2009-11-25

An Improved Cube Cell Assembly for the Use With High Pressure/ High Temperature Cubic Apparatus in Manufacturing Polycrystalline Diamond Compact Inserts

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An Improved Cube Cell Assembly for Use With High-Pressure/High-Temperature Cubic Apparatus in Manufacturing Polycrystalline Diamond Compact Inserts

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A thesis submitted to the faculty of
Brigham Young University
in partial fulfillment
of the requirements for the degree of

Master of Science

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December 2009

ABSTRACT

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The goal for this research was to reduce the current manufacturing cost of the polycrystalline diamond compact (PDC) inserts utilized in the natural gas and oil drilling industry while not reducing their current performance. Polycrystalline Diamond is added to the tungsten-carbide (WC) substrates commonly utilized in these applications because of its greater wear and thermal resistance.

With the current cube cell design for the high-pressure/high-temperature apparatus, it is necessary to bond an extra WC substrate to the polycrystalline diamond insert to achieve the sizes generally ordered by the customers.

The problem of bonding the extra WC substrate was solved by increasing the operating volume of the cube cell assembly and changing the heating pattern within the cell while maintaining the temperature and the pressure required for the successful diamond sintering.

The new cell design was proposed and tested. The test data were captured and analyzed to prove the hypotheses. The proposed manufacturing methods resulted in reduced cost, processing time, and reduced the need for equipment and operators without diminishing the performance of the PDC insert.

Keywords: polycrystalline diamond compact, PDC, high-pressure/high temperature apparatus, cubic press, cube cell assembly, diamond inserts, diamond cutters.

ACKNOWLEDGMENTS

I would like to show my appreciation to those who supported and helped me through this journey on becoming a professional.

I express deep gratitude to my wife Veronica, my son Malcolm, and my daughter Miah for their support, patience, and understanding during my years of education. They were my primary motivation to continue improving. I would also like to acknowledge my mother and my grandparents who taught me that perseverance leads you to success.

I am grateful for the support I had from Dr. Mike Miles, who helped and guided me through the graduate program; and extend this appreciation to Dr. Perry Carter who dedicated many hours to make this thesis possible, and to Dr. Charles Harrell for his support and contribution through my graduate education.

I feel the need to specially recognize the people at US Synthetic Corp. who encouraged, funded and directed me to the right path and helped make this thesis possible.

Among those who helped are Dr. Ken Bertagnolli, Dr. Deb Mukhopadhyay, Bart Crockett and Paul Jones who supported the idea, Bryce Jensen and Ryan Bird who worked very hard to get the design, the components, and the experiments on time; the testing department, and a special appreciation to Eugene McMurray for his insights.

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1.1 Introduction to Diamond Making

Since their early discovery in ancient India, diamonds have always fascinated humankind, mainly because of their unique characteristics. Diamond is the hardest known naturally occurring mineral. Once polished, it is crystalline clear with extraordinary light dispersion properties.

Historically, diamond was primarily a symbol of power, utilized as gemstones to decorate jewelry. It is a rare mineral generated by Mother Nature under the high-pressure and high-temperature conditions, which occur deep within the earth. Diamonds are rare at the Earth's surface because they convert back to graphite while moving to the surface via geological processes.

Scientists have always been intrigued with this material and its properties. In 1704, Sir Isaac Newton was the first well-known scientist to do research on diamonds, describing them as a coagulated material composed of carbon, oil, and amber (Newton, 1721). But it was not until the late seventeen hundreds that Antoine Lavoisier (Lavoisier, 1799), and shortly after Smithson Tennant (Tennant, 1797), were able to discover by experimentation that diamond and graphite were allotropic forms of carbon. This discovery was what ignited the research race in the scientific world to develop a process to transform worldwide abundant graphite into the scarce gemstone.

There were many apparently successful but unrepeatable experiments which, in theory, were capable of creating diamonds. But it was not until December of 1954 when it became possible to synthesize diamond from graphite. General Electric, utilizing the “Belt Press”, a design of Dr. H. Tracy Hall, officially declared that they were able, in a repeatable manner, to change the structure of the graphite to diamond by applying constant high pressure and high temperature (HP/HT).

This revolutionary announcement generated a new technology race. A great amount of information was published by Dr. Hall while he was working as a research director on the Synthetic Diamond project at Brigham Young University (BYU). Many scientists started developing new equipment and researching new materials to improve the diamond synthesis process.

Dr. Hall, while working at BYU, developed two more high-pressure/ high-temperature devices; the Tetrahedral and the Cubic presses (Hall, 1961). The latter is the one that pertains to our current research.

Once again, because of its unique qualities, diamond has a great impact on the industrial business. Its strength, thermal stability, electrical insulating properties, and capacity to conduct heat make diamond a great abrasion-resistant product, utilized to machine, grind, and polish extremely hard materials. It is also utilized as a heat sink in many high-technology applications.

Initially introduced by General Electric in the early 1970’s, the manufacturing of super-abrasive machining inserts was a key component in the development of super hard metal alloys and ceramics. The industrial market motivated many entrepreneurs in the development of diamond manufacturing corporations where the massive manufacturing of diamond grit and polycrystalline diamond inserts for machining or drilling applications is their core competence.

In the beginning, manufacturing of Polycrystalline Diamond Compact (PDC) inserts was very difficult and expensive. There were just a few manufacturers, and they were only capable of supplying a very limited quantity at a high price to their customers.

Polycrystalline Diamond Compact was first offered to the copper wire industry in 1974. It revolutionized the wire industry by providing longer life to the dies, which improved productivity, consistency and finish quality. The PDC had the same impact on the machining of nonferrous materials because of its capability to remove more material in the milling, boring, and turning operations while providing sustainable, high-quality surface finishes. However, its highest impact was in the oil and gas drilling industry because its toughness and thermal stability provide a faster rate of penetration and a longer life to the drill bit (Miess, 1996). Because of these qualities it represents an important factor in the oil economy, reducing the necessity of bit exchange which represents long down times and expenses (Harper, 2001).

The super-abrasive components, mainly Polycrystalline Diamond Compact (PDC) and Polycrystalline Cubic Boron Nitride (PCBN), are considered part of the abrasive industry. In the US, the abrasive industry has combined annual revenue of $4 billion (Harper, 2001). The PDC industry has estimated annual revenue of $500 million, and the cutting tool and wire dies have another $500 million of estimated annual revenue.

China currently has 50% of the world's production capacity of abrasive products and the lowest manufacturing cost in the industry. With the help of the new technologies and lower manufacturing costs, Chinese grit manufacturers can offer their lapping grade diamond grit for less than five cents a carat and their PDC inserts at about 20 U.S. dollars each. This creates a highly competitive environment where it is imperative for the United States manufacturers to develop new process technologies to reduce cost and improve product manufacturability and at

the same time maintain their product performance in order to preserve a competitive advantage in the market.

1.2.1 The Conventional Diamond Making Process

The process of manufacturing polycrystalline diamond cutters is accomplished by sintering diamond powder on a cemented carbide substrate with cobalt as a binder by applying high pressure and high temperature to the components (Filed, 1992). The combination of the high pressure and the heat allows the catalytic material (usually cobalt) to flow from the substrate to the diamond enabling the diamond-to-diamond sintering process and the substrate to diamond bonding (Bertagnolli, 2000).

In Figure 1-1 we can observe a Polycrystalline Diamond Compact insert (a) where the sintered diamond layer (b) is bonded to the tungsten carbide substrate (c). We can also observe in Figure 1-1 (b) the synthesized diamond crystals denoted as the darker areas. The brighter areas are remaining cobalt. In Figure 1-1 (c) the cobalt binder is indicated in the darker areas between the tungsten carbide crystals.

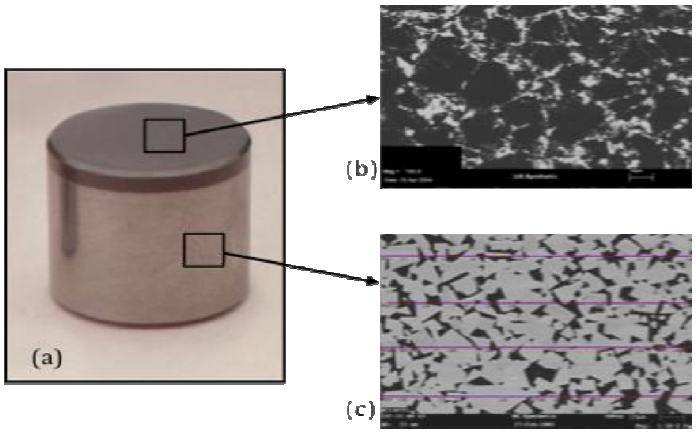

Figure 1-1: (a) Polycrystalline diamond compact insert, showing (b) the sintered diamond layer bonded to (c) the carbide tungsten substrate and their pertinent microstructure pictures (Haddock)

A high-pressure/high-temperature apparatus is utilized to exert the pressure and conduct an electric current to a cube-cell assembly. The cube-cell assembly is the combination of many components responsible for transferring the pressure, heating, and containing the samples to be sintered. The cell assembly components will be described more extensively through this chapter.

Since the early days of the manufacturing of Polycrystalline Diamond Compact or Polycrystalline Cubic Boron Nitride inserts in cubic presses, the size of the cube cell assembly has been limited by the size of the HPHT apparatus chamber. The chamber, as shown in Figure 1-2, is composed of six anvils with a face surface area of 1.5” high by 1.5” wide, limiting the size of the cube assembly to 2” wide by 2” tall. The difference in size between the anvil faces and the cube cell assembly faces is a ratio that has been optimized in order to provide the material necessary to create the gasket between the anvils. This gasket is a key component to support the press anvils, distribute pressure, and confine the sample being pressed (Hall, 1961).

After being pressed, the cube assembly dimensions remain slightly larger than the anvil face dimensions. This anvil-to-cube ratio has been utilized and optimized by US Synthetic through the last 30 years. US Synthetic is a polycrystalline diamond insert manufacturer founded by Dr. Bill Pope and Louis Pope in 1978. It is located in Orem, Utah. US Synthetic is one of the current leaders in the manufacture of polycrystalline diamond cutters, which are utilized by most of the oil and gas bit manufacturers.

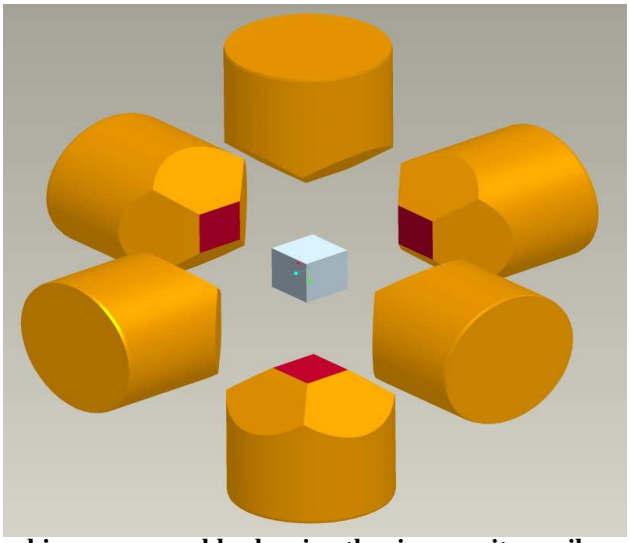

Figure 1-2: Schematic of cubic press assembly showing the six opposite anvils and the cube assembly in the middle.

The Polycrystalline Diamond Compact insert manufacturing procedure has three main processes: Assembly, Press, and Finishing.

1) Assembly: The assembly process is divided into three main operations:

    a) Can Assembly: The objective of this operation is to combine the components of the insert together and protect them from possible contaminants of the process environment. The insert is composed of diamond powder and a cemented tungsten carbide substrate. These components are contained by a set of cans, therefore its process name.

    b) Heater assembly: In this operation the can assembly is placed inside a liner and separated with a disc in the middle and each end made out of isostatic material to ensure a uniform pressure distribution through the sample and insulate the samples from grounding. Because of its properties, salt is the most common material utilized for these components. Once in the liners, the sample is placed inside the heater

tube and the graphite discs are placed at each end of the assembly. These last two components are responsible for the resistance-heat generation necessary for the sintering process.

c) Cube Assembly: Once the heater assembly is completed, it is placed in a pressure media cube that has been bored to an oversized diameter to accept an insulating liner between the heater assembly and the pressure media cube. A refractory metal disc is placed at each end of the heater assembly and a steel ring at the outermost end to conduct the current, necessary for the resistance heating, from the anvils to the heater. A pressure media button is placed inside of each steel ring to support the steel rings from deformation, distribute pressure to the sample, and insulate the anvils from the assembly heat.

The limited size of the cube assembly restricts the volume capacity in the heating element, also referred to as “furnace” or heater. The heater, in most cases, is made out of machined graphite, and its height or diameter depends on the area available in the cube assembly. The furnace is the only source of heat utilized for the diamond synthesis process. The reason why the heater assembly limits the size of the sample is mainly because of the current path design which is necessary to generate heat.

As described previously in the cube assembly process, the cell design has a steel ring at each end of the cube assembly, which allows the current to get introduced into the cell from the press anvils. Then the current flows from the steel ring to the heater assembly by a titanium or molybdenum disc. In all the cell assemblies for the cubic press researched a graphite disc is

placed at the end of the heater tube, with the purpose of generating end heating which is believed to be necessary for the synthesis process. Figure 1-3 illustrates the conventional arrangement of components which, when pressed in the high-pressure/high-temperature apparatus results in two polycrystalline diamond compact inserts represented as the two samples in the middle of the cell with the sintered diamond layers facing outwards.

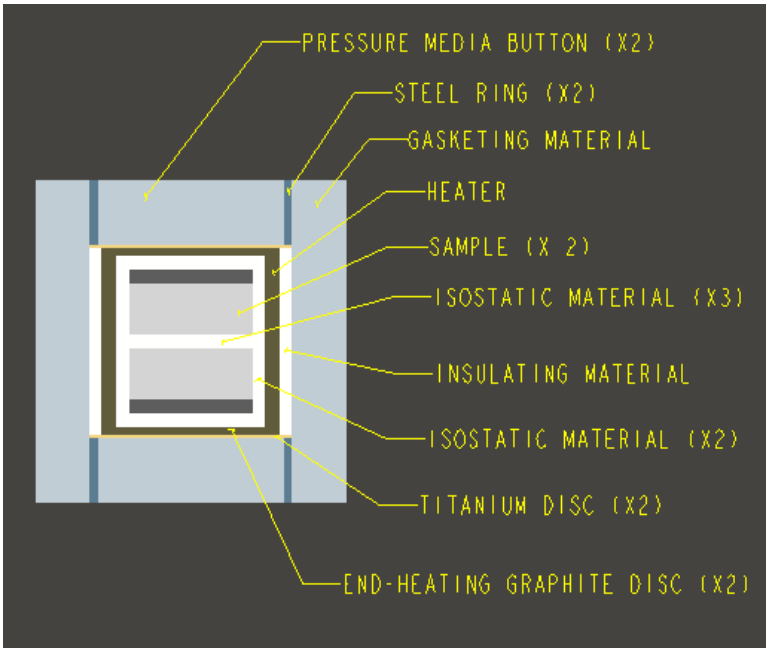

Figure 1-3: Original cube cell assembly design for the cubic HT/HP press

2) Press: The cube assembly is placed in an evenly aligned position on the bottom anvil of the cubic high-pressure/high-temperature apparatus. Then the operator starts the press, causing the six cylinder rams to advance the anvils simultaneously to guarantee a proper alignment of the press components and the cube assembly. This will prolong the life of the press and provide a consistent pressure to the cell. Once the pressure in the cell is at approximately 66 kbar, the current is turned on, and it is allowed to flow

from the top and bottom anvils to the cell assembly. Once the temperature in the cell is in the region of 1400° C, the current is stabilized and the samples soak for a predetermined period of time to ensure a uniform heat distribution through the sample. Then the current gets gradually turned off, and when the temperature is low enough so the diamond will not convert back to graphite, the pressure is released and the sample assembly is pulled from the apparatus. Then the insert gets extracted from the cube assembly, the can material at the diamond side is removed, the parts are inspected, and then the diamond surface gets lapped to a specified height.

3) Finishing: The finishing process of the polycrystalline sample is divided in several operations (refer to Figure 1-4).

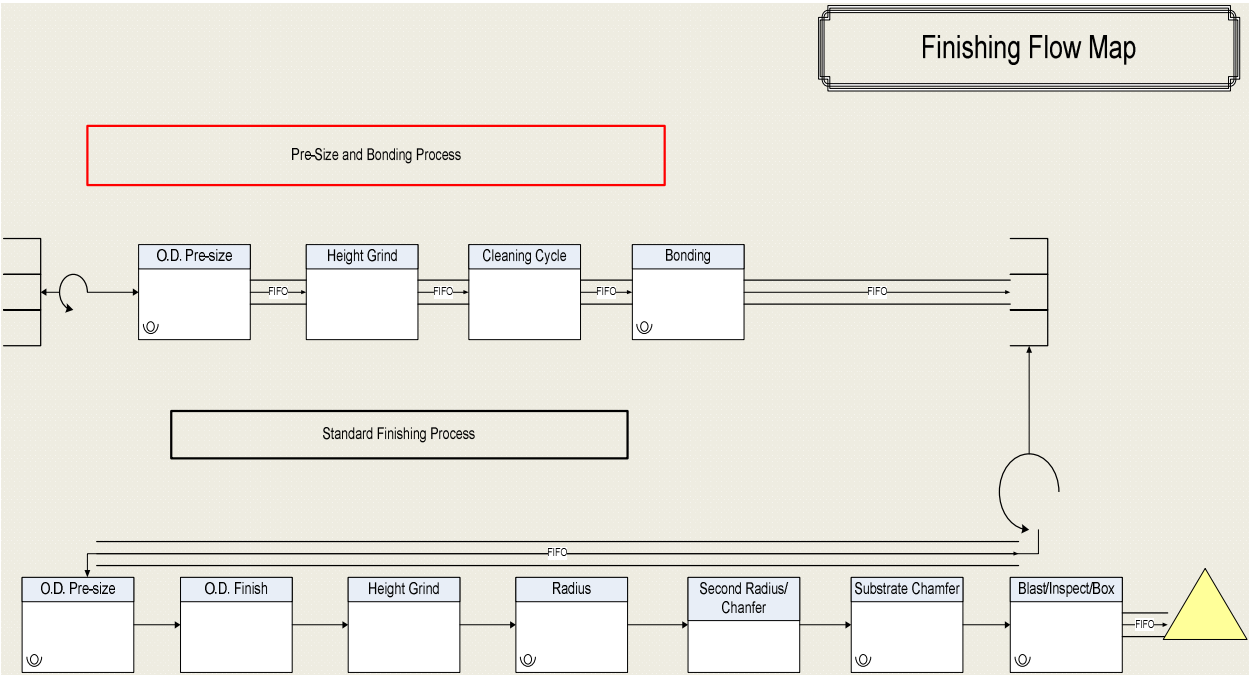

Figure 1-4: Finishing process flow for polycrystalline diamond compact inserts.

a) Outer diameter pre-sizing: Once the inserts have been lapped to the specified diamond thickness, the outer diameter gets ground to a dimension approximately 0.010” larger than that specified by the customer finish size. In this process the can material is removed and the operators have a better reference point for the next grinding operations.
b) Pre-bonding height grind: The bottom side of the insert gets surface ground to remove the can material and to prepare the surface for the bonding operation.
c) Cleaning cycle: The inserts are submerged in a solution in an ultrasonic bath to remove all the residues from the previous processes.
d) Bonding: A secondary tungsten-carbide substrate is bonded to the insert utilizing a special high-temperature brazing alloy. This process is done to achieve the customer’s required height. Steps (a) though (d) would not be necessary if the insert could be originally sintered in a cube assembly to the customer’s required height.
e) Outer diameter second pre-size grinding: After the insert gets its extension bonded, its outer diameter is ground to a dimension 0.001” larger than the finish requirement.
f) Finish outer diameter grinding: The insert is ground to finish outer diameter as per customer requirement.
g) Second height grind: The bottom of the insert gets surface ground to the finish height.

h) Diamond Radius/Chamfer: The diamond outer edge gets ground to a specified radius or chamfer as per customer requirements.

i) Blasting and inspecting: The insert gets bead blasted to clean its surfaces and a full inspection is performed to ensure all the dimensions match customer's expectations.

1.2.2 Process Cost Drivers

Each of the steps described in the previous section: Assembly, Press, and Finishing, contributes to the cost of producing polycrystalline diamond compact inserts.

