douglas was employed at the Canadian Bureau of Mines in 1948. In 1952 he earned a PhD degree from Imperial College at the University of London. Douglas joined the General Electric Research Lab in Niskayuna, NY, in December 1955, and among many accomplishments there, he was part of a team that developed Lucalox lamps to illuminate large spaces such as highways. He was elected a Fellow of the American Ceramic Society in 1966.

The following year he was appointed Manager of Engineering of GE’s Industrial Diamonds, a new division starting in Detroit, Michigan. He was responsible for the design of new products and processes for manufacturing diamonds. At that time the first diamond for resin and vitrified grinding was the key diamond being manufactured, so his major task was to make other products like saw diamond, metal bond diamond, cubic boron nitride (cBN) and polycrystalline products economical to produce on a large scale. In 1968, the Diamond Division was relocated to Worthington, Ohio under the name Specialty Materials Department and then it became GE Superabrasives!

Douglas retired in 1988 after 32 years with GE. For ten years, he volunteered as a newspaper reader for Ohio Radio Reading Services for the blind and dyslexic. In 1990, he taught a course at the University of Illinois on synthetic bone, and for several years traveled and gave lectures. The quintessential scientist with a brilliant mind, Douglas often engaged people, whether scientifically inclined or not, in conversations about his complex and innovative ideas. Even in his early 90s, he was developing theories that he hoped to test, applying scientific principles in materials sciences to solving problems in medicine.

Douglas was a kind and loving person, devoted to his wife and family ... he will be missed by all those who knew him.

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Contact_________________________________________________________________
Address ______________________________________________________________________________________________________________________________
City ________________________________________  State/Province _______________________  Country____________________________________________
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<thead>
<tr>
<th>Issue:</th>
<th>Editorial Feature*:</th>
<th>Closing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spring 2020</td>
<td>IDA Biennial Meeting Registration Issue</td>
<td>Mar. 1, 2020</td>
</tr>
<tr>
<td>Summer 2020</td>
<td>IMTS Preview, Machine Tools and Technology</td>
<td>June 1, 2020</td>
</tr>
<tr>
<td>Fall 2020</td>
<td>INTERTECH 2021 Early Call For Papers</td>
<td>Sept. 1, 2020</td>
</tr>
</tbody>
</table>

*Editorial topics & closings subject to change

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The Industrial Diamond Association of America, Inc. is a non-profit trade association and was incorporated on March 29, 1946 in the State of New York. It is the oldest and most prestigious association in the superabrasive/ultrahard materials industry. Activity and focus has evolved from natural diamond to superabrasives and most recently to all ultrahard materials including CVD Diamond. Members include material suppliers, tool manufacturers, component producers, machine tool builders, end users, academia/research affiliates and other companies related to the research, manufacture, application, use and sales of superabrasives.

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◆ Establishes Industry Standards
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Understanding Sources of Residual Stress in PDC by X-Ray Diffraction

By: D.K. Mukhopadhyay and K.E. Bertagnolli
US Synthetic Corporation

INTRODUCTION

Since their introduction over 30 years ago, polycrystalline diamond cutters (PDC) have made a significant impact on the oil and gas drilling industry [1]. High penetration rate and long life are some of the attributes of PDC bits. However, PDC cutters may fail prematurely during hard rock drilling. Figure 1 shows a typical PDC failure mode which may be attributed to residual stress. Conventional PDC inserts are made by sintering diamond powder on a cobalt-cemented tungsten carbide (WC-Co) substrate under high pressure-high temperature (HPHT) conditions around 6 GPa and 1400°C. During sintering, cobalt from the substrate melts and infiltrates through the pores among the diamond crystals, producing a zone of depleted cobalt in the substrate near the interface. The molten cobalt dissolves carbon atoms from the diamond crystals and quickly saturates. The dissolved carbon precipitates and forms diamond-diamond bonding among the diamond crystals [2]. As the pressure is released and the PDC cools from sintering, the diamond layer and the carbide substrate shrink at different rates, thus giving rise to residual stress in the PDC.