- Assembly: Materials and labor are the two main cost components for this process. Over 60% of the cost pertains to materials, and about 30% of that corresponds to the additional substrate material and brazing alloy utilized to extend the inserts to the customer's required height.
- Press: Machine cycle time and power consumption are two small components on this processing cost of the inserts. The most important component on the press process cost is the press anvil life. With a price of more than 3,000 U.S. dollars per unit, it is important to ensure a long life to the anvils to amortize the initial cost over a long period of time.
- Finishing: In this process labor is the main cost. Machines allocation, equipment supplies, and machine wear are important components, too. With the current method, a tungsten-carbide backup is bonded to the PDC cutters in a separate bonding operation in order to attain the customer's size requirements. This process takes four operations, equipment, two operators, processing time, and extra material cost, which

constitute about 40% of the finishing cost. In Figure 1-5 the finishing process flow is represented with the extra operations required for the bonding process defined within the red ellipsis. Eliminating the extra operations would reduce the cost of finishing the inserts by 40%.

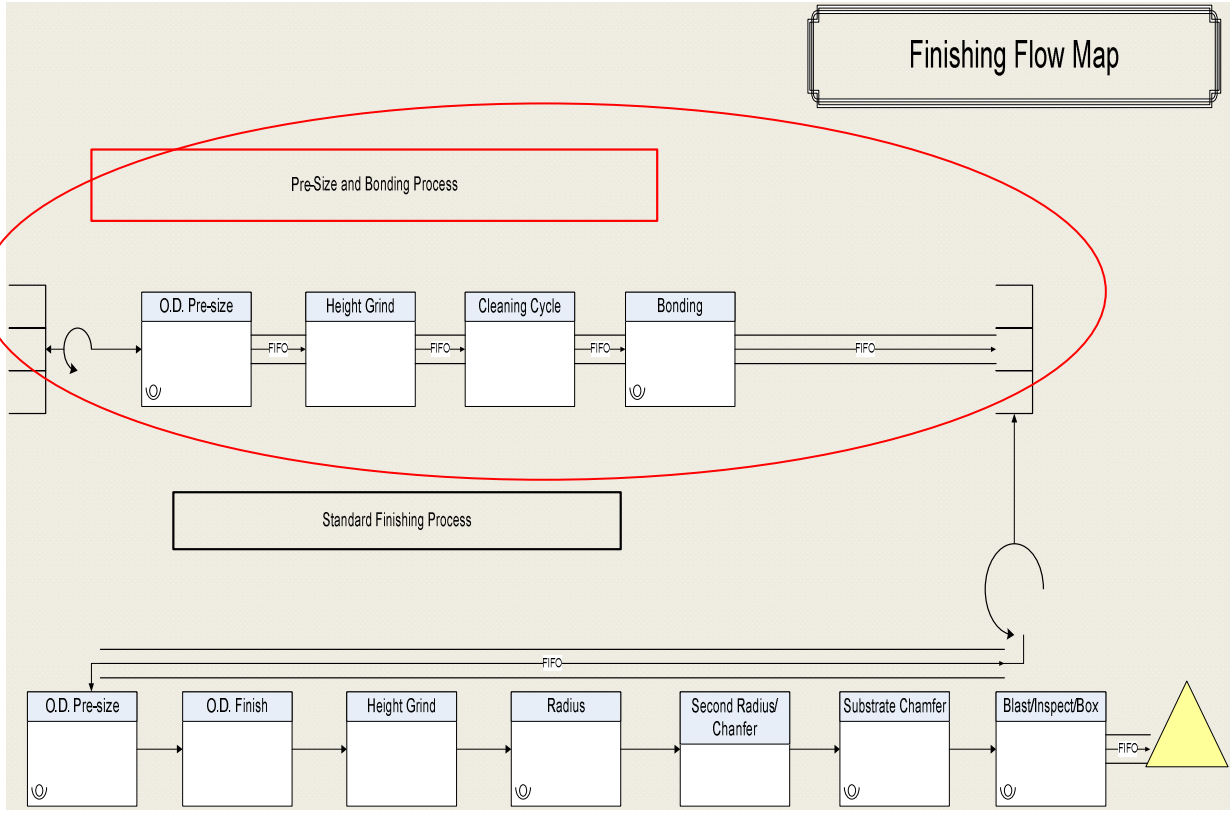

Figure 1-5: Finishing manufacturing flow for PDC inserts. On the top portion of the flow chart are the processes required to extend the length of the part to the customer's specification.

1.3 Objective

The purpose of this research is to prove how modifications can be made to the cube cell assembly for a high-pressure/high-temperature cubic press that allow larger inserts to be processed. Larger samples will eliminate the finishing steps shown in red in Figure 1-5 thereby

reducing the material cost in the assembly. It will be proved how through testing and experimentation that this new process can produce Polycrystalline Diamond Compact inserts with lower manufacturing cost and no loss in performance (wear resistance) compared to those produced with the traditional process.

1.4 Thesis Statement

A modified cube cell assembly that accommodates larger inserts for use in the cubic high-pressure/high- temperature apparatus for the manufacturing of polycrystalline diamond compact inserts can be developed that will lead to reductions in cost while not diminishing the insert performance.

1.5 Hypotheses

1. It is possible to design a new cube-cell assembly for use in the high-pressure/high-temperature cubic press, which will allow the synthesis of larger inserts (two .600" tall inserts) per press cycle without using end heating.

The outcome of the proposed cell design will be:

- The reduction of process steps of the parts
- Minimization of material inventory
- Free-up equipment and operators
- Reduction of the total process time of the finish goods

2. The new cell design for the use in the high-pressure/high-temperature cubic press does not negatively affect the performance of the PDC cutter produced. The Cutter performance of concern in this study includes:
    - Evaluating the inserts wear resistance on the heavy wear test.
    - Comparing the exaggerated carbide grain growth at the diamond-WC substrate interface.
    - Analyze and measure the microstructure difference between the inserts from both processes.

1.6 Methodology

The first part of the research will be to gather information regarding cell designs for high-pressure/ high-temperature cubic presses for the manufacturing of synthetic diamond, PDC, PCBN or any process that involves a cubic press and sintering processes. This information will be used as the foundation for the thesis and for the design of the new cell assembly because it will show the requirements for the diamond synthesis process, its limits, and the function that each of the cube cell components has in the process. Many test press cycles will be performed in order to qualify the new components and to set the press power parameters necessary for the sintering process, as well as for gathering the cost and the processing time information for the traditional and the modified cell designs.

The in-house mechanical test results and material analysis will be presented in the paper to show if there is any impact on the performance of the product manufactured utilizing the modified cell design compared to the traditional bonded product.

For the internal material analysis, the PDC inserts will be cut using Electrical Discharge Machining (EDM), mounted in bakelite, polished, and analyzed with a Scanning Electron Microscope (SEM) and X-Ray element analysis. In the case of the mechanical wear of the part, heavy wear tests will be performed on the PDC cutters at US Synthetic's testing facility. This information will be compiled and a t-test will be utilized to compare the data from the testing of the two processes.

1.7 Scope and Delimitations

The research will be conducted at US Synthetic's facility. The corporation's core competency is the manufacturing of PDC inserts for the oil and gas industry. In this thesis, the research work will be limited to the most common PDC insert size utilized in the oil and gas bit drilling industry, 16mm in diameter by 13 mm high.

The time for implementation, observation, testing, and analysis will last approximately one year.

The researcher will assume that the other manufacturing companies utilize the same or very similar cell design as the one used by US Synthetic, which is very similar to the one shown in most of the researched papers.

Because of the sensitivity of the information captured through this research, some of this information has to be kept under proprietary status.

Impact on the processing yields or scrap rate will not be studied on this research paper.

1.8 Conclusion

This research has relevance for Polycrystalline Diamond Compact inserts for the gas and oil drilling applications, but it can also impact the manufacturing of Polycrystalline Cubic Boron Nitride for cutting tools and the fabrication of superconductors, all of which employ a high-pressure/high-temperature operation like the one which will be studied in this proposed thesis research. Therefore, if the new method is shown to work by reducing cost and maintaining performance, a number of different products in various industries should benefit from what is learned. The product will be processed at finished size, eliminating secondary bonding processes and opportunities for generating rejects. As a result of this study, diamond-sintering companies will be able to reduce inventory and cost, increase their cash flow, and maintain a competitive advantage in their market.

The remainder of this thesis will cover the following topics:

- Chapter 2: Literature review, what is known about the diamond synthesis method, and the importance of the cube cell assembly components in the diamond sintering process.
- Chapter 3: Differences between the traditional and the proposed process and how these processes will be compared.
- Chapter 4: Test results from the tests suggested in Chapter 3.
- Chapter 5: Conclusion and recommendations.

2 REVIEW OF LITERATURE

2.1 Diamonds

Diamond is the hardest known natural material. Because of its properties, diamond has intrigued mankind since its early discovery.

Diamonds were first found around 2700 years ago in the alluvial deposits in ancient India by the originators of the Indus Valley Civilization, the Dravidians.

India was the only source of diamonds until the Brazilian deposits were found. By the time both suppliers started depleting their natural resources, South Africa's deposits were found. South Africa became the largest producer of diamond.

The next region following South Africa to become a natural diamond producer was Russia. The discovery of diamond deposits in kimberlite, which is a low-quartz-containing igneous rock, was quite different than the previous mentioned diamond suppliers that found them in the alluvial deposits (Longford, 1977). Currently Australia and Canada have a share on the supply of natural stones.

2.2 Diamond and Science

One of the earliest documented experiments to analyze the nature of the diamond was made between 1694 and 1695 by G. Averani and C.A. Targioni of the "Accademia del Cimento". In their experiment, they established that diamonds vanished when a high heat was

applied to them; they assumed that the diamond was getting converted into vapor by the burning heat. At the present time, we know that because of the presence of oxygen and a heat source, it was converted into carbon dioxide (Hall, 1970).

Sir Isaac Newton reported in 1704 the first analysis of diamond where he described it as an “unctuous substance coagulated”, which bears a resemblance to carbon mixed with oil and amber (Newton, 1721).

In 1792 Antoine Lavoisier reported on the smoldering of diamond to identify its composition, but he was not able to achieve a clear conclusion with the results of his experiments (Lavoisier, 1799).

Later in 1797, Smithson-Tennant, by incinerating diamond crystals in a fused nitre bath and collecting the remnant gas (CO_2), was the first scientist to demonstrate that the diamond consisted of only one material: carbon. He repeated the experiment utilizing the same weight of graphite and got the identical results as his previous experiment with diamond (Tennant, 1797). In his experiment, Tennant established that diamond and graphite are allotropic forms of carbon. Since then, the conversion of graphite to its precious relative has been one of the most controversial problems of the science (Hall, 1961).

L.B. Guyton de Morveau was able to reproduce Lavoisier’s experiment and observed the same results as Tennant’s experiment. By heating the diamond with a burning-glass; he was able to perceive black spots forming on the surface of the diamond. Shortly after, he compared his results with the experiments done by Clouet in 1798, and they were able to reaffirm that the diamond composition is purely carbon. In Clouet’s experiment, iron and diamond were heated together, and as a result steel was formed, which is the same result that can be obtained by heating up and dissolving iron with carbon black or graphite (Streeter, 1892).

2.2.1 Diamond and Graphite, Allotropic Form of Carbon

In 1961 Professor H. Tracy Hall from Brigham Young University, in his research paper "The Synthesis of Diamond", describes the differences between diamond and graphite.

In his paper, he explains that the difference at the atomic level between diamond and graphite could not be observed until the decade of 1910 to 1920, when the X-ray diffraction technique was developed to help study the crystal structures.

Diamond consists of carbon atoms aligned in creased, hexagonal rings lying in the crystallographic plane 111, which is the natural cleavage plane of the diamond. These carbon rings are piled one on top of the other, duplicating every four times the initial sequence. Then every central atom is bounded by four other atoms at an equal distances. All distances between atoms are 1.54Å as shown in Figure 2-1.

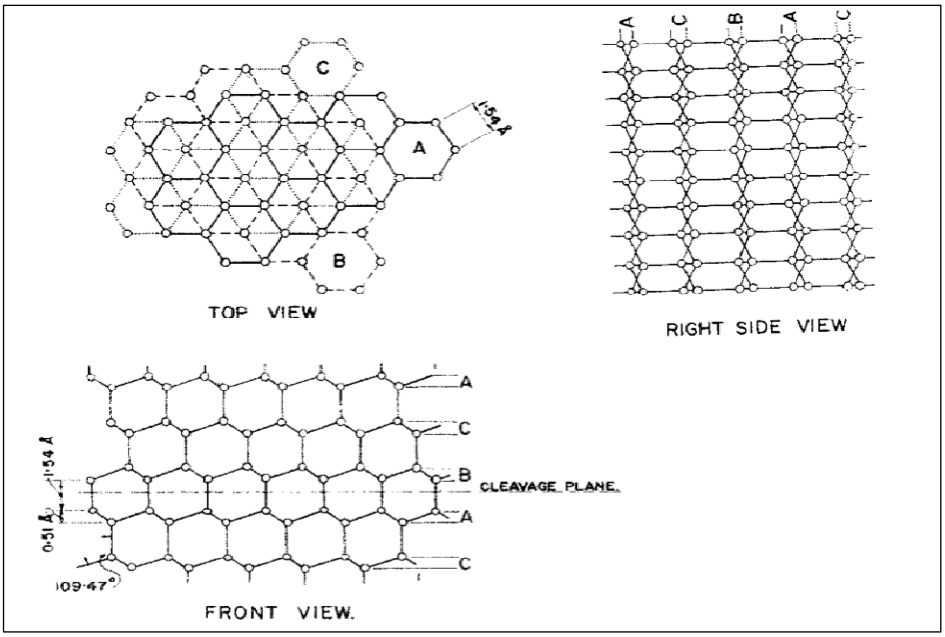

Figure 2-1: Orthographic projections of diamond space lattice. The hexagonal rings of layer A are outlined with solid lines, layer B with dashes, and layer C with dots.

In the case of graphite, the arrangement of the atoms is comparable to that of diamond in the sense that the layers are heaped in a parallel way one on top of the other creating hexagonal rings. The main difference is that they are not creased as closely as the diamond. The distance between the atoms in the same layer is closer than in the diamond formation at 1.42Å, but the individual planes are spaced farther apart at 3.37Å. This large spacing is shown in Figure 2-2.

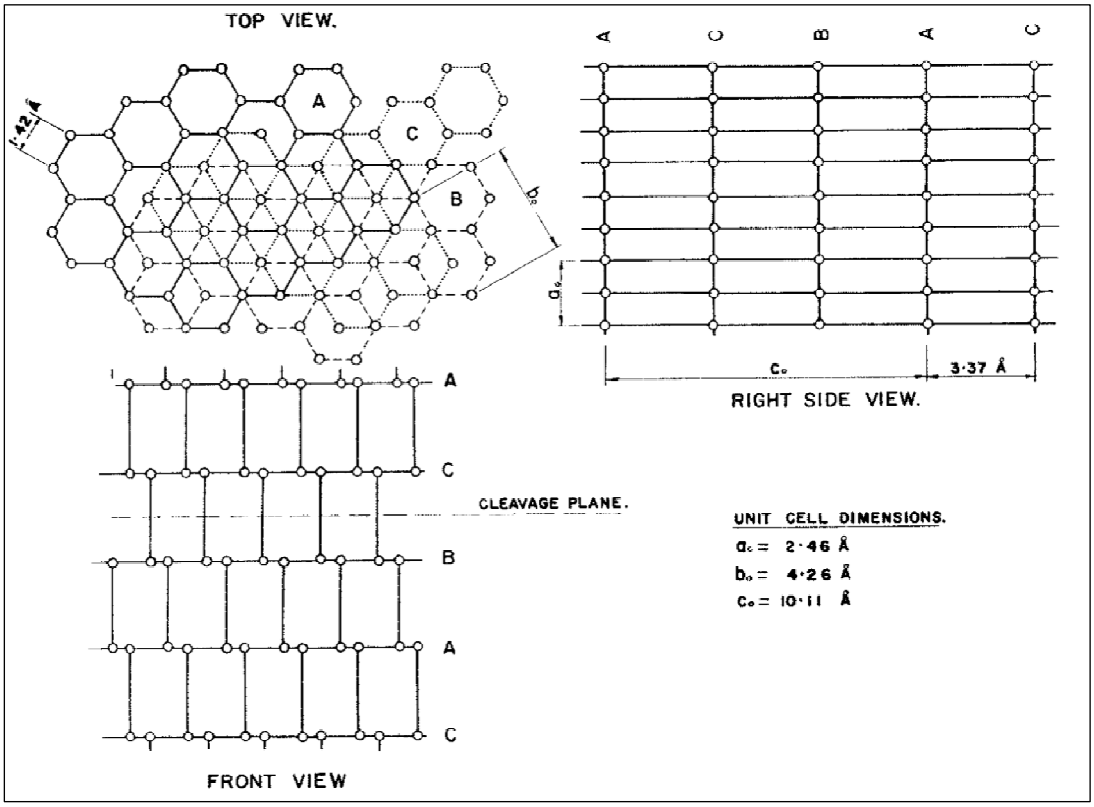

Figure 2-2: Orthographic projections of rhombohedra graphite space lattice.

The bonding between the atoms for diamond is predominately covalent due to the formation of sp^3 hybrid bonds. All bonds are aliphatic in character, and equal, meaning that they are aligned at equivalent distances from each other. In the case of graphite, it has double bond character in its rings, so the molecule is aromatic in character (Hall, 1961).

In the solid state of carbon, there are two favored forms of electronic atom bonding:

- Sp^2 type where each atom is bonded to three (3) other atoms at an equal distance in a 120^o hexagonal form, called Hexagonal Close Packed (HCP) structure.
- Sp^3 type where each atom is bonded to four (4) other atoms in a tetrahedral formation. The distance between the atoms is equal. This is diamond cubic structure (Bundy, 1995).

By comparing the two crystal arrangements, it can be deduced that graphite can be converted into diamond by utilizing high pressure to shorten the distance between the graphite bonds, forcing the rings to crease closer to each other to emulate the ones in the diamond crystal lattice (Hall, 1961).

2.2.2 Transformation Problems

The process of changing graphite to diamond is more complex than just applying force to the graphite to realign its atom's bonding. As expressed by Professor Hall in his paper, there are two main problems when we are talking about a change in the polymorphic form of a material. These two problems are: the thermodynamic problem and the chemical kinetic problem.

In his paper, Hall explains the importance of having both in consideration when we are trying to convert graphite into diamond. Thermodynamics is concerned with the relative energies of the reactants and the resultants of a chemical response. So, if we want to change graphite into diamond, we need to ensure that we have a negative relative energy in order to have the thermodynamic permission to convert one to the other. In case of having a very large negative relative energy number, meaning that the free energy from the reactant is much greater than the

free energy of the product, the reaction will still happen, but it will be difficult to control the process. Here is where the kinetics comes into place.

We now know that at atmospheric pressure the diamond is thermodynamically unstable with respect to graphite. So if we increase the temperature of the diamond it will convert back to graphite. To prevent this reaction and to keep the relative energy negative, when synthesizing diamond it is necessary to increase the pressure proportionally to the increase in temperature. The higher the temperature utilized, the higher the pressure that needs to be applied (Hall, 1961).

In 1976 the equilibrium boundary between diamond and graphite was determined over a temperature range that included 1100°C and 1625°C, and the pressure was computed in force/area measurements. Dr. C. Scott Kennedy and George C. Kennedy published a research work in which an equilibrium equation for the diamond-graphite boundary was formulated and tested. The equation is

$$P(\text{kbar}) = 19.4 + T(^{\circ}\text{C})/40 \text{ kbar} \qquad (2\text{-}1)$$

The results of the equation were plotted, and they matched the results from previous research works done on determining the diamond-graphite temperature-pressure phase diagram. Figure 2-3 shows the comparison of Kennedy's work and the prior work published by General Electric Company's research group. All G.E.'s work was done on a "belt" press, which is quite hard to calibrate for a precise internal pressure measurement. Kennedy's work was done in a piston-cylinder apparatus with a "Zero-friction" cell design, to ensure a better pressure representation (Kennedy, 1976).