There are several techniques to measure residual stress in PDC [3]. Diffraction methods include X-ray diffraction (XRD), neutron diffraction, synchrotron X-ray, and Raman scattering. Diffraction techniques are generally non-destructive. Both neutron and synchrotron techniques have high penetration depth in comparison to XRD or Raman but they need special facilities. Shallow penetration depth of XRD and Raman techniques allows measurement of stress on the surface of the PDC. Additionally, stress can be measured using relaxation methods including hole drilling, ring coring, slitting, and grinding [4]. Relaxation methods are destructive and often difficult given the super hard material properties of PDC.

EXPERIMENTAL PROCEDURE

High pressure sintering was carried out using a cubic-type HPHT apparatus. Temperature was measured with a W5%Re-W26%Re thermocouple without correction for the pressure effect. Pressure was calibrated at a W5%Re-W26%Re thermocouple without correction for the pressure effect. Pressure was calibrated at room temperature using the known transitions of Bi, Tl, and Pbt6 [5]. Diamond powder (30 mm grain size) and a WC-Co substrate (13 wt.% Co, 2–4 mm WC grain size) were enclosed in a niobium capsule, surrounded by sodium chloride, placed inside a graphite resistance heater, and enclosed in a pyrophyllite cube as shown in Figure 2. The samples were first subjected to a pressure of 8 GPa. The temperature was then raised to approximately 1400 °C by passing electric current through the heater. The sample was maintained at temperature for approximately 2 minutes. Power was then turned off and the cell was cooled before pressure was released. Typical run times were on the order of 6 minutes. Once sintered, the samples were removed from the cube assembly, and the diamond table was lapped flat. The parts were then ground to a diameter of 15.9 mm and an overall height of 8.38 mm. The diamond table thickness was 1.52 mm.

A Rigaku Ultima IV powder diffractometer was used for the XRD residual stress measurements. Copper Kα beam was used. The (311) diamond peak at a two-theta value of 91.5 degree was used for the measurement. The sin2θ method was used where θ is the tilt angle. Stress measurements were made at the top center of the cutter with a spot size of 3 mm x 1 mm. The substrate was ground to different thicknesses until it was fully removed, and stress measurements were made at every thickness. Stress was also measured in a finished 16mm PDC insert. Changes in residual stress were observed as the substrate was removed by grinding. Residual stress was also measured in the remaining diamond disc after leaching out the cobalt. Comparison between the as-pressed PDC, after removing the substrate, and after removing the cobalt show the relative contribution of geometry, cobalt, and high pressure-high temperature processing to overall residual stress. An additional measurement was made of diamond powder compacted at HTHP without sintering to evaluate the influence of diamond particle deformation on residual stress.

ABSTRACT

An attempt had been made to identify the sources of residual stress in polycrystalline diamond cutters (PDC) by X-ray diffraction (XRD). Residual stress was measured in a finished 16mm PDC insert. Changes in residual stress were observed as the substrate was removed by grinding. Residual stress was also measured in the remaining diamond disc after leaching out the cobalt. Comparison between the as-pressed PDC, after removing the substrate, and after removing the cobalt show the relative contribution of geometry, cobalt, and high pressure-high temperature processing to overall residual stress. An additional measurement was made of diamond powder compacted at HTHP without sintering to evaluate the influence of diamond particle deformation on residual stress.
Since their introduction over 30 years ago, polycrystalline diamond cutters (PDC) have made a significant impact on the oil and gas drilling industry.

Figure 3 – Residual stress change in the PDC with decrease in the substrate length.

Figure 4 – Residual stress map shows presence of stress in the diamond disc after complete removal of cobalt.

Figure 5 – Residual stress map shows presence of stress in diamond particles after HTHP process in the absence of cobalt.

Figure 6 – Presence of micro twins in the diamond particle after HTHP compaction in the absence of cobalt.

The presence of cobalt in the diamond table tends to pull the diamond into compression even in the absence of the substrate. To test this hypothesis, we measured the diameter change of the diamond table before and after removal of the cobalt phase by acid leaching. The diameter increased an average of 0.0051 mm (.0042-.0060, 95%, n=18) after leaching. This indicates that the cobalt phase pulls the diamond into compression after complete leaching of cobalt (see Figure 4). In fact, the diamond disc appears to still be in compression after complete leaching of cobalt, but the magnitude of the compressive stress is less than that observed in the un-leached diamond disc.