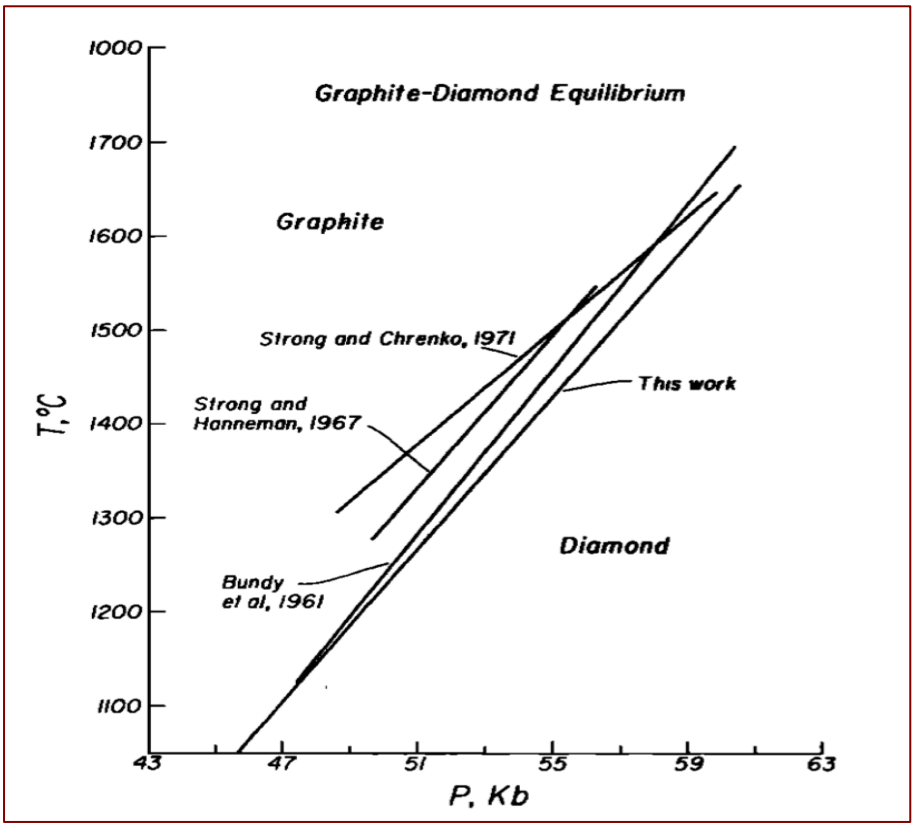

Figure 2-3: Graphite-diamond equilibrium graph which shows the comparison between Kennedy's work (This work) and previous published work by G.E's researchers (Kennedy).

This graphite-diamond phase diagram is a good source of information on which researchers and diamond manufacturers to base their processes. Most of the information on this topic does not get published. More recent work shows that spontaneous transformations from one solid phase to another can happen at room temperature, but it gets reverted to graphite after decompression (Bundy, 1995).

The use of a solvent-catalyst, like nickel, cobalt, or iron is required to take the carbon atoms apart from the graphite into a solution allowing them to rearrange, precipitating as diamond crystals.

In the case of sintering diamond to carbide tungsten substrates, as in the manufacturing of polycrystalline diamond compact inserts, the cobalt utilized as a binder in the cemented carbide infiltrates through the diamond crystals with the function of catalyst on the nucleation of the diamond to diamond. Without the help of the cobalt, the synthesis of the diamond crystals will be almost impossible (Katzman and Libby, 1971).

2.3 High-Pressure/High-Temperature Apparatus

Now that we understand the importance of the high-pressure/ high-temperature conditions for the diamond synthesis process, we are going to compare and analyze the different types of high-pressure/high-temperature apparatus.

When we talk about high pressure, it is important to be able to differentiate between the two foremost classifications:

1) Dynamic technique, which creates a high-pressure in only a fraction of a second. In order to achieve this pressure the utilization of impact or explosive is required.
2) Static technique, where the high pressure can be maintained for long periods of time. (Bundy, 1988)

The pressure generated by these techniques can be categorized in three classifications depending on how its force is directed. Hydrostatic is when the pressure is directed in all directions; uniaxial is observed when the force is directed to a definite direction; and quasi-hydrostatic is when there is a combination of the previous two (Huppertz, 2004).

2.3.1 Multi-anvil Apparatus

Static high-pressure apparatus can create almost hydrostatic pressure conditions that can be held at different pressure levels depending on the volume of the sample.

The pioneer in high-pressure experiments was P. W. Bridgman, who, with his high-pressure apparatus made of two Carboloy plates, was able to generate the pressure necessary for his experiments (up to 100,000 kg/cm²) on a thin disc (Bridgman, 1952). Bridgman was able to supply to the science world a large amount of data regarding the shear strength of materials at high pressure. In Figure 2-4 we can observe Bridgman's press (A, B) anvils, (C, D) support rings, and (E, F, G) sample with gasket.

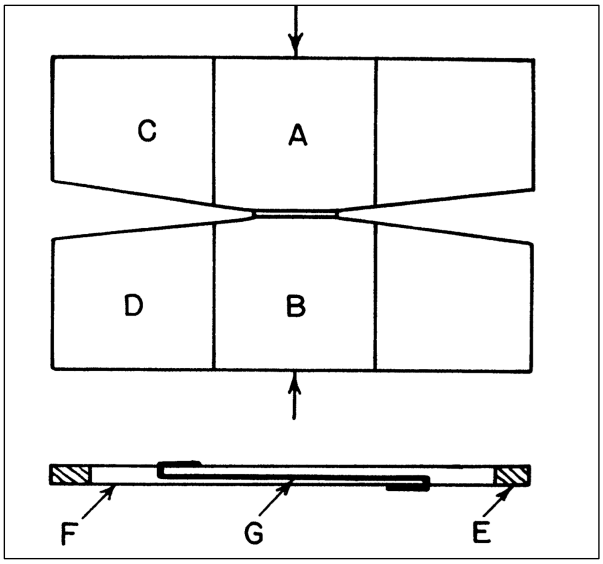

Figure 2-1: Bridgman's pressure apparatus.

F. P. Bundy modified Bridgman's two-dimensional device by adding dish shape to the anvils, allowing a larger working volume for experimentation. Bundy also added a heating

source for the sample (Huppertz, 2004). Figure 2-5 illustrates (A) Bundy's "saucer" press with its (a) sample and (b) carbide anvils, while (t) denotes the gasket thickness.

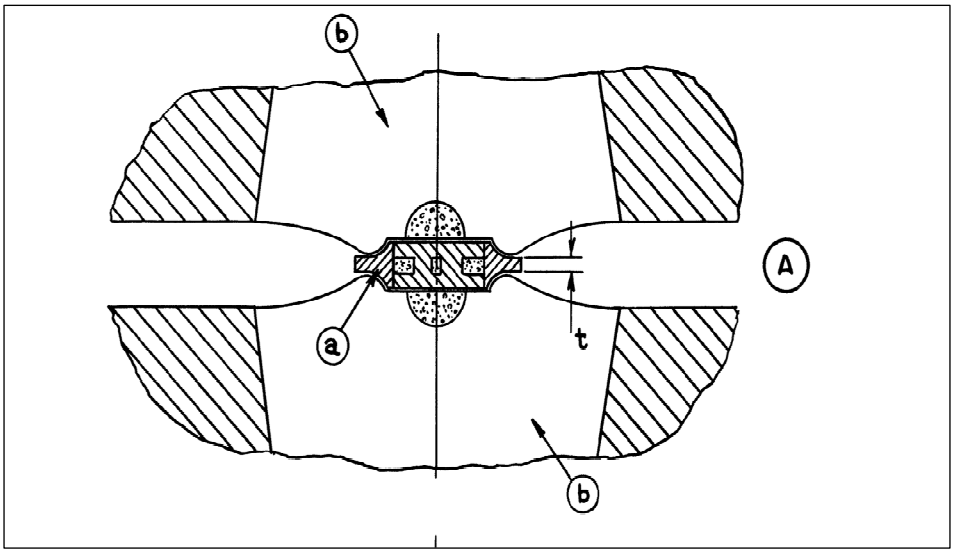

Figure 2-5: Bundy's "saucer" high- pressure apparatus

In January of 1953, while employed at General Electric, Dr. Hall invented the "Belt" press. This was the first apparatus capable of generating enough pressure and temperature to sinter diamond. General Electric was the first company to synthesize diamond utilizing this apparatus on December 16, 1954.

The belt press was able to reach pressures of up to 150,000 atmospheres and a temperature of 2000° Celsius for long periods of time. This apparatus was composed of two main carbide anvils, supported by several steel rings that apply axial pressure to the sample cell assembly. At the same time, lateral support is given to the cell assembly by a carbide die and a series of steel rings, which provide lateral thrust to the cell (Hall, 1980). A representation of the

belt press is shown in Figure 2-6. We can observe the carbide anvils vertically opposed, in the middle inside the carbide die the cell assembly, and all supported by several steel rings.

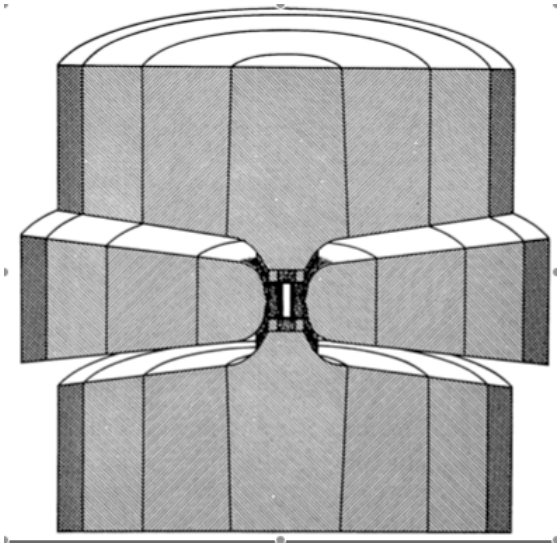

Figure 2-6: Belt press cross-section image.

Because of company secrecy and government nondisclosure orders, Dr. Hall was not able to disclose any information regarding the belt press design. When he left G.E. in 1955 and took the job of Director of Research and Professor at Brigham Young University, he felt encouraged by peers and students to develop a new high-pressure/high-temperature apparatus. That is how the two multi-anvil apparatus were developed: Tetrahedral and Cubic. The cubic is the press the researcher utilized for this thesis.

In Figure 2-7 we can observe the tetrahedral press that was Dr. Hall's first multi-anvil press design. He described it as a three-dimensional extension of Bridgman's anvil press. Four cylinders pushed on tungsten carbide anvils that were supported by steel binding rings. The anvils advanced simultaneously to the center of the press where the cell assembly was placed.

Then each piston was advanced separately, allowing the gasket to form and the pressure to be induced in the sample. This was a very complicated process, took a long time to coordinate the rams, and needed a highly qualified operator to run it.

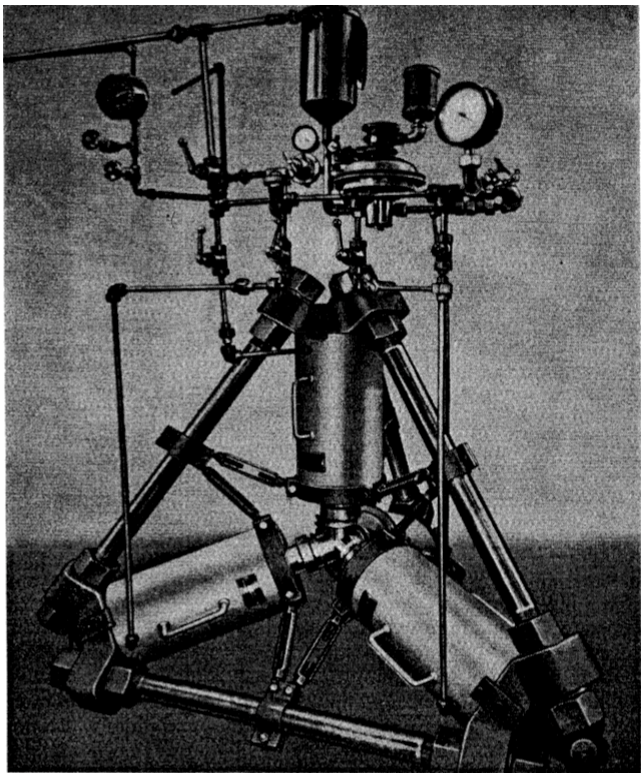

Figure 2-7: First tetrahedral press design.

By coming to a solution for the Tetrahedral press, Dr. Hall came up with a new press design: the Cubic. It had a synchronized hydraulic system that kept each anvil equidistant from the center of the press, being guided by position-indicating transducers. The press was supported in place by a series of tie bars to hold the cylinders together, and four guide pins per cylinder to keep the alignment of the anvils in a cubic shape and to prevent a possible anvil collision (Hall, 1980).

The cubic high-pressure/high-temperature apparatus has six electrically-insulated anvils as shown in Figure 2-8. Each advances inwards simultaneously in the three axis directions (X, Y, Z). Electrical power connections are normally set on the top and the bottom anvils, where a controlled flow of current is applied to the cell assembly to generate heat. The six rams are hydraulically interconnected to apply the same amount of pressure on each side of the assembly.

It is crucial to prevent the anvils from touching to avoid a short circuit and to prevent anvil cracking. The current life of an anvil that is run at 66 kbar and at temperatures within the range of 1400° to 1500° C is over 10,000 cycles.

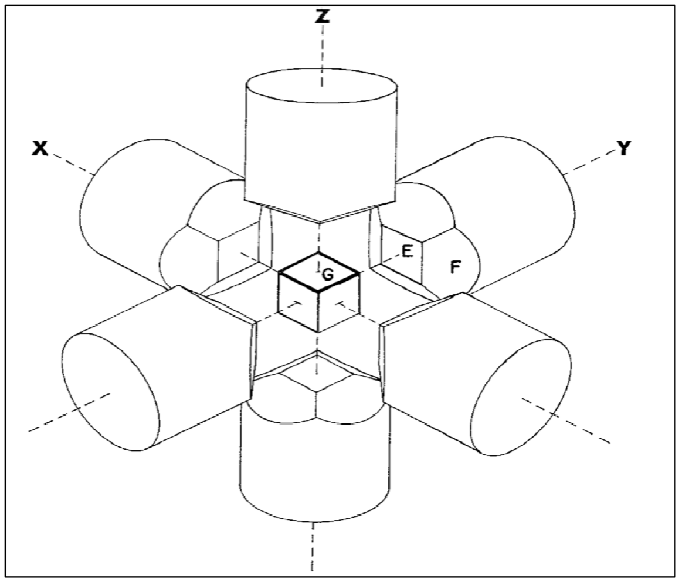

Figure 2-8: Cubic press axes (X, Y, Z), anvils (F), and cell (G).

The cubic press is widely used in the manufacturing environment because it is smaller, requires less maintenance, and is easier to utilize than the belt press. Today, because of more advanced computer technology, the cubic press can be loaded and left alone without the need of continuous supervision by an operator. Most of the diamond grit manufacturers in China make

use of this kind of press for their operation. With less than one tenth of the price tag of a belt press, it is easy to afford the capacity difference (lower for the cubic press) and buy multiple presses that will consistently produce with less intense operator dependency.

The cube cell assembly size is limited by the size of the press chamber and it is what mostly limits the manufacturing capacity of this press. That is why the researcher saw the necessity to redesign the cube cell assembly to allow larger samples to be processed. In most cases the chamber size only allows assemblies no larger than a two-inch cube.

2.4 Cube Cell Assembly

Now that we have a good background of the high-pressure/high-temperature apparatus history and function, it is crucial to spend time understanding the function and composition of the key constituent of the diamond synthesis process: the cube cell assembly.

The cell assembly has the purpose of confining and supporting the elements to be nucleated. It is responsible for keeping the components aligned in the proper position to ensure a good final product and at the same time protecting the samples from possible contamination. This is why it is so important to design the cell with tight tolerances and also to utilize the right elements that will sustain the pressure and the temperature in which the synthesis process takes place.

If we recall the definition of the diamond synthesis process, the two fundamental components are pressure and temperature. Therefore, the cell assembly has to be able to transmit the pressure exerted by the apparatus uniformly to the sample. The gasket and the pressure transmission media support this process.

The cell also has to transform the current induced through the anvils into heat in order to achieve the nucleation of the diamond-to-diamond particles, and the bond of the diamond to the tungsten carbide substrate. This synthesis process is accomplished by passing current through the heater at a low voltage and high amperage (Bhaumik, 1996).

The current cell design for the cubic high-pressure/high-temperature apparatus is comprised of a cube made of gasket/pressure media material, initially pyrophyllite, which has been bored to accept an insulating material tube, made out of zirconia, alumina, magnesia, or the most common of the materials, sodium chloride. Salt is preferred because of its easy accessibility and lower cost. Inside the insulation material tube a graphite heater with an isostatic material tube and the samples are centered in the cube assembly. An isostatic material disc is placed at each end and in between the samples. A graphite disc is placed at the most outer edge of the heater. Then, a titanium disc is placed against the graphite disc to conduct the current that flows from the anvils to the disc through the steel current rings. A pressure media button is located inside the current rings to support the cell and transmit the pressure. Figure 2-9 show the current cube cell assembly as previously described.

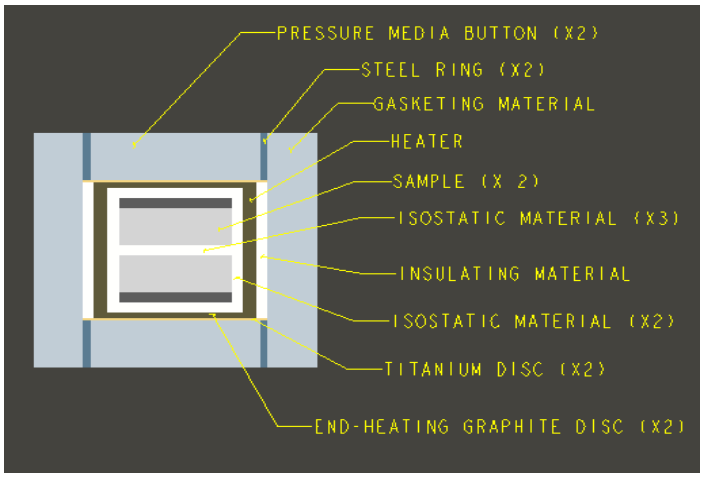

Figure 2-9: Current cell design for the cubic HP/HT apparatus.

Figure 2-10 exhibits the first cell assembly for the cubic press designed by Dr. Hall, we can observe that the first design did not have insulation between the heater and the gasket/pressure media material.

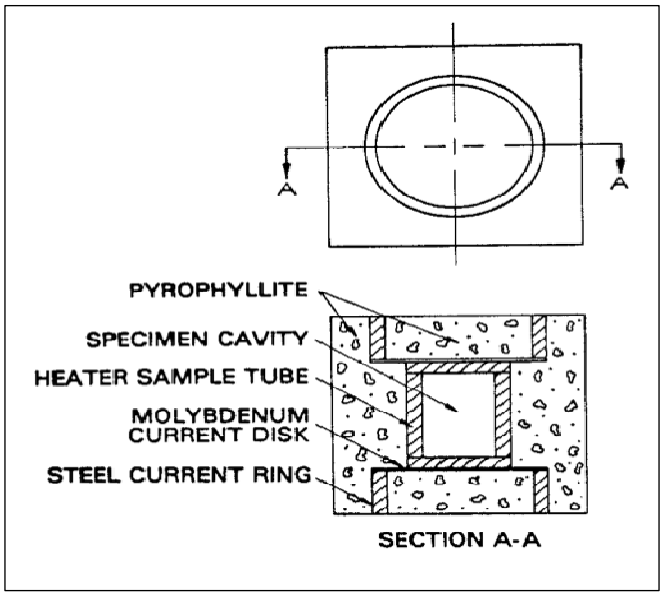

Figure 2-10: "Typical cubic cell" as described by Dr. Hall in 1956

There are not many substantial changes on the configuration of the cube cell since the first time Dr. Hall ran his experiments at B.Y.U. laboratories. The most significant modification was the addition of insulation between the pyrophyllite and the heater element to prevent the melting of the pyrophyllite and reacting with the graphite of the furnace at high temperatures (Corrigan and Bundy, 1975). The other important function of the insulating media placed between the pyrophyllite and the heater was to support the heater from cracking which can result in non-uniform heat patterns, jeopardizing the sintering process.

Therefore, three main components of the cube cell assembly are: the gasket and pressure media material, the heater assembly (heater and graphite disc), and the insulation and isostatic

pressure media. Below I will illustrate some of the essential information published pertinent to the prior listed components.

2.4.1 Gasket/ Pressure Media Material

In 1951, P.W. Bridgman reported his first high pressure research work. He was the first scientist to develop a fully operational high-pressure anvil apparatus. He used his press to analyze the electrical resistance of 72 different materials at room temperature. The important information for us is how, through the use of different materials, he was able to ensure a safe gasket with a higher coefficient of internal friction, and thus was able to prevent possible blowouts. He also describes that by using AgCl discs (made from pressed powder), which has a very low coefficient of internal friction (.03), he was able to transfer the pressure to the sample (Bridgman, 1952).

Ten years later Dr. Hall describes the functions executed by the gasket material with the goal to give a better understanding and motivate other scientists on the research of new possibilities to improve the high-pressure/high-temperature process.

Hall describes the three main functions of the gasket as follows:

- "Yielding": The material should yield to the thrust applied by the apparatus' anvils. It can take place by the compression of the gasket material, by flow, or by the mixture of both forces. The yielding should be an adequate amount to compress the components of the cell.
- "Confining" is mainly not yielding to the thrust of the advancing anvils, which in part is contradictory to the yielding concept, but it is necessary to prevent

the extrusion of the internal components through the gaps between the press anvils.

- "Support" is the third gasket function described by Dr. Hall. He describes this role mainly as a requirement to prolong the life of the apparatus' anvils. He explained that the pressure of the gasket at the edge of the anvil is the same as the pressure inside the chamber; and at the outer edge of the gasket the pressure decreases all the way down to one atmosphere. Ideally, the gasket against the high-pressure/high-temperature apparatus anvil should support a pressure gradient. This pressure gradient will prevent a sharp line of demarcation between the pressure from the interior of the chamber and the pressure of the most outer edge of the gasket, preventing the formation of a high stress-concentration line. This pressure reduction allows the tungsten carbide anvils to sustain higher loads of pressure than their normal limits.

In order for the gasket material to be able to accomplish these three previous functions, it is very important to choose the right substance and the proper thickness for the job. By reducing the thickness of the gasket we will also limit the motion of the anvils, thereby reducing the pressure in the apparatus chamber, limiting its effective capacity.

In some cases a material with a lower coefficient of internal friction can be utilized to ensure a superior transmission of pressure to the sample. By adding a thin layer of a high coefficient of internal friction material to the most outer layer of the cube assembly it is possible to increase the coefficient of sliding friction between the cube assembly and the press anvils.

This process will ensure a better grip of the material against the apparatus components, preventing decompression failures (Hall, 1961).

In the case of the natural pyrophyllite, its pressure transmitting properties are different depending on its impurities. Its impurities are dependent upon to the location where the rock was quarried. Some companies grind the pyrophyllite rock into powder, blend it, and then press it into a block to ensure a homogenous pressure media component (Li et al, 2007).

By utilizing man-made powder mixtures it is easier to control its composition and ensure a homogenous distribution of its elements. Most of the manufacturing companies utilize a mixture of materials to attain a better combination of coefficients of internal friction to support the press components and ensure the maximum possible pressure transmission. These mixtures have been tested and improved through the years. On the “High-Pressure Apparatus” paper Dr. hall describes that most of the ultrahigh-pressure gasket materials currently in use have a coefficient of internal friction that varies from 0.25 to 0.50. In the same paper he offered a table with the most common gasket materials and their coefficients of internal pressures collected at 24,200 atmospheres utilizing Bridgman’s shear apparatus (Hall, 1961).

Table 1 represents the data collected by Dr. Hall from the work done by Dr. Bridgman on the different coefficients of internal friction of materials that he found relevant to his high-pressure/high-temperature research.

Table 2: Coefficient of Friction of some Materials at 24,200 Atmospheres

Ferric Oxide Powder	0.71	“Micro-Cell” Earth Powder	0.37
Zinc Oxide Powder	0.58	Calcium Hydroxide Powder	0.27
Pumice Stone Powder	0.52	Pyrophyllite Powder	0.25
Chromic Oxide Powder	0.50	“Permagel” Clay Powder	0.18
Pyrophyllite Natural Block	0.47	Boric Acid Powder	0.14
“Attasol” Clay Powder	0.47	KCl Powder	0.12
Lead Dioxide Powder	0.46	NaCl Powder	0.12
Manganese Dioxide Powder	0.45	Mica Sheet	0.07
Titanium Dioxide Powder	0.45	Boron Nitride Powder	0.07
Molybdenum Trioxide Powder	0.42	Graphite Powder	0.04
Tin Oxide Powder	0.41	Molybdenum di-sulfide Powder	0.04
Boron Carbide Powder	0.40	Silver Chloride Powder	0.03
Aluminum Hydroxide Powder	0.39	Indium Sheet	0.01

In order to get all the requirements necessary for a material to achieve the function of gasket material and at the same time provide to the sample the pressure required for the diamond synthesis process, its is crucial to combine different materials. In order for a material to transmit pressure hydrostatically it is necessary to have a lower coefficient of friction and low compressibility. The pressure media material has to have low thermal and electrical conductivity, preventing possible electrical shorts in the cell and heat loss in order to support the apparatus and the sample in the synthesis process. It also has to be chemically inert and thermally stable with a high melting point that should increase with pressure. By melting and reacting with the other components it could jeopardize the process, and it would be quite difficult to have a repeatable method.

Li describes in his paper that changes in diamond manufacturing technology led to the use of higher pressures and temperatures which at the same time led to the rearrangement of the

pressure in the cell and to change in the hydrostatic stress state. He re-affirms that non-uniform pressure in the cell affects the diamond synthesis process. In order to prove this theory he designed a finite-element analysis (FEA) combined with numerical engineering analysis to model pressure-distribution differences (Li et al, 2007).

He also described that the cube cell assembly suffers plastic deformation and volume compression. The unequally distributed deviatoric stresses and isotropy hydrostatic pressure generate interior stresses on the cube material, and for his test he uses the Mohr-Coulomb elastic-plastic model.

$$|\tau| = C - \sigma \cdot \tan\varphi \qquad (2\text{-}2)$$

Where τ is the shearing stress, C is the cohesive strength, σ is the normal stress, and φ is the angle of internal friction.

For the FEA the hydrostatic pressure created by the volume compression was defined by the equation of state in his test.

$$P = K_0 + K_1\mu + K_2\mu^2 + K_3\mu^3 + (K_4 + K_5\mu + K_6\mu^2)E \qquad (2\text{-}3)$$

Where $\mu = \rho / \rho_0 - 1$, and ρ is the density, and ρ_0 is the initial density. When K_1 is the bulk modulus then equation 2.3 can be simplified by

$$P = K_1 \cdot \mu \qquad (2\text{-}4)$$

From his test, he was able to see that hydrostatic pressure is constant over most of the cube assembly but because of the extrusion of material at the gasket zone, the pressure distribution becomes non-uniform and volume-compressing rate decreases as well as the hydrostatic pressure. He was also able to observe that the strain intensity of the edges was higher than in the middle of the cell. The most important findings were that 90% of the pressure in the cell was isotropic hydrostatic pressure derived from the volume compression and that the pressure gradient is a result of the non-uniform distributing deviatoric stress which comes from the plastic deformation of the cell at the gasket area. Represented in Figure 2-11 is Dr. Li's FEA model analysis where it shows the plastic deformation of the pyrophyllite at the gasket area.

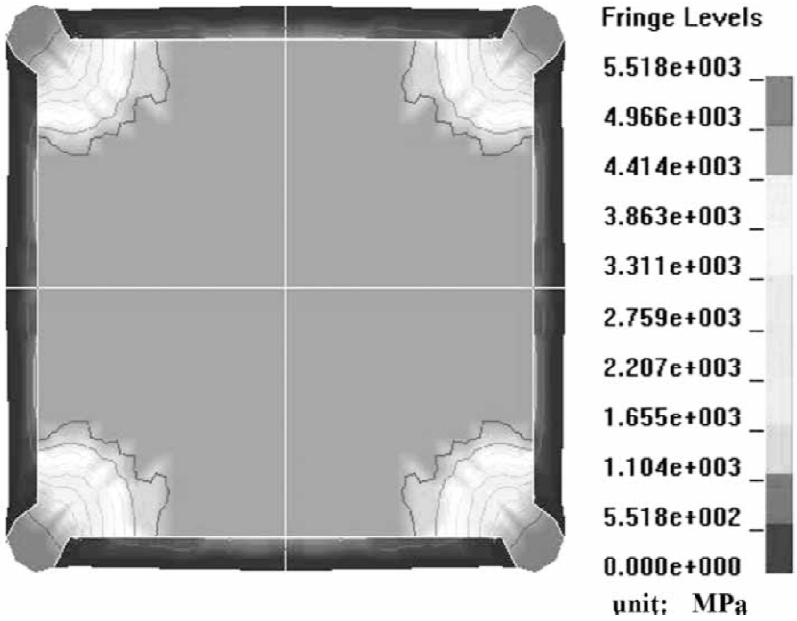

Figure 2-11: Contour plot of the hydrostatic pressure on the symmetrical section of the pyrophyllite block from Li et al FEA model.

In his test, Li utilized reconstituted pyrophyllite because in the natural pyrophyllite there are many impurities that could negatively influence the pressure transmitting properties of the

material. Pyrophyllite is also susceptible to moisture content; the amount of moisture in the material changes its mechanical properties (Li et al, 2007). These two issues described by Li are the main reasons why most of the PDC and the PCBN manufacturers utilize their own synthetic formula for the pressure media material. One of the most common elements utilized in synthetic pressure media powder is talc, the chemical composition of which ($3Mgo.4SiO_2. H_2O$) is quite similar to pyrophyllite ($Al_2O_3.4SiO_2. H_2O$).

Silica, zircon, garnet, olivine, and other abrasive materials are utilized in the synthetic "pyrophyllite" to increase stiffness of the cube and some kind of binder, such as sodium silicate, is utilized to unify the mix components (McMurray, 2009).

Another important reason why the companies that utilize high-pressure/high-temperature processes produce their own version of synthetic pyrophyllite is because of the scarcity of suppliers. South Africa has the main pyrophyllite quarry in the world creating a high raw-material dependency from the PDC and CBN manufacturing companies. By these companies producing their own pressure media material they can control their powder inventory and their cube supply. By these means the companies can reduce their cost by 80%, free up cash flow and real estate normally utilized to acquire and store excess pyrophyllite ordered to compensate for the long lead times.

2.4.2 Furnace/ Heater

As we previously learned, high pressure and high temperature are necessary for the diamond synthesis process to happen. In this section we are going to describe and understand the heat-generation process.

The heat is generated inside the cell assembly by the resistance-heating process, which is the conversion of one energy form to another in a current –carrying medium. In this case is electrical energy is converted to thermal energy. The thermal energy E_g is generated by passing energy in form of electric current I through a medium with electrical resistance R_e *(Incropera, 1996).*

$$E_g = I^2 R_e \qquad (2\text{-}5)$$

In the case of the cube assembly for the high-pressure/high-temperature apparatus the electrical current is conducted to the cell by the press anvils. The current passes through the steel rings to the metal disc (molybdenum, titanium, etc) and then to the end heating discs and the heater tube. Figure 2-12 shows a traditional cell configuration where the current follows its path from the top anvil in the cell and out through the bottom anvil.

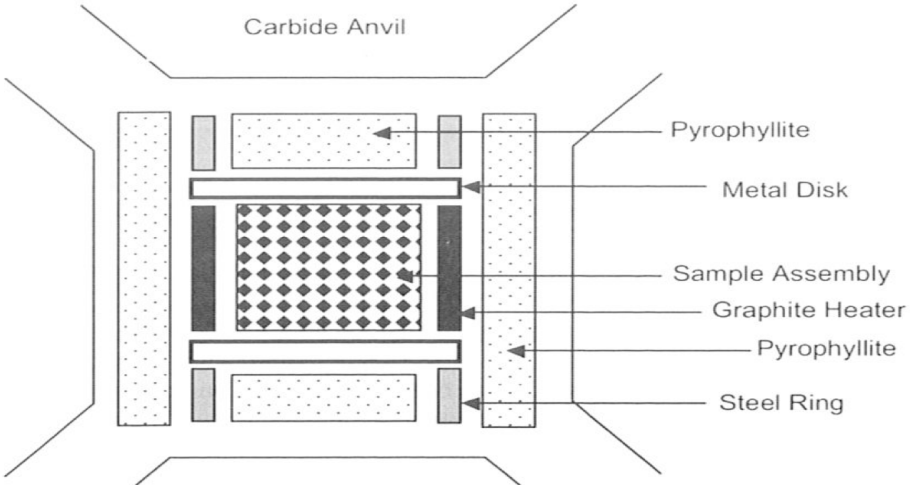

Figure 2-12: Cube cell assembly with anvils, the current is conducted from the top anvil to the steel ring, then conducted from the ring to the heater by the metal disc and follows the same cycle through the bottom anvil.

By passing low voltage (0 to 10 V) and high current (0-1000 A) through the heater, resistance heat is generated in the cell. In most cases, the heater is made out of graphite because it is easier to manufacture and it has the ideal resistance properties to achieve the desired temperatures. The heater has commonly a cylindrical shape, which eases the manufacturing of the component and contours around the sample guaranteeing a more uniform heat to the sample.

Choosing the right components for the cell assembly is crucial to accomplish a uniform heat distribution to the sample. A tight tolerance between the components of the cell is important to control the heat distribution and reduce the thermal gradients in the area where the synthesis process is taking place. Not having a uniform heat distribution around the sample might lead to an incomplete sintering of the diamond or an abnormal chemical reaction in the sample (Schmidt & Ulmer, 2004).

How the components are selected has an important impact on the thermal gradients in the cell assembly, mainly because of the capacity of the components to transport the heat by conduction. By the heater being cylindrical in shape the temperature inside the cell increases radially from the center. Also by being axially limited in size it induces heat conduction along the sample axis giving paraboloid isotherms with temperature variations of almost 100□C from the center of the cell towards the end of cell on the axial direction. In Figure 2-13, Dr. Hernlund's work represents the thermal profiles calculated with his model within a cell heated with a graphite heater.

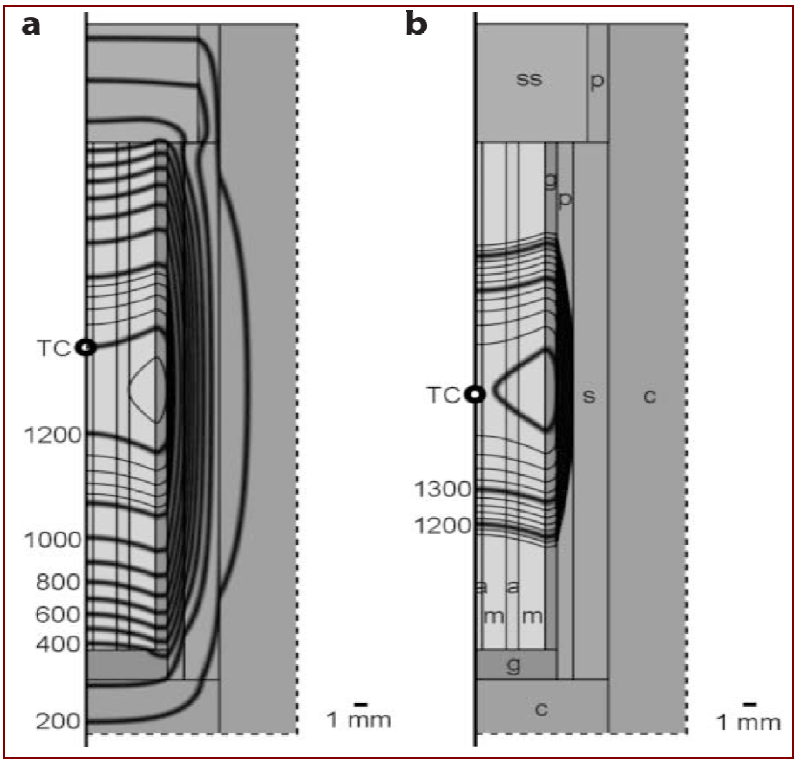

Figure 2-13: Thermal profiles calculated by Hernlund's model on a cube assembly using a graphite heater.

This problem is increased when the aspect ratio of the heater is increased (Hernlund et al, 2006). In our case it could be beneficial because we want to have uniform heat at the center of the cell to ensure the accomplishment of the diamond synthesis process, but we do not want to jeopardize the quality of the WC of the samples nor that of the press anvils.

A common complication caused by utilizing graphite as a heater element is its reaction with the other components. In the case of the steel rings or nickel discs, the graphite will start converting into diamond, losing its electrical conductivity qualities. It will also react with pyrophyllite at high temperatures, making it necessary to insulate the heater with a zirconia liner to prevent failures that could result in heat loss and non-uniform heating which will result in a faulty sintering process (Bhaumik et al,1996). The cell utilized by Bhaumik in his high

temperature research work is shown in Figure 2-14, where the molten pyrophyllite zone (1) did not affect the heater because it was protected with the zirconia liner (2).

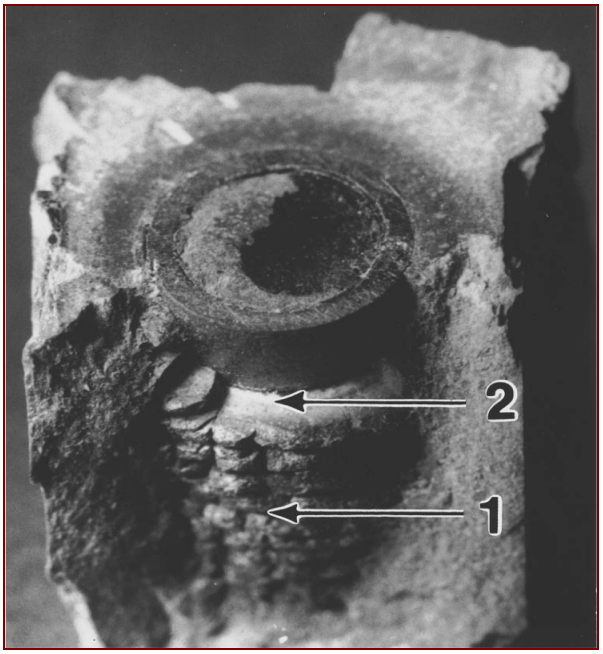

Figure 2-14: Cell used at high temperature presenting: molten pyrophyllite zone (1), zirconia liner (2) (Bhaumik, 1996).

Another common failure in the heating assembly is the deterioration of the steel rings at temperatures over 1800°C. Graphite can be utilized to replace the steel, but in order to prevent hot spots on the anvil faces it is necessary to place a tantalum disc between the rings and the anvil faces (Bhaumik et al, 1996). From the steel rings, heat loss is also observed to occur towards the top and the bottom anvils. This has an influence on the cell heat uniformity and also on the life of the anvils, which lose their strength as their face temperature rises (Wakatsuki et al, 1970). Replacing failed anvils represents the highest expense for a researcher or a manufacturer in the diamond synthesis process (Hall, 1964).

2.4.3 Insulation/ Isostatic Pressure Support

Insulation (thermal and electrical) and pressure support is the primarily function of the liner in the cube cell assembly for the cubic high-pressure/high-temperature press. If we recall in Figure 1-3, the traditional cube cell assembly shows that the isostatic material liner is utilized around the sample and also between the heater and the gasket media cube. When located around the samples it prevents grounding with the heater, which would produce a hot spot resulting in a scrap insert and possibly, as a consequence, a press blowout (Bhaumik et al, 1996). It also provides the insert with isostatic pressure support, which is necessary to ensure that the pressure exerted by the apparatus anvils will not plastically deform the shape of the sample.

In order for the liner to transfer pressure quasi-hydrostatically to the sample it needs to have a low coefficient of internal friction, which results in a low shear strength. Silver chloride has a low coefficient of internal friction, but it is expensive to be used in manufacturing. Boron nitride (BN) has also a low coefficient of internal friction, it is easy to press into shapes, and its cost is lower that the silver chloride. Some of the inconveniences of using boron nitride is that it has a higher thermal conductivity, which requires more power to maintain uniform heating in the cell, and at high pressure and high temperature it gets transformed into cubic boron nitride (CBN) losing its capacity to transmit pressure (Hall, 1980)

Sodium Chloride is the most common substance utilized in the industry. It has a low thermal conductivity under pressure and low temperature and it is more hydrostatic that pyrophyllite (Hall, 1980). Salt has no measurable strength at the temperatures at which the diamond synthesis is effectuated (Kennedy & Kennedy, 1976), meaning that pressure will be distributed hydrostatically through the sample. It has satisfactory thermal and electrical resistivity at high temperatures, and it remains chemically inactive in contact with the heater. Its

melting temperature increases with pressure, from 800°C at 1 atmosphere to approximately 1800°C at 100 kbar (Bundy, 1988).

At high temperatures the primary mode of heat transfer is by radiation. Pure salt transmits infrared radiation easily. Its thermal insulation can be improved by adding materials that will absorb infrared and re-radiate it in all directions, reducing the thermal differences. The most common materials added to improve the salt's insulating qualities are graphite, lampblack, zirconia, magnesia, and iron oxide (Mc Murray, 2009).

2.5 Possible Diamond Sintering Complications

Most of the manufacturing rejects in the diamond synthesis process are caused by not being able to maintain the sample within the diamond pressure and temperature boundaries throughout the process. If we recall Dr. Kennedy's work in section 2.2.2, he shows that there is a window where the diamond synthesis happens. It depends upon the capabilities of the apparatus and the cube cell design to maintain these properties throughout the process to have a complete diamond synthesis.

Other complications can occur during the PDC manufacturing process which are related to the dimensions of the cutters and equipment capability. After the inserts were pressed, the rest of the operations are mainly to shape the insert to customer's requirements. It is important to understand the capability limits of the equipment and the operators in the long run to ensure the manufacturability of the desired cutter.

Both of the previous possible complications are easy to detect, and they can be prevented with manufacturing instructions, proper components, and the right equipment for the job. But

there are other mechanical complications in the synthesis process that could have a substantial impact on the performance of the cutter, and they are more difficult to identify. The abnormal growth of diamond crystals and/or tungsten carbide could diminish the strength the polycrystalline diamond compact insert.

Dr. Shin and his team talk about the abnormal grain growth (AGG) of the polycrystalline diamond during the sintering process in a belt press at a pressure of 6 Gpa and 1600º C. For the test, the same process normally used in the commercial manufacturing of PDC inserts was utilized: diamond powder and a WC substrate, assembled in a can, heater, and cube assembly and later run on a press at high-pressure/high-temperature for a set period of time. For his tests, he utilized the same process as standard polycrystalline diamond compact inserts production, the only difference was that he extended the soak time and tested different diamond powder sizes.

Shin explains that the abnormal growth of large crystals of diamond occurs in the cobalt liquid phases. One of the reasons for the abnormal growth is the difference in the pressure distribution, by not being homogenous. The area with the higher pressure had the most abnormal growth. The area against the WC-Co substrate was less prone to have abnormal diamond grain growth, compared to the area against the can material. This was mainly because the can material is softer that the 10% WC-Co substrate, allowing higher pressure to be present on that area restricting the flow of WC in the catalytic solution.

Adding powdered tungsten carbide or cubic boron nitride to the diamond powder can control abnormal grain growth, and the tungsten dissolving from the tungsten carbide substrate is a good inhibitor as well. Dr. Shin also supports the idea that with the increase of the initial diamond particle size, the abnormal grain growth of the diamond crystals becomes less common (Shin et al, 2004). Figure 2-14 shows (a) a sample assembled with a WC-Co substrate and

confined in a tantalum (Ta) can assembly. It has a region (A) with a higher amount of AGG, in Figure 2-15 (b) that region is amplified utilizing a scanning electron microscope (SEM) and we can observe that the area closer to the Ta can has more AGG.

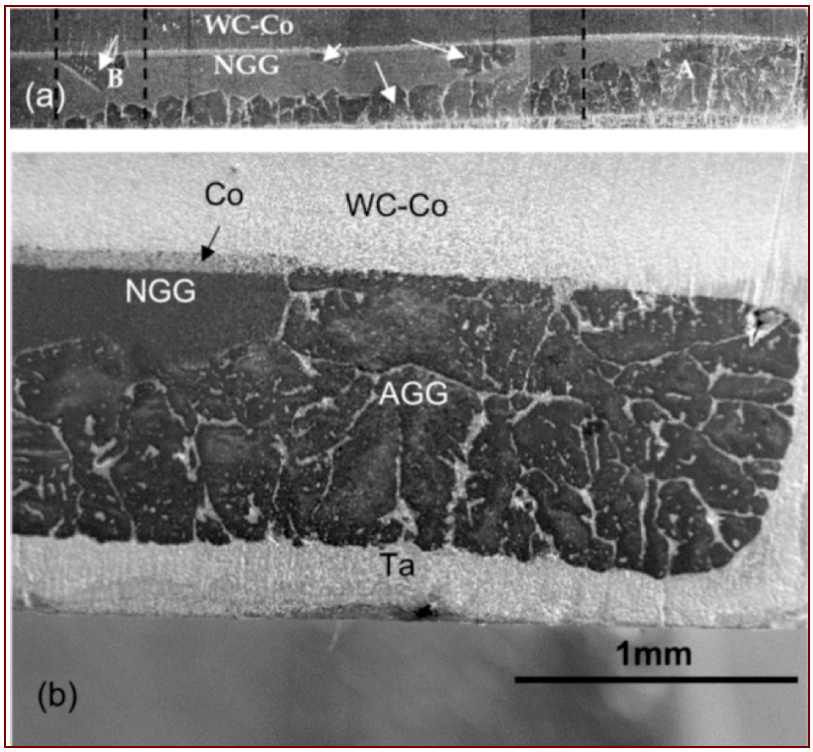

Figure 2-15: (a) Structure of cross-section of specimen A. Arrows point out abnormally grown grains. (b) SEM micrograph of area A in (a). AGG and NGG stand for AGG and NGG regions, respectively (Shin).

Dr. Hong linked the diamond abnormal grain growth to the behavior of the cobalt at the sintering time. He stated that the larger the diamond crystal size and the higher the temperature, the easier the cobalt infiltrates into the diamond powder, and cobalt flows in because of the negative pressure in the empty spaces between the diamond crystals. He observed that the abnormal grain growth of the diamond was mainly in the boundary between the diamond compact and the cobalt disc during the project experimentation.

The correlation between the temperature and the infiltration of cobalt is mainly because of the reduction of its viscosity at higher temperature allowing it to penetrate through smaller cavities decreasing the heterogeneity of its infiltration. It was also stated that by increasing the grain size of the diamond powder the flow ability of the cobalt through the diamond layer could be improved. In this test it was proven that the abnormal grain growth increased with the rise of the temperature and elongating the soak time.

Hong concluded that based on the experimental results, the abnormal grain growth can be explained by the re-crystallization process of the dissolution and precipitation of the sintered diamond into the molten cobalt, and this phenomenon was seen predominantly in the single cobalt disc test and not on the WC-Co base (Hong, 1988).

In 2009 Dr. Mukhopadhyay and Dr. Bertagnolli presented a paper where they explain the exaggerated tungsten-carbide grain growth at the diamond-carbide interface on the PDC inserts and how to control it. The presence of exaggerated tungsten-carbide grain growth at the interface is normal in the PDC inserts; they can be as long as 100 microns long with an aspect ratio of 50:1. These abnormal tungsten carbide crystals can also grow into clusters that will weaken the strength of the diamond-to-substrate bond, diminishing the performance of the PDC insert (Mukhopadhyay, 2009).

Dr. Mukhopadhayay discovered in his experimentation that the exaggerated tungsten carbide grain growth could be controlled and decreased by reducing the carbon-to-tungsten ratio. This problem can be solved by adding tungsten powder to the diamond mix or by utilizing a substrate with less cobalt percentage in its composition. By utilizing a substrate with lower cobalt content the interaction between diamond and cobalt could lower the overall carbon content (Mukhopadhayay, 2009).

In this test it was also observed that the press cycle temperature had an influence on the size of the abnormal WC grain growth. When Dr. Mukhopadhayay pressed the samples at 1500º C and 1700º C with pressure constant for both samples, he noted that the inserts that ran at a higher temperature had an overall increase in the size of the tungsten carbide clusters. Figure 2-16 shows the samples at 1500º C (left) and 1700º C (right). The difference between the cluster sizes is easily seen, the higher the temperature the larger the clusters are.

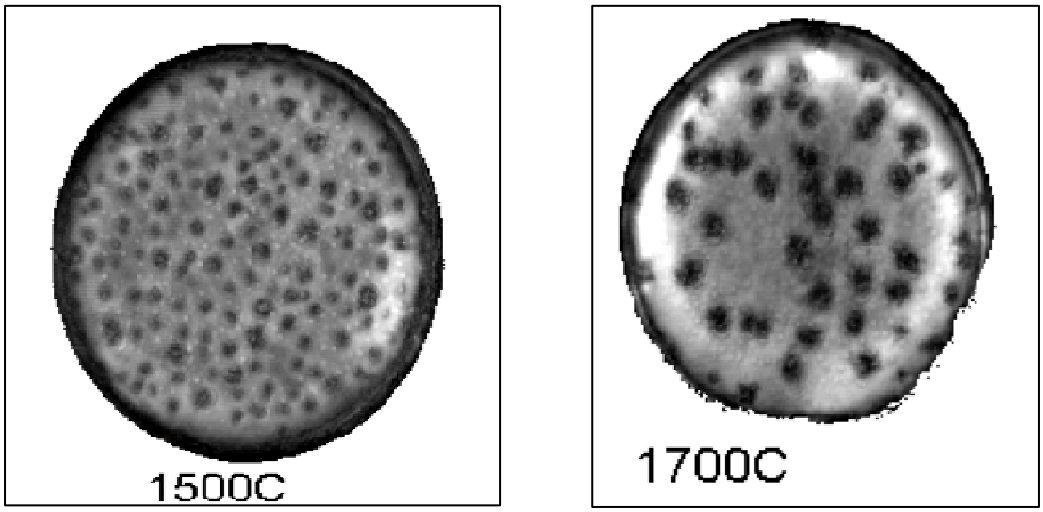

Figure 2-16: C Scan images of samples sintered at 1500°C and 1700°C at 60 kbar with 90 s cooling show increase in the exaggerated grain growth with temperature (Mukhopadhyay)

2.6 Conclusion

The literature review brought us a clear understanding of the importance that the cell components have on the diamond synthesis process. It is very important to choose the right materials to ensure that the pressure and the temperature required for the diamond nucleation is always attained with minimum variation.

We have to have a clear understanding of the diamond-graphite boundaries in the carbon phase diagram when we set up our press profiles to ensure we can arrive at the temperature necessary to get the catalyst to flow and allow the diamond crystals precipitation to happen. At the same time we must control the heat-loss by insulating the cell appropriately to ensure uniform heat and protect the inserts and the apparatus from hot spots that could end up in a process failure.

3 Process Optimization and New Design Evaluating Methods

After achieving a good understanding of the diamond synthesis process and the manufacturing procedure for polycrystalline diamond compact inserts, we can recognize the importance that the cube cell assembly components and high-pressure/high-temperature apparatus have on the completion of those processes. A volume/pressure ratio is present between the current cube assembly design and the press anvils to meet the vital internal pressure required by the sintering process. To make certain that the same product quality and performance will be attained, it is possible, however, to make changes within the established cube envelop.

3.1 Proposed Solution

With the current cell design, shown in Figure 2-9, the cube cell assembly capacity is only large enough to process two 0.400” tall inserts at each press cycle. Note that the diamond powder shrinks during the diamond-to-diamond bond growth process. To ensure the diamond thickness specified by the customer, the actual size of the insert assembly prior to the synthesis process is about 0.400” tall, but it can only yield a 0.315” tall finished insert. The size of the press chamber and the dimensions of the various components of the current cell assembly impose this limitation in sample size.

The heater, which is the key component for heat generation, has a height of 1.150”, and at each end it has a graphite disc with a height of 0.050”. There are also three isostatic material

discs (one at each end of the two inserts and one in between them) each of which is 0.085” thick. These electrically insulate and distribute hydrostatic pressure on the samples. One titanium disc 0.025” thick is placed at each end of the furnace to conduct the current from the steel rings to the heater. Because of the end heating, it is necessary to have a larger pressure media button (~ 0. 400” tall) at each end of the cell to insulate the cell heat from the face of the anvils. It is important to prevent the heat from getting to the anvils because the strength of the tungsten carbide anvils diminishes substantially once temperatures exceed 700º C.

Thus, the stack height for the cell is 2.00”, which is the allowed capacity of the pressure apparatus chamber. Figure 3-1 shows the dimensions of all the components in the current cell design. We can observe how the two inserts in the middle of the cube assembly are limited to a maximum height of ~0.400”.

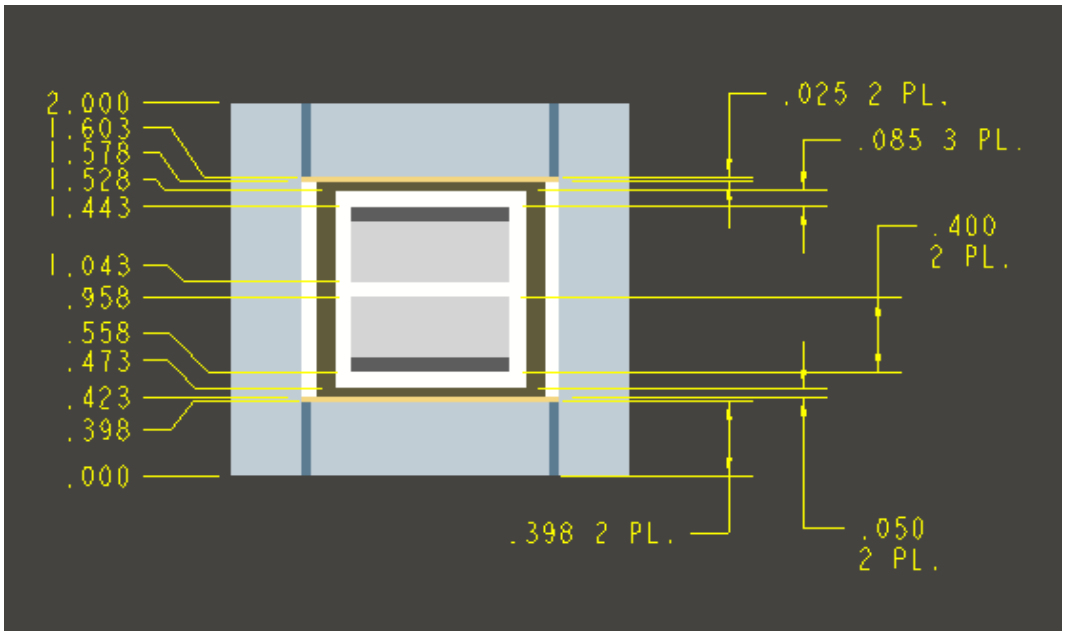

Figure 3-1: Current cell assembly component size description. It shows how the end heating limits the size of the sample to two .400” inch samples per press cycle.

Because of the capacity limitation of the existing cell design, a new cell design is proposed in this current research. The new cube/heater assembly for the cubic press must allow the current to flow from the anvils to the heater in an almost straight form. A titanium retainer ring will replace the titanium disc currently utilized to conduct the power from the steel ring to the graphite disc.

Figure 3-2 shows how the titanium retainer wraps around the outer ends of the heater assembly and makes contact with the steel rings on the other end conducting the electrical current from the anvils to the heater tube. The graphite disc will also be eliminated, and by these means, the heater assembly does not generate end heating allowing larger size samples to be fitted in the cube assembly. Utilizing this proposed design the graphite heater will generate heat at the center of the cell rather that the ends, radiating the heat outwards.

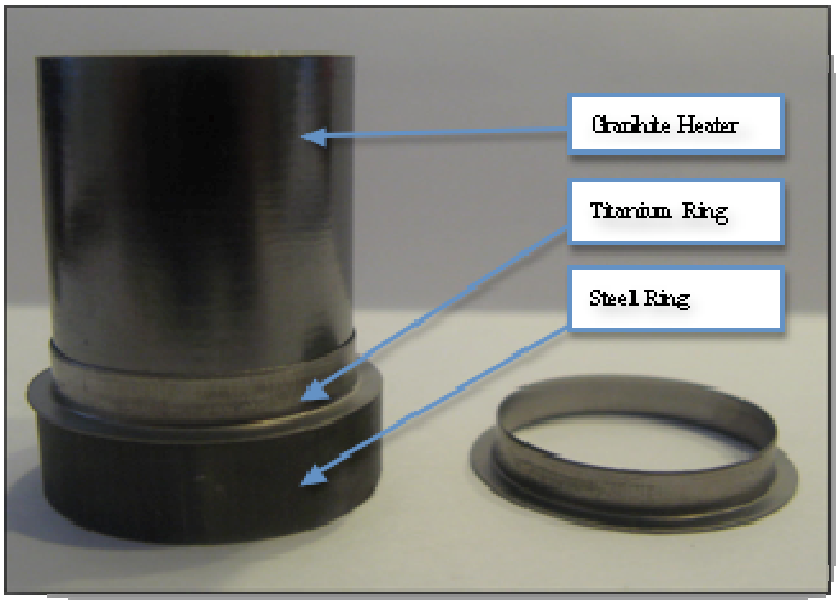

Figure 3-2: Proposed heater assembly with titanium retainer ring resting on the steel ring.

The polycrystalline diamond compact inserts will be placed diamond facing inwards where the highest temperature is generated in the new cell assembly. It is expected that the diamond synthesis will still be possible ensuring a successful bonding between the diamond crystals and the tungsten carbide (WC) substrate. The goal for the new cell design is to produce polycrystalline diamond compact inserts with the same performance as the ones manufactured with the traditional cube cell design, and allowing two 0.600" tall inserts or taller to fit in the cell assembly without jeopardizing the integrity of the high-pressure/high-temperature apparatus. Figure 3-3 shows the proposed cube cell assembly. The most evident changes in the new design compared to the current cube cell assembly are: the diamond (darkest section in the insert sample) faces inward, the titanium disc was replaced by the titanium retainer, and the graphite disc and isostatic pressure media discs were removed from the cube assembly.

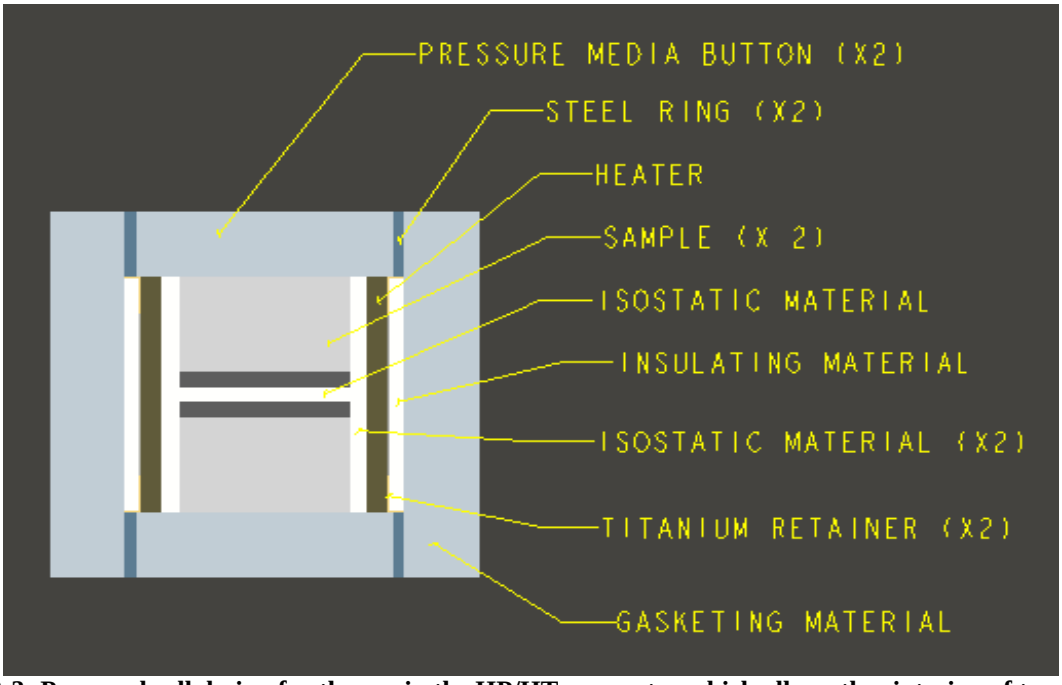

Figure 3-3: Proposed cell design for the use in the HP/HT apparatus which allows the sintering of two ~ 0.600" tall parts per press cycle.

It is proposed that the end heating be eliminated and to place the back of the polycrystalline diamond compact inserts against the pressure media buttons, which would allow extra space for taller inserts to be processed. By reducing the height of the pressure media buttons, enough space would be available to sinter up to two ~ 0.700” tall inserts per press cycle. This new insert height will allow processing the tallest standard PDC insert ordered by the customers without a need for bonding an extra substrate. Figure 3-4 shows a sample processed utilizing the proposed method compared to one using the current cube cell design.

Figure 3-4: Proposed method finished sample compared to current method finished sample without bonding process.

The dimensions of the new cell design are shown in Figure 3-5. Eliminating the end heating components will increase the space available to grow the height of the heater to 1.250” tall. This will allow for larger samples within the heated space in the cube assembly, up to 0.600”

tall maintaining a 0.085” isostatic material disc in between the samples to ensure an even pressure distribution to the samples.

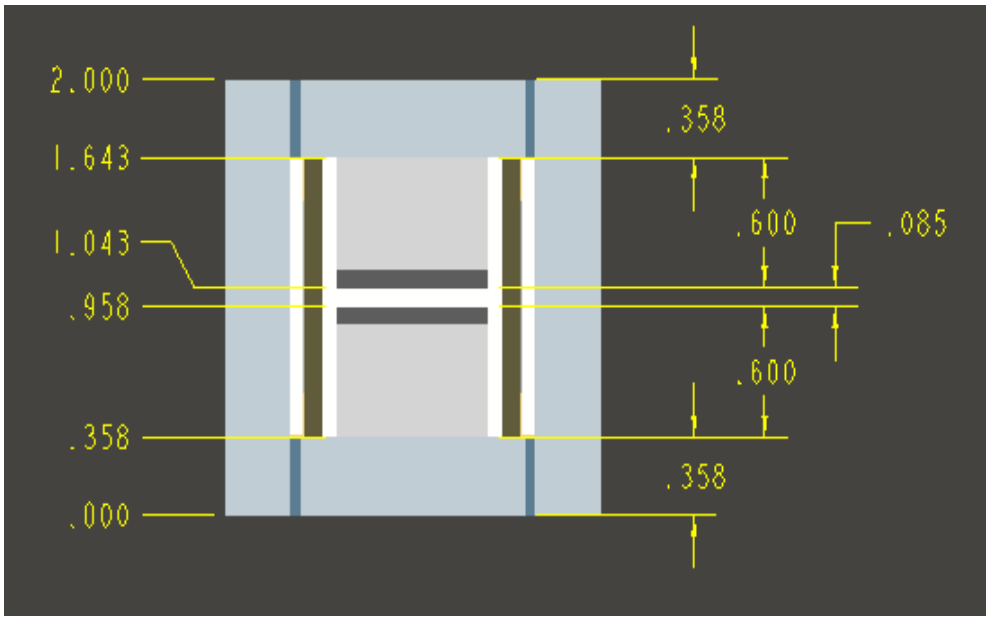

Figure 3-5: Proposed new cube cell assembly component dimensioning. This cell will allow two 0.600” inches parts per press cycle.

3.2 Testing the Impact of the New Design on the PDC Insert Production

Allowing the cube cell assembly to accept larger inserts will impact the cost efficiency of the manufacturing process of polycrystalline diamond compact inserts. This new process should reduce material and processing cost, without jeopardizing the product quality and performance. The next two sections will describe the methods utilized to demonstrate how the process was improved with the new cube cell design, and if the change of the manufacturing process negatively impacted performance of the polycrystalline diamond compact insert.

3.2.1 Process Improvement Analysis

In order to establish if the new process actually has an impact on the manufacturing of polycrystalline diamond compact inserts, a time-cost analysis will be done to compare it against the current process. Flow-time studies will be done on the current and the proposed manufacturing processes and the results will be compared to arrive at a conclusion. The process time study will be done with the same operators and the same machines for both methods to reduce variability in the data. With the same goal, a series of data points will be collected at each station and the inputs will be averaged. Figures 3-6 and 3-7 show the press and the finishing flow maps utilized to capture the cycle and touch times and the operator required for each process operation.

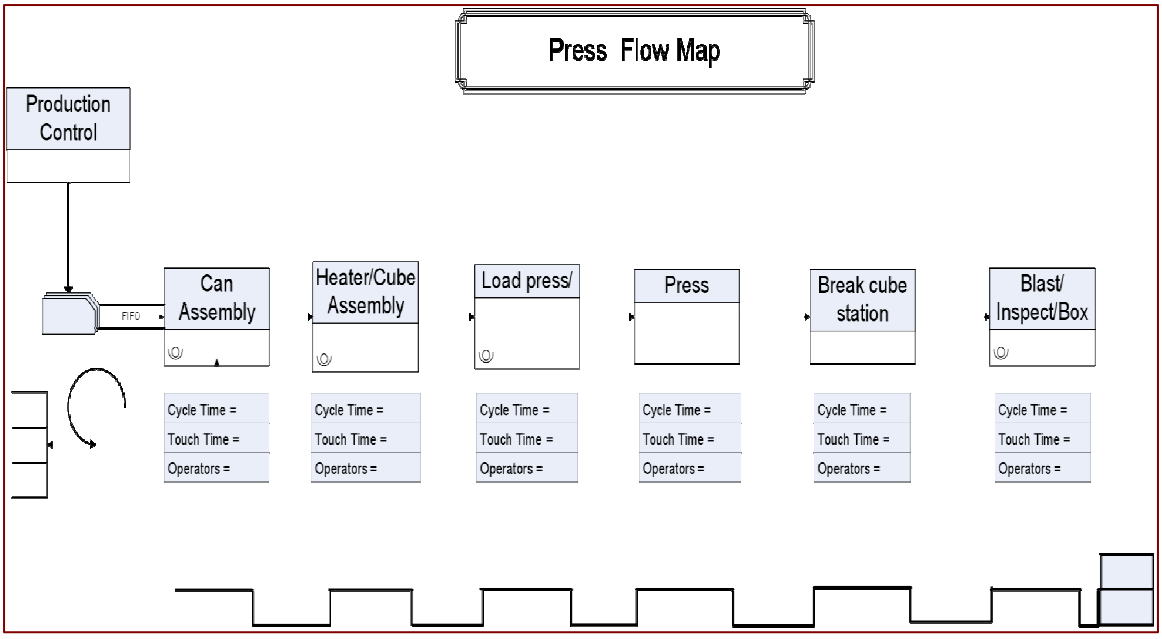

Figure 3-6: Flow map utilized to capture the processing times for the assembly and press operations.

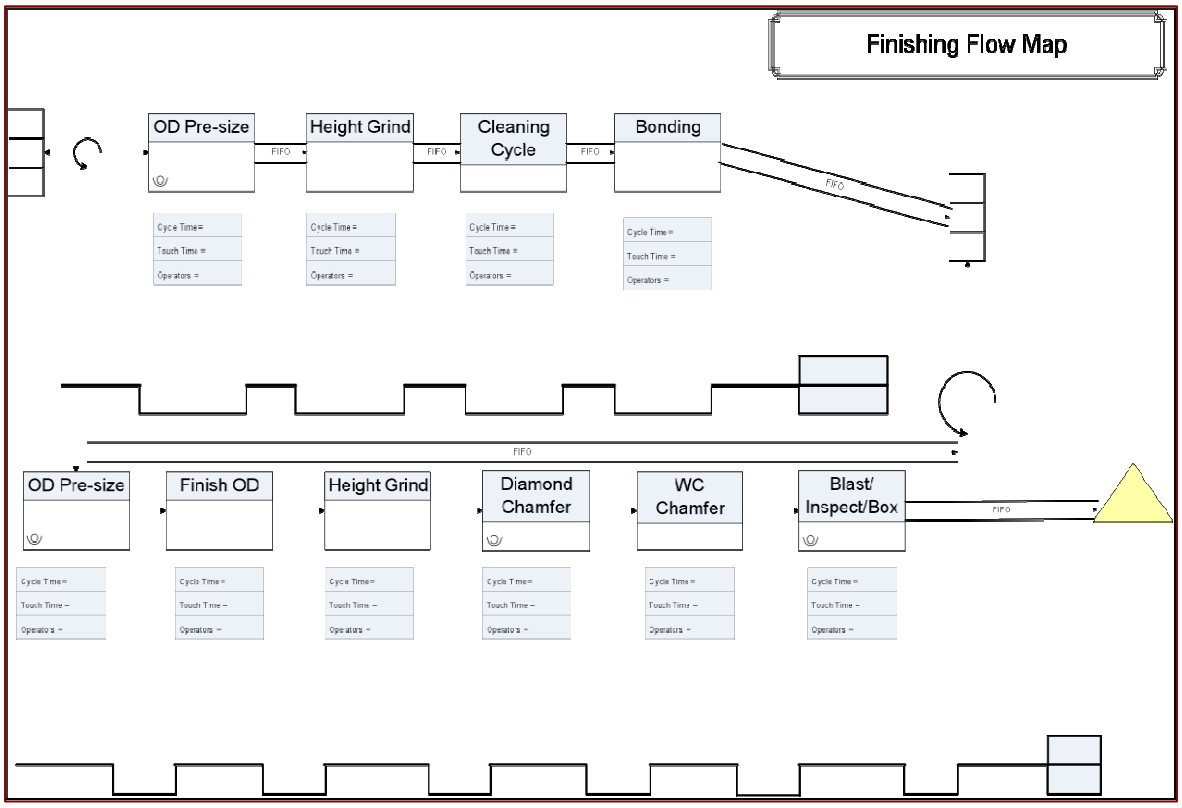

Figure 3-7: Flow map utilized to collect the processing time for the finishing operations.

3.2.2 Components Cost Analysis

Material cost for both processes will be compared by creating a list of the components utilized in the traditional manufacturing of a polycrystalline diamond compact insert which would normally require bonding in order to achieve customers' expectations. The cost per component will be itemized and added together, and the grand total for the current method will be compared against the total for the proposed method.

The press anvil life will be compared between the two cube cell assembly methods. Anvil life represents the highest cost of the pressing process, if the anvil's life is not maximized, cost per part increases relative to it. In order to acquire anvil life information, number of press cycles per press will be collected for a period of time, ensuring one press will only produce

polycrystalline diamond compact inserts utilizing the proposed cell assembly. The data from this press will be compared to the historical data for US Synthetic's press anvil life. The expectation for the proposed cube cell design is to achieve the same quantity of press cycles as the current cell between anvil failures.

3.3 Polycrystalline Diamond Compact Inserts' Performance and Composition Comparison

The performance and the composition of the polycrystalline diamond compact inserts will be tested utilizing three independent methods. The methods to be utilized in this thesis are heavy wear test, comparison of exaggerated tungsten carbide grain growth at the diamond-carbide interface, and diamond microstructure comparison.

3.3.1 Heavy Wear Test

With the end of corroborating that the wear performance of the polycrystalline diamond compact inserts made utilizing the proposed cube cell assembly does not diminish compared to the current method, a heavy wear test will be performed on groups of both samples.

The heavy wear test consists of machining a block of granite with a PDC insert. This is the most commonly used method in the polycrystalline diamond compact insert industry to test the wear resistance of the cutters. This test reproduces the extreme conditions that can be present on the oil and gas drilling applications.

The test starts by finishing a set of randomly selected PDC inserts from both cube cell assembly methods to a standard size. Once the samples are finished, a technician measures the

diamond thickness and the diamond chamfer size to ensure there are no other differences between the inserts that can affect the test result. Once the testing technicians inspect the inserts, a slit is ground on the back of the insert and a pin locks it in the testing fixture, shown in Figure 3-8. It is important to lock the sample in place to ensure it will be tested on the right position and to prevent it of rotating through the test.

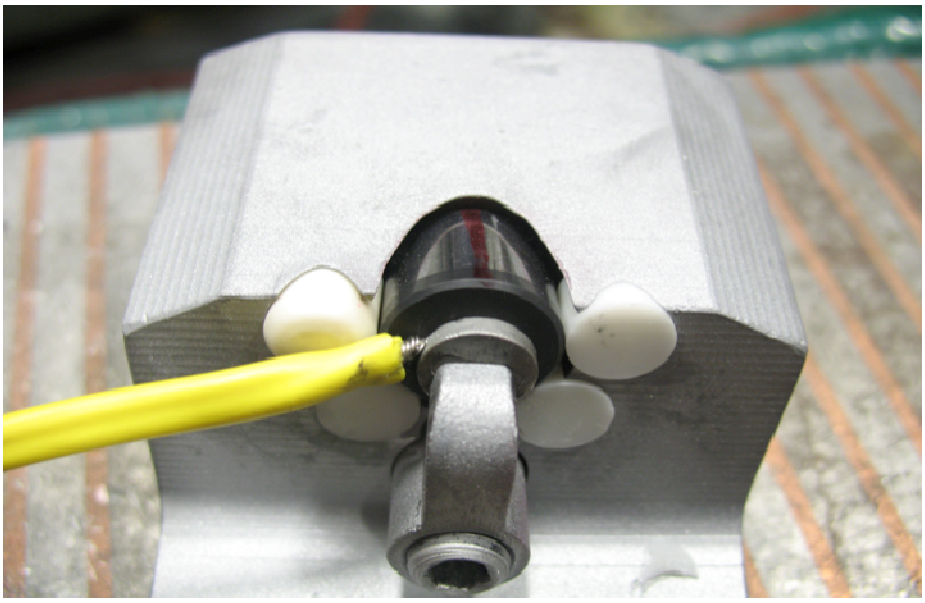

Figure 3-8: PDC insert clamped into the heavy wear test fixture.

The testing fixture is then mounted to a vertical turret lathe, as shown in Figure 3-9, and a standard set of parameters is utilized to machine a block of granite. The testing department previously qualifies the granite block to ensure repeatable, controlled results. The volume of rock removed per volume of diamond worn away is the central result utilized in this test to measure the wear resistance of the PDC insert. This wear measurement is commonly denominated as the grinding ratio (G-Ratio).

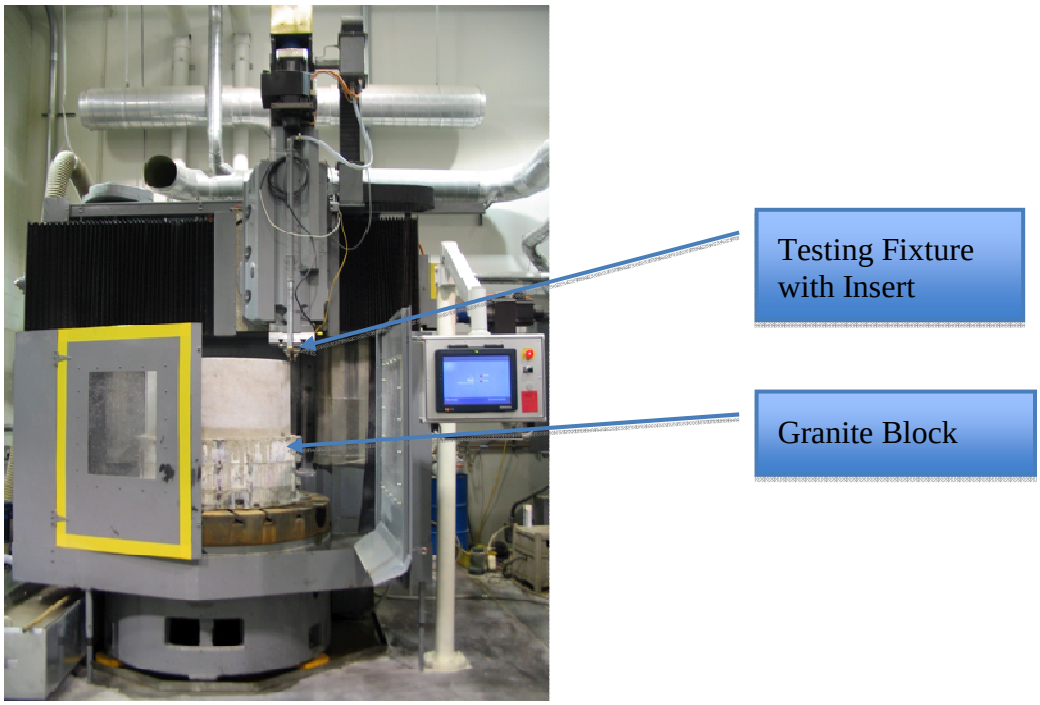

Figure 3-9: Vertical Turret Lathe utilized for testing PDC inserts

Figure 3-10 shows a PDC insert after 50 passes of grinding granite. The volume of cutter missing will be calculated and placed on the G-ratio formula to calculate the wear resistance of the insert.

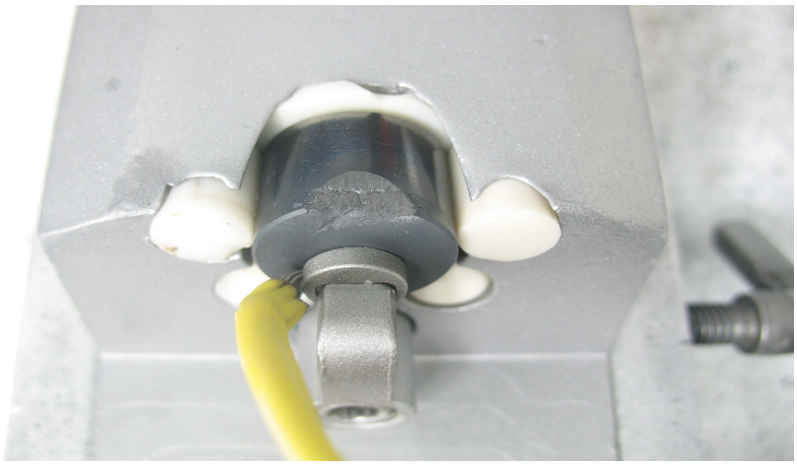

Figure 3-10: PDC insert in the testing fixture after heavy wear test.

Once the G-ratio data for both sample groups are collected, a t-test will be done to the data to compare the means of the samples. The data from the new process will also be compared with the historical data for the product using the current cube cell design.

3.3.2 Exaggerated WC Grain Growth Comparison

A randomly chosen group of finished polycrystalline diamond compact inserts made utilizing both manufacturing methods will be imaged acoustically from the top surface of the diamond. The images reveal the extent of exaggerate tungsten carbide grain growth occurring at the interface. To capture the surface micro-image at the diamond-WC substrate interface, a Sonoscan D-6000C-mode scanning acoustic microscope (C-SAM), as shown in Figure 3-11, was utilized.

Figure 3-11: Sonoscan D-6000 C-mode scanning acoustic microscope.

Once the images are taken, they will be printed and distributed to four inspectors to visually count the occurrences of exaggerated tungsten carbide grain growth present on each insert. Then all the sample data points will be added and both sample groups will be compared utilizing a t-test.

With this test we will determine if the proposed cube cell design has a negative effect on the sintering process. If we recall section 2.5 on the previous chapter, the exaggerated WC grain growth at the interface of the polycrystalline diamond compact inserts weakens the bond between diamond and the WC-Co substrate. Therefore, by comparing the two samples groups we will show if the proposed cube cell assembly does or does not increase the amount of exaggerated WC grain growth on the PDC inserts.

3.3.3 Microstructure Analysis

Two samples with the same product characteristics from each manufacturing process will be randomly selected for microstructure analysis. In this test we will compare the grain size and the diamond layer composition in different zones within the diamond layer of the inserts. The goal for this test is to identify there is any difference in the microstructure of the diamond layer of the inserts manufactured utilizing the current and the proposed cube cell assemblies.

The samples will be EDM cut in the middle of the cutter to expose the diamond layer and mounted in a Bakelite fixture. The mounted sample will be polished with a diamond compound wheel to achieve a mirror finish necessary for the x-ray material analysis. The polished samples will be analyzed utilizing a scanning electron microscope (SEM) Philips XL30 S-Feg, shown in Figure 3-12. The diamond crystal size and the material elements analysis data

will be compared between the samples to determine if there is a significant difference between the samples made utilizing the current process and the ones made with the proposed one.

Figure 3-12: Scanning Electron Microscope Phillips XL30 S-Feg.

Figure 3-13 shows an example of a micrograph of a polished sample taken at 800x magnification. The black objects in the micrograph are the diamond crystals and the lighter filler between the crystals is the catalyst residual metal.

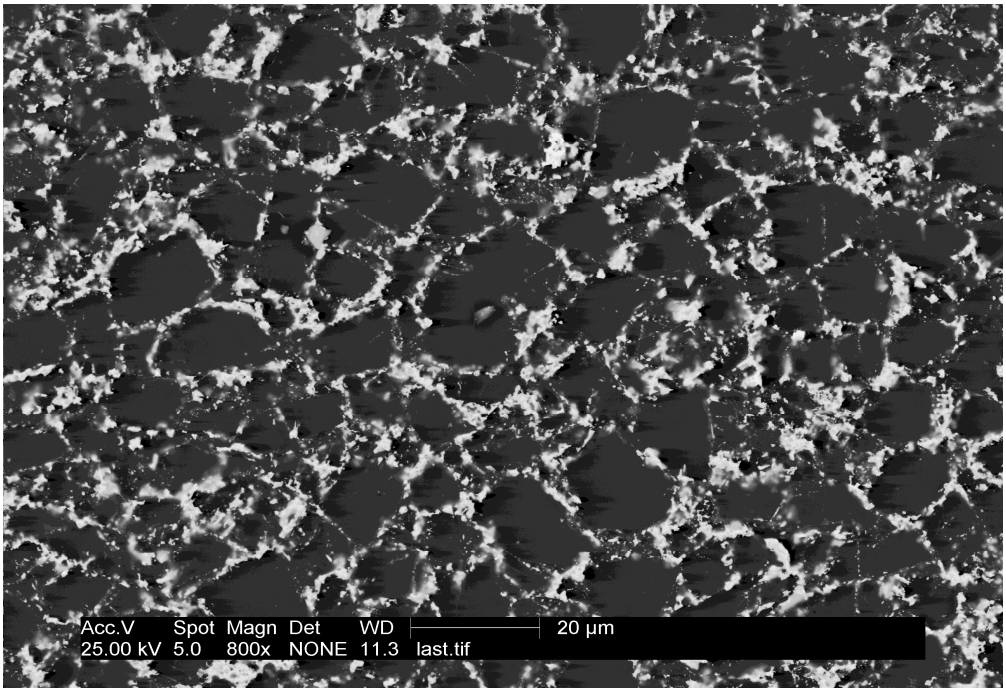

Figure 3-12: SEM micrograph of diamond layer of a PDC insert at 800x magnification

3.4 Conclusion

All the tests explained through this chapter will be put in place with the goal of supporting the implementation of the proposed method to improve the manufacturing process utilizing a high-pressure/high-temperature cubic apparatus. Process cost reduction is the objective of the new cube cell assembly, but it is necessary to maintain insert performance in order for the new process to be implemented in production.

In Chapter 4 the data captured through each analysis will be presented, and in Chapter 5 we will discuss our analysis conclusions and the recommendations for further research.

4 Results

4.1 Introduction

The objective of this thesis is to demonstrate that modifications can be made to the current cube cell assembly for the high-pressure/high-temperature cubic press that allow larger inserts to be processed. By increasing the capacity of the cube cell assembly it is possible to reduce the amount of materials required for the manufacturing of Polycrystalline Diamond Compact inserts and the number of processing operations necessary to achieve the customers' requirements. This reduction translates into lower manufacturing costs thereby improving the competitive advantage of the manufacturers.

With the purpose of supporting this thesis, two hypotheses were put in place, and in this chapter we are going to present the pertinent data to prove them right or wrong.

The first hypothesis stated that it is possible to design a new cube cell design for the high-pressure/high-temperature apparatus, which will allow larger samples to be run in the cube assembly by eliminating the end heating. The outcome of this hypothesis would be the reduction of manufacturing processes, therefore reduction of the total processing time of the finish goods, freeing-up operators and equipment, and the minimization of the materials utilized in the current manufacturing processes.

The second hypothesis stated that this new cube cell assembly design would not negatively affect the performance of the polycrystalline diamond compact inserts. Thus, through

this chapter we are going to present the data obtained through internal testing that will prove this hypothesis true or false.

The processes utilized to capture and analyze the data were presented in Chapter 3. The tests were effectuated following the pre-stipulated parameters and the same test was performed in both parts randomly to ensure non-biased results.

4.2 Processing Time Comparison

Processing time comparison on the current and the proposed method was done to prove whether or not there is a benefit for the manufacturers in the proposed design. Processing time information was captured utilizing process flow maps for each of the main two manufacturing operations: Press and Finishing.

Figure 3-6 shows the flow map for the press process, which includes can assembly, heater and cube assembly, loading and cube breaking, press, blasting/ inspecting, and lapping. The times for each of these operations were captured for a period of time and utilizing different operators, then an average of the times was assigned to each individual operation.

Figure 3-7 shows the finishing operation flow map, which includes the operations required to bond the extension substrate to the insert. Those operations are O.D. grind pre-size, height grind, cleaning cycle, and bonding. These are followed by the finishing operations which are pre-size and finish O.D. grind, height grind, diamond chamfer, WC chamfer, and blast/inspect and box the finished polycrystalline diamond compact inserts.

Each of these time studies was done for the current and the proposed manufacturing methods. The data were then analyzed and compared. Table 4-1 shows the data compiled for each of the operations of the current manufacturing method with a total of 100%. The proposed

method's operation times were expressed as proportions of the total manufacturing time of the current method.

Table 4-1: Processing time study comparison between current and proposed cube cell designs.

Processing Time Per Part Comparison by Process (in Percentage)		
Process	**Current Design**	**Proposed Design**
Can Assembly	5.20%	5.20%
Heater Assembly	2.60%	1.56%
Cube Assembly	3.38%	1.04%
Load/Break cube	4.16%	4.16%
Press	22.36%	22.36%
Blast & Inspect	4.68%	4.68%
Lapping	23.91%	23.91%
Bonding Pre-Size	13.00%	0.00%
Bonding	5.72%	0.00%
OD Pre-size	3.12%	4.16%
OD Finish	1.48%	1.48%
Height Grind	1.56%	1.04%
PC Chamfer	1.56%	1.56%
WC Chamfer	1.04%	1.04%
Blast/Inspect	6.24%	6.24%
Total Time	100.00%	78.42%

Table 4-1illustrates the difference in processing time between the current and the proposed design is 21.58%. These timesavings are distributed though the whole process, but bonding is the largest portion. When we did the time study comparison between only the finishing operations for the two methods, shown in Table 4-2, it was observed that the proposed method took only 46.03% of the time that the current method takes.

Table 4-2: Finishing processing time study comparison between current and proposed cube cell designs.

Finishing Time Per Part Comparison by Process (in Percentage)		
Process	**Current Design**	**Proposed Design**
Bonding Pre-Size	38.55%	0.00%
Bonding	16.96%	0.00%
OD Pre-size	9.25%	12.34%
OD Finish	4.39%	4.39%
Height Grind	4.63%	3.08%
PC Chamfer	4.63%	4.63%
WC Chamfer	3.08%	3.08%
Blast/Inspect	18.50%	18.50%
Total Finishing Time	100.00%	46.03%

The time reduction is not the only benefit for the manufacturing plant. By eliminating the bonding process, it also eliminates the need for four machines required for the pre-sizing of the insert and the bonding process. These four machines represent an initial investment of approximately US $500,000 that can be utilized in other equipment improvements. It also frees-up two operators per manufacturing cell, which can be relocated to support other areas or be utilized to create a new manufacturing cell if demands require. By reducing these operations, the possibility of producing defective parts is reduced as well, and scrap has a direct impact on manufacturing margins.

4.3 Material Cost Reduction

To identify if there is a difference in the material cost of the two polycrystalline diamond compact insert manufacturing methods, lists of materials utilized in each of the methods were made and the data compiled to create Table 4.3. The material cost of the current cell design was

totalized as 100% of the material cost, and then the cost of the materials utilized on the proposed method is reflected on the table as a proportion of the total cost of the current cell design.

Table 4-3 shows the cost of the materials utilized in the manufacturing of the polycrystalline diamond compact inserts. The data are represented as a proportion of the total cost of the materials required for the current method. We can observe that it would only cost 72.42% of the total cost of the current method to manufacture polycrystalline compact diamond inserts utilizing the proposed method.

Table 4-3: Material cost per part comparison by process in proportion to the total cost of the current manufacturing method.

Material Cost Per Part Comparison by Process (in Percentage)		
Process	**Current Design**	**Proposed Design**
Can Assembly	59.53%	60.59%
Heater Assembly	6.34%	6.31%
Cube Assembly	11.59%	5.52%
Bonding	22.54%	0.00%
Total Cost	100.00%	72.42%

The largest difference in cost is from the bonding process, where we can observe that 22.54% of the total cost is allocated. The other 5.04% corresponds to the elimination of the component utilized on the end-heating of the cube cell. We can also observe that the cost for the proposed can assembly is higher, and it is mainly because we increased the height of the tungsten-carbide substrate. When we compare this increase in price to the price of a second tungsten-carbide insert necessary to increase the height of the polycrystalline diamond compact insert and the bonding braze, it becomes insignificant.

Another important cost that concerned us in the implementation of the proposed cube cell design was the anvil life. At a current rate of 7500 average runs per anvil, and a cost of 3,000 US dollars per unit, it represents a fixed cost in the manufacturing of polycrystalline diamond compact inserts. So if we reduce the anvil life, then we will directly increase the manufacturing cost of the insert. But it is not only the cost of the anvils; it is also the downtime of production and the cost of the maintenance technician labor every time an anvil failure event occurs. In our test, we utilized two presses to run only the proposed method, and we are currently counting 9500 press cycles on each of them without any anvil failure. At this point there is not enough data to support the idea that the proposed cell design increases the average life of the anvils, but it can be concluded that the proposed cube cell design does not negatively affect the anvil life.

4.4 Insert Performance Test

To prove or reject the second hypothesis, which stated that the new cube cell design for use in the high-pressure/ high-temperature apparatus does not negatively affect the performance of the polycrystalline diamond compact insert, the following test was made. As explained in Chapter 3, we utilized the heavy wear test, which is the most commonly used wear resistance test in the PDC industry, to compare the performance of the proposed method against the current one. We also compared the exaggerated tungsten growth at the interface, and we did a microstructure analysis of random samples from both methods to observe and establish if there is a structural difference between them.

4.4.1 Heavy Wear Test

For each of the experiments done on the implementation of the proposed cube cell design, a set of random samples was collected to be tested on the heavy wear test (commonly called the VTL test). The wet VTL test was designed to reproduce the worst drilling conditions that a PDC insert will encounter in the field. The to-be-tested samples from the proposed process were made at the same time as a set of standard samples made with the current process. The current and proposed samples were made on the same press by the same operator to reduce the variables between them and to isolate any difference in performance between the samples down to one factor: manufacturing method.

Once the samples were collected, a testing technician at US Synthetic measured the inserts to ensure that all the samples tested had similar dimensions to reduce possible variations. After inspection, the inserts were mounted on a testing fixture and run on the VTL test utilizing the parameters shown on Table 4-4. The granite block was previously tested with a standard cutter to set a base line for the test and to capture the possible differences between granite blocks.

Table 4-4: Heavy wear test parameters for the wear resistance test.

Test	Depth of Cut (mm)	Infeed (mm/rev)	RPM	Surface Speed (m/s)	Coolant
Heavy Wear Resistance (Wet VTL Test)	0.254	6.35	101	Variable	On

After testing the samples for 50 passes on the wet VTL test, the amount of diamond worn from the PDC insert and the amount of rock removed were calculated. In Figure 4-1 we can observe the wear on the insert after the wet test was completed. Once the data from the test were

compiled the G-Ratio was calculated for each insert and added to a database for further analysis. The G-Ratio is calculated by dividing the amount of rock removed by the amount of diamond worn from the PDC insert, therefore, the higher the G-Ratio the higher the wear resistance of the insert.

Figure 4-1: Polycrystalline diamond compact inserts after 50 passes on the wet VTL test.

Having the G-Ratios compiled for samples, we selected the data from the samples made with the same tungsten-carbide grade and the same diamond particle size. Depending on the application, different grades of WC substrates and different particle sizes of diamond are combined to obtain an optimal PDC insert for the application. For our test we used samples made with substrates with 13% cobalt binder and a 2µ to 20µ average diamond particle size.

Minitab 15 was for utilized data analysis. The G-Ratio information was extracted from the database and imported to Minitab's worksheet. The sample groups were first tested for normality as shown in Figures 4-2 and 4-3. Both sample groups showed normal distributions.

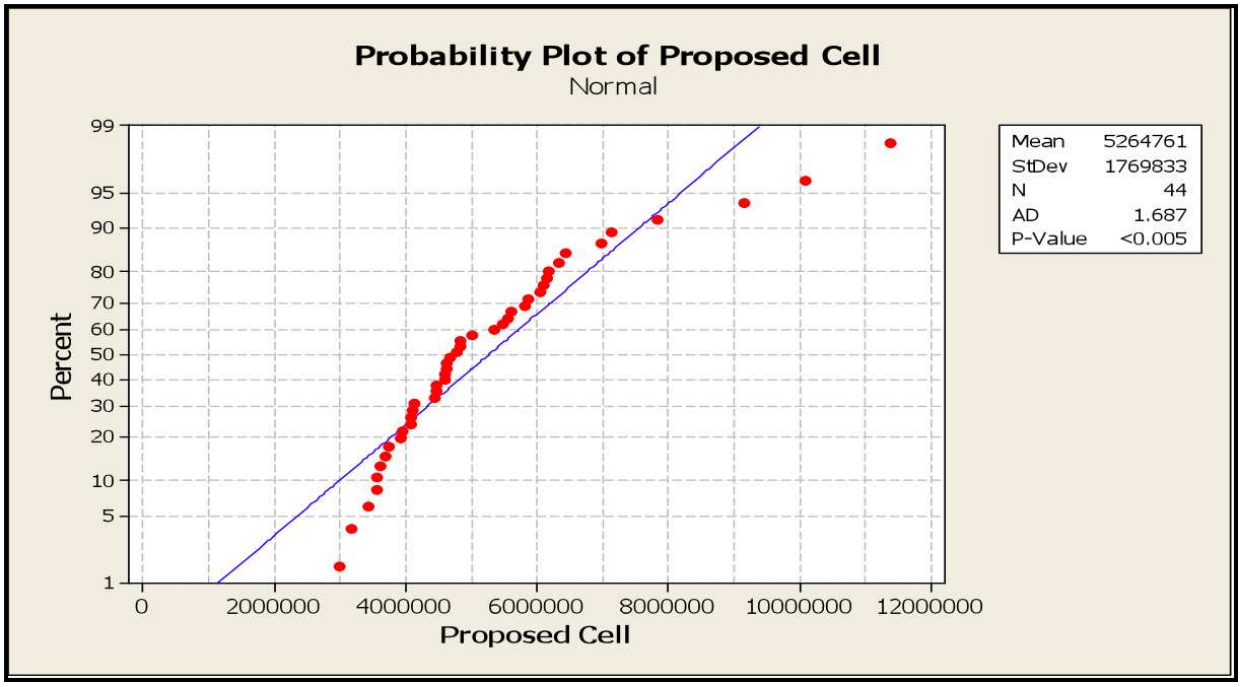

Figure 4-2: Normality test on the proposed method's G-Ratio data

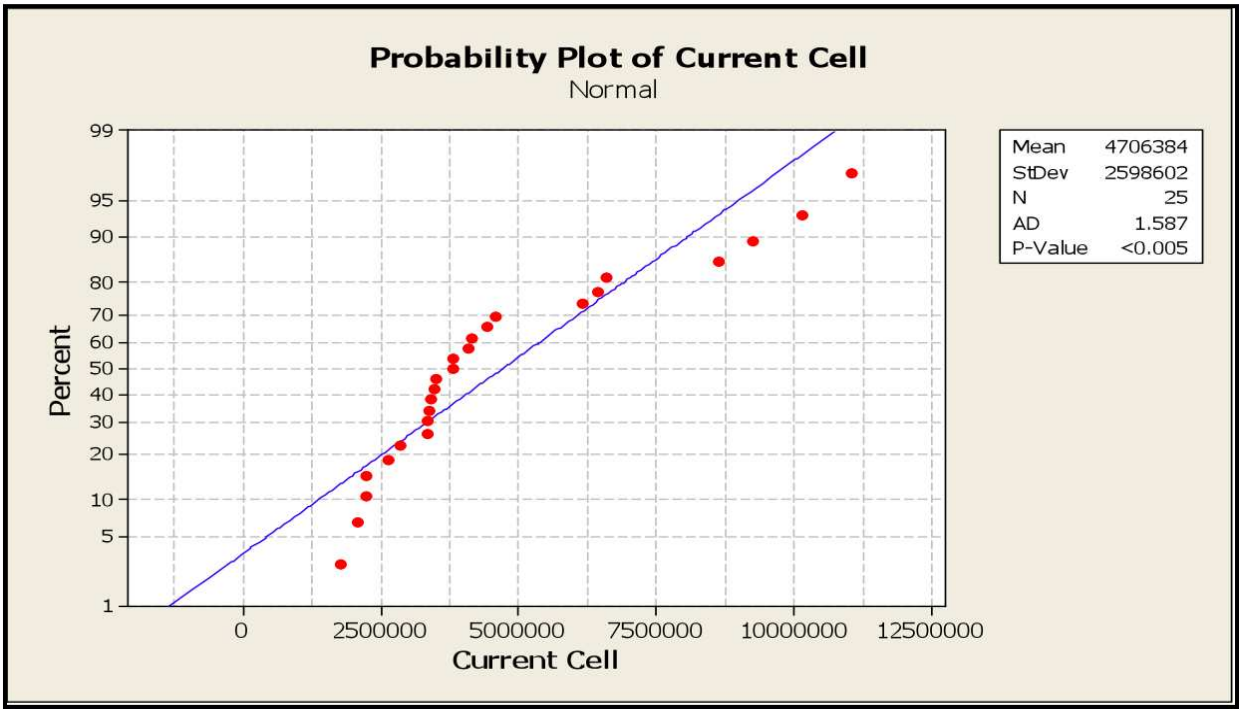

Figure 4-3: Normality test on the current method's G-Ratio data, both sample groups wrap around the normal slop line denoted in blue.

After the data were proven normal, a two-sample t-test was done on the data to show if the mean of the proposed method G-Ratios was less that the mean of those of the current method. If this theory was proven true, then the wear resistance performance of the proposed method inserts was inferior to that of the current method inserts.

Figure 4-4 shows the 2-sample t-test box plot of the results of the comparison between the G-Ratio means for the proposed method inserts compared to the current method inserts G-Ratios. On this test we have a 95% confidence level that the mean of the proposed cube cell design G-Ratio's are not less than the mean from current method samples. This results support the truthfulness of the second hypothesis of this thesis.

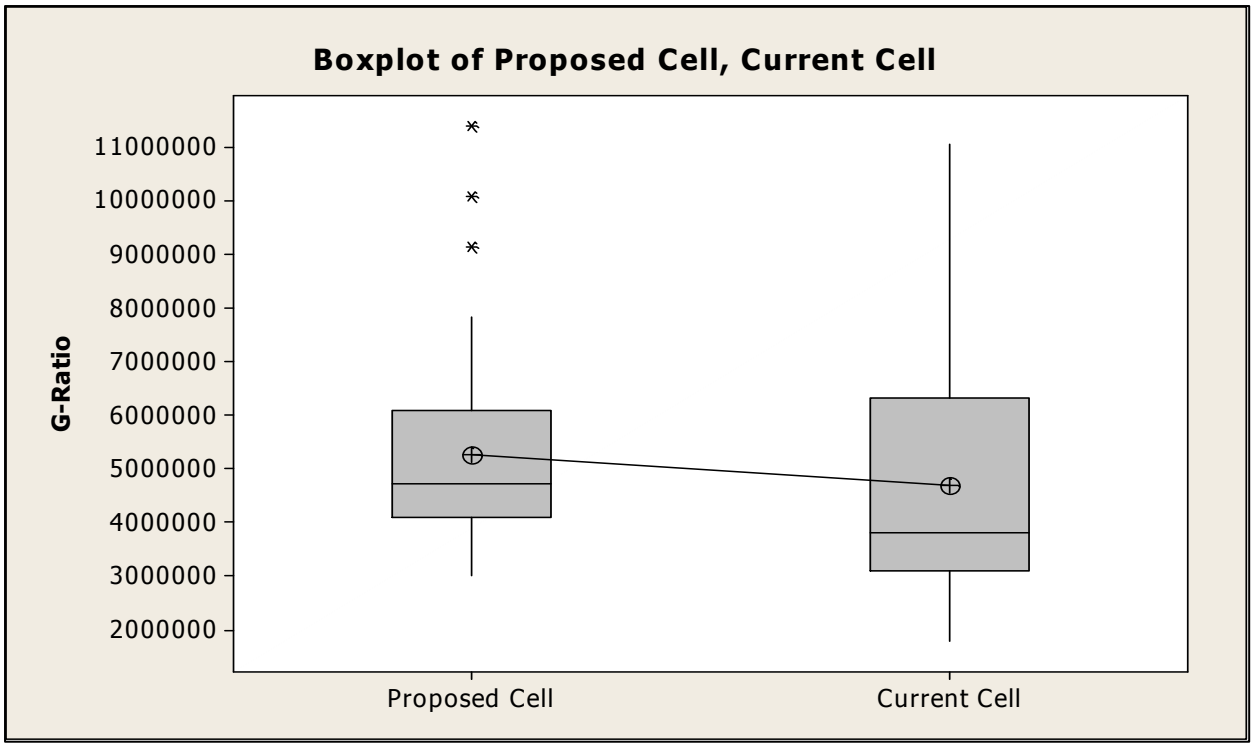

Figure 4-4: Boxplot of the 2-sample t-test on the G-Ratio data of the proposed compared to the current method.

It was also plotted the individual G-Ratio values for each of the samples to compare the scatter of the data. We can observe in Figure 4-5 that the G-Ratio data for the proposed method are grouped closely together, which can be interpreted as higher consistency of the product.

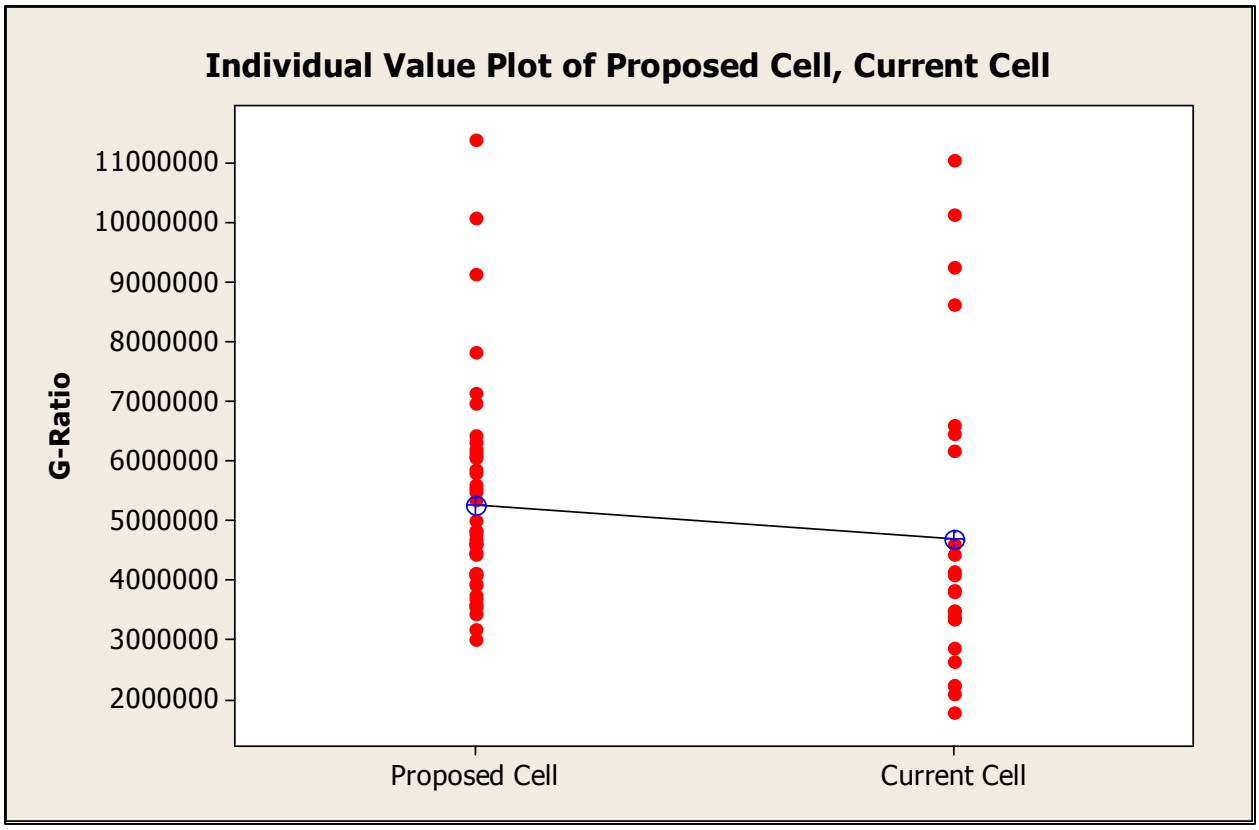

Figure 4-5: Individual value plot for the G-Ratio data from the proposed and the current manufacturing methods

From previous tests at US Synthetic we understand that different chamfer sizes and diamond layer thicknesses have an impact on the insert performance in the wet VTL test. So we did a t-test on the chamfer size data shown in Figure 4-6 and on the diamond layer thickness shown on Figure 4-7 to evaluate if they were not equal and if that might have affected the sample G-Ratio distributions.

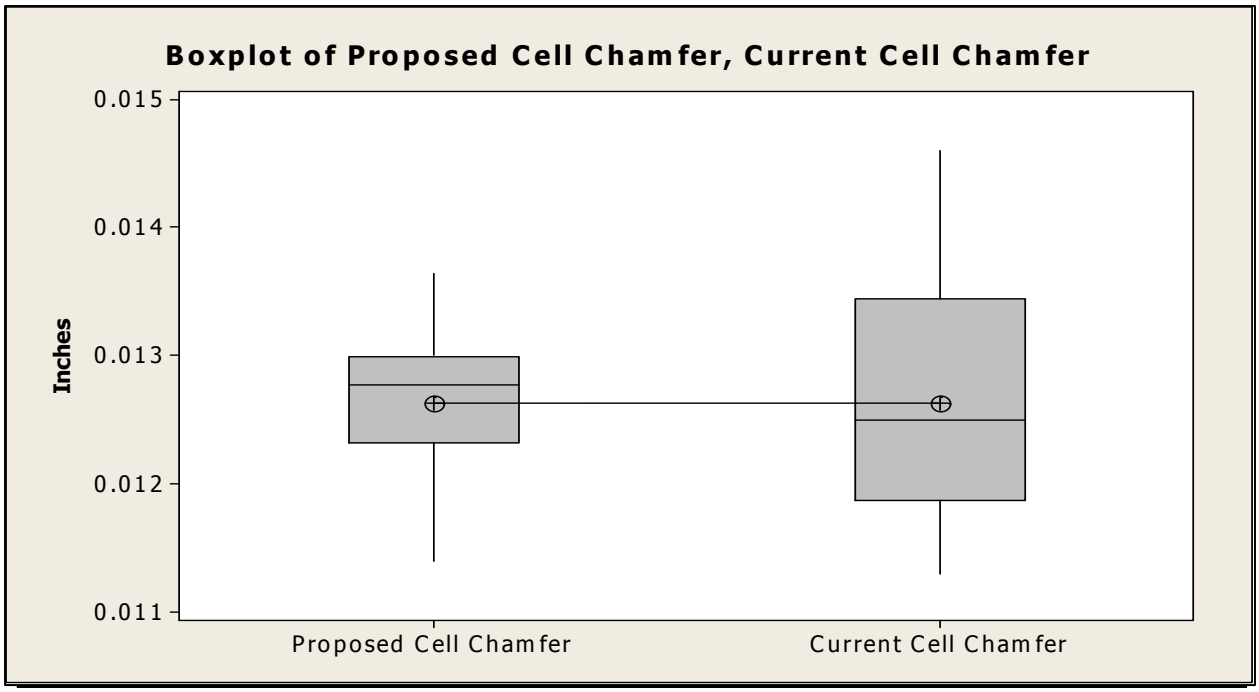

Figure 4-6: Boxplot of the diamond chamfer size of the proposed cell compared to the current cell data.

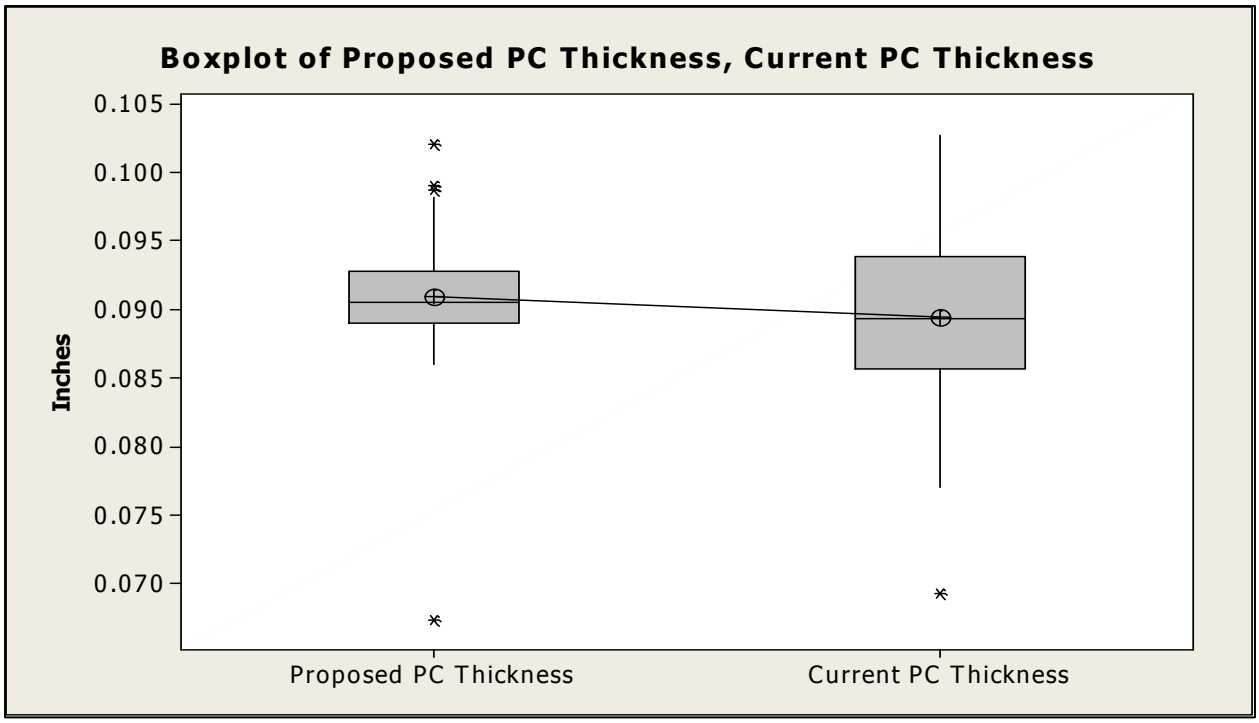

Figure 4-7: Boxplot of the diamond layer thickness of the proposed method samples compared to the current method ones.

As it be can observed in both boxplots, the measurements for the proposed method's samples were not significantly different from those of the current method. It does not appear that variations in chamfer size or diamond layer thickness were influencing the result of the G-Ratio 2-sample t-test.

In order to corroborate the information obtained with the initial G-Ratio 2-sample t-test, it was decided to compare the G-ratio data from the proposed method against the historic US Synthetic's G-Ratio data. US Synthetic's G-Ratio data is currently collected as a method to qualify the granite rock utilized in the heavy wear test. After verifying that the historic data were normal, we did a 2-sample t-test, where once again with a 95% confidence level we can determine that the mean from the proposed method's G-Ratio it is not less that the one from the historic data. In Figure 4-8 we can observe that the data distribution and standard deviation for both sample groups are shown to be similar.

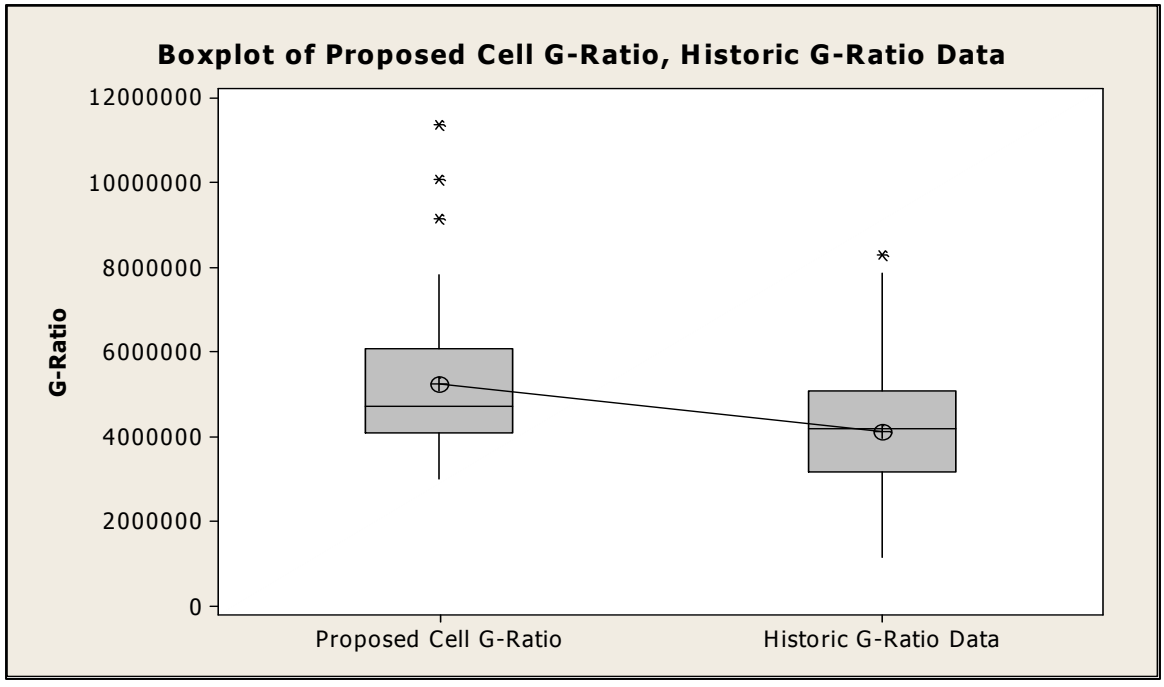

Figure 4-8: Boxplot of the G-Ratio data for the proposed and US Synthetic's historic current methods.

After comparing the G-Ratio data generated from inserts utilizing the proposed and the current method, we can conclude that the wear resistance performance of the polycrystalline diamond compact inserts does not diminish when manufactured with the proposed cube cell assembly.

4.4.2 Exaggerated Tungsten-Carbide Grain Growth at the Diamond-Carbide Interface

As described in Chapter 2, Section 2.5, the exaggerated tungsten-carbide grain growth at the diamond-WC substrate interface is common in polycrystalline diamond compact inserts. If these exaggerated agglomerations grow into clusters, it can reduce the strength of the diamond-to- substrate bond, diminishing the performance of the PDC insert in the field (Mukhopadhyay, 2009).

Because we are changing the heat pattern and possibly the pressure with the proposed cube cell assembly, we believe it is important to verify if the new method has an influence on the exaggerated grain growth of tungsten-carbide grain at the diamond-substrate interface.

In his research work, Dr. Mukhopadhyay noticed that at higher temperatures, while maintaining the pressure constant, the size of the tungsten-carbide exaggerated crystal grain growth increase in size. Figure 2-15 shows C-Scan images of samples sintered at 1500º and 1700º Celsius, and we can visually perceive the difference in these grain sizes from one sample to the other.

To demonstrate if the proposed cube cell assembly could have an impact on the exaggerated tungsten-carbide grain growth, we designed the following test.

Ten random samples from each of the two manufacturing methods were imaged acoustically from the top surface of the diamond. To capture the surface micro-image at the diamond-WC substrate interface, as shown in Figure 4-9, a Sonoscan D-6000 C-mode scanning acoustic microscope (C-SAM), was used. Copies of the images were randomized and distributed to four of US Synthetic's quality technicians.

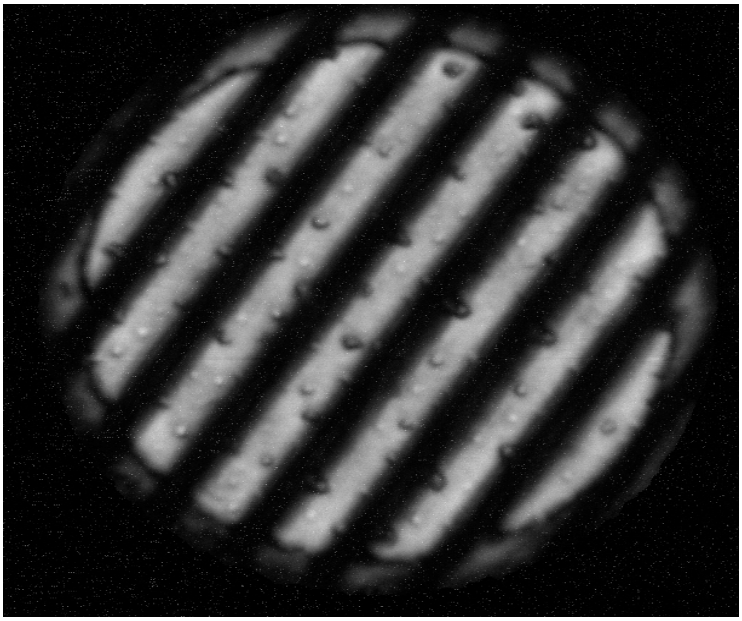

Figure 4-9: Sonoscan image of the diamond-substrate interface of an insert manufactured utilizing the current method. The darker spots on the lighter bands are the exaggerated WC grain growth.

Each of the quality technicians did a visual count of the exaggerated grain growth per sample and entered the data on the corresponding slot in a spreadsheet. The data were then uploaded into a Minitab 15 worksheet for further analysis. After verifying the data samples were normally distributed, a 2-sample- t-test was done. The goal of this t-test was to identify if the grain-growth mean of the proposed method was statistically greater than the one from the current method. If the previous statement is true, it would indicate that the proposed method might

facilitate the growth of the exaggerated WC agglomerations at the interface, thus it could diminish the performance of the PDC insert.

The result from the two-sample t-test showed that it was not possible to statistically differentiate the samples of one process from those of the other. Even though this test was subjective, because the technicians' judgments were required to determine the amount of exaggerated WC grain growth present on each sample, by averaging the results we believe the error in the data was reduced and the data became more objective.

In Figure 4-10 we can observe the two sample distributions on a Boxplot. It shows that the mean for the proposed method's sample data was higher that the one for the current method's data. This means that on the average, the proposed method has more counts of exaggerated WC grain growth.

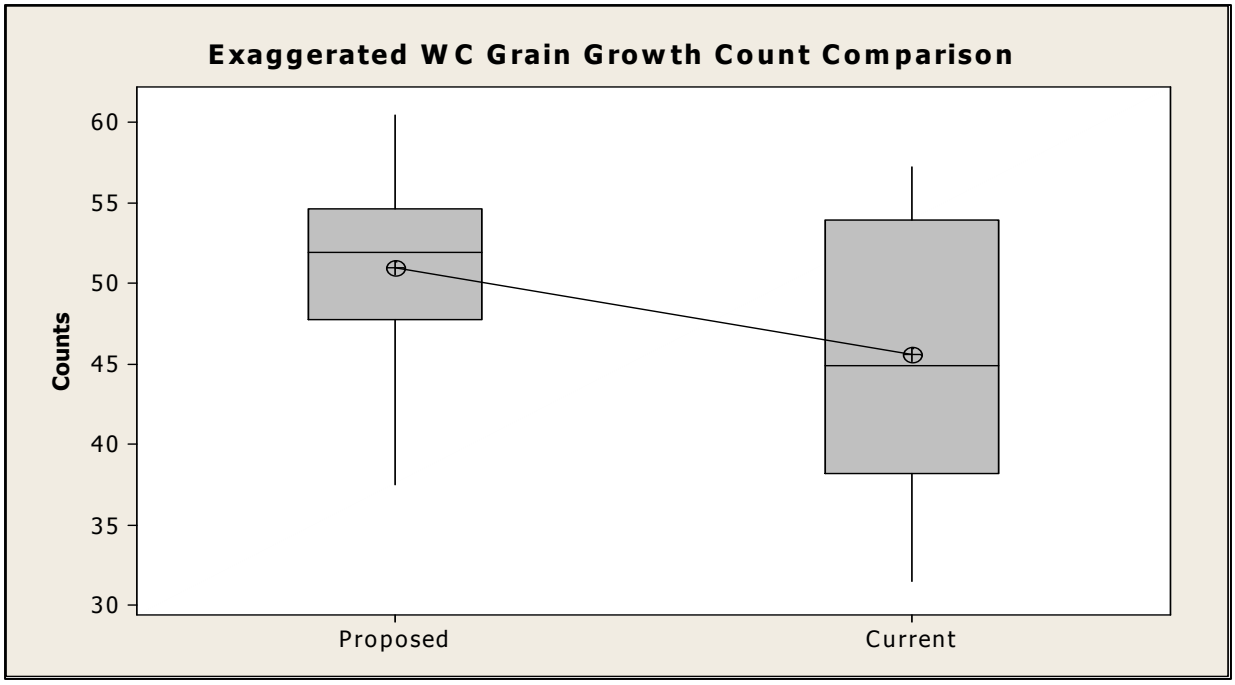

Figure 4-10: Boxplot of the sample data of the exaggerated WC grain growth agglomerations collected from C-Scan images of the diamond-substrate interface of samples made with the proposed and the current method.

However, when the technicians were interviewed they commented that the agglomerations on some of the images appeared larger than in others, and when the data were sorted the current method was identified to be the one with the apparent larger exaggerated tungsten-carbide grain growth. This difference between the samples can be compared in Figure 4-11, where a Sonoscan image of the interface of a proposed sample (left) is compared with one of the current process (right).

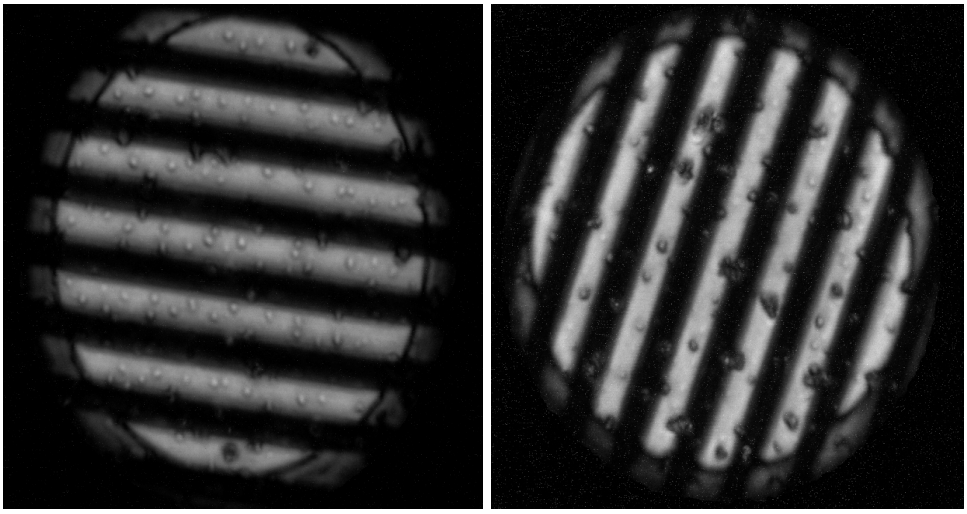

Figure 4-11: Sonoscan images of proposed (left) and a current (right) inserts, we can observed that the exaggerated WC grain growth on the current sample appears to be larger than the ones on the proposed one.

So it can be concluded that with the current data it can not be statistically identified a difference between the amounts exaggerated tungsten-carbide grain growth in the samples from the two manufacturing methods. Thus we cannot claim that the non-end-heating cube cell assembly proposed in this thesis could diminish the performance of the polycrystalline diamond compact insert.

4.4.3 Microstructure Comparison

The objective of this section is to compare the diamond crystal grains and material composition of the polycrystalline diamond compact inserts manufactured utilizing two different cell designs. By comparing the diamond crystal size and the material analysis of the samples from the two cube cell designs, it can determined if one of the cube assembly methods leads to a difference significant enough to be quantified in the structure of the PDC inserts.

The consistency