The presence of residual stress in the diamond table after full leaching of cobalt may be due to deformation of the diamond particles by the HTHP process. To test this hypothesis, we pressed diamond powder at 8 GPa and 1400°C without a substrate or added cobalt. The diamond powder formed a hard, compacted disc, but no evidence of diamond-diamond bonding was observed. Figure 5 shows the residual stress measured in the disc of compacted diamond particles.

The disc of compacted diamond particles was then evaluated in a high resolution TEM to look for the evidence of deformation. The TEM image in Figure 6 shows the presence of [111] micro twins and dislocations in a single diamond grain. The presence of dislocations and micro twinning in plastically deformed diamond particles has been observed previously [7].

CONCLUSIONS
The XRD technique was used to identify the sources of residual stress in the diamond table.

RESULTS AND DISCUSSION
A crystalline material is defined as a solid that consists of definite periodic arrangement of atoms in three dimensions. These planes of atoms can create constructive or destructive interference of X-ray beam, and diffraction of an X-ray beam happens by constructive interference when the beam obeys Bragg’s law [6], λ = 2d Sin θ, where λ = X-ray wavelength, d = interatomic spacing, and θ = diffraction angle. If the residual stress is present in the sample, then the interatomic spacing (d) will be different than in an unstressed state. If the lattice is under under compression, the interatomic spacing (d) will be smaller, and the peak will shift to higher angles according to Bragg’s law. This change in the lattice spacing can be measured from the peak shift. Thus, the lattice spacing acts as a strain gauge in this XRD stress measurement technique. In our present experiment the (311) diamond peak at 91.50 was chosen due its relatively higher intensity and high angle position. A high-angle peak is chosen because the error in Sin θ for a given error in θ decreases as the value of θ increases [6]. The diffraction data was collected at three psi angles. The Young’s modulus (E) of the diamond table used in this calculation is 850,000 MPa. The Poisson’s ratio (v) used was 0.2. Figure 3 shows the residual stress change in the PDC with decrease in the substrate length. Residual stress of the PDC sample was measured at the center of diamond table. XRD measurements show the diamond disc is not stress free even after removal of the whole substrate.

The presence of stress in the diamond table after complete removal of substrate could be due to the interstitial cobalt. The cobalt in the diamond table tends to pull the diamond into compression even in the absence of the substrate. To test this hypothesis, we measured the diameter change of the diamond table before and after removal of the cobalt phase by acid leaching. The diameter increased an average of 0.0051 mm (.0042-.0060, 95%, n=18) after leaching. This indicates that the cobalt phase pulls the diamond into compression after complete leaching of cobalt (see Figure 4). In fact, the diamond disc appears to still be in compression after complete leaching of cobalt, but the magnitude of the compressive stress is less than that observed in the un-leached diamond disc.

The presence of residual stress in the diamond table after full leaching of cobalt may be due to deformation of the diamond particles by the HTHP process. To test this hypothesis, we pressed diamond powder at 8 GPa and 1400°C without a substrate or added cobalt. The diamond powder formed a hard, compacted disc, but no evidence of diamond-diamond bonding was observed. Figure 5 shows the residual stress measured in the disc of compacted diamond particles.

The disc of compacted diamond particles was then evaluated in a high resolution TEM to look for the evidence of deformation. The TEM image in Figure 6 shows the presence of [111] micro twins and dislocations in a single diamond grain. The presence of dislocations and micro twinning in plastically deformed diamond particles has been observed previously [7].

CONCLUSIONS
The XRD technique was used to identify the sources of residual stress in
polycrystalline diamond cutters (PDC). Measurements show that the diamond surface is in compression when attached to the full substrate. As the substrate is removed by grinding, the diamond begins to expand outward, relieving some of this compression. When the substrate is completely removed, the diamond table is not stress free but remains in a state of compression. After leaching the cobalt from the diamond table, the compressive stress is reduced but is still not zero. TEM evaluation of HTHP pressed diamond particles showed the presence of dislocations and micro twins. We speculate this deformation is partially responsible for the remaining compressive stress.

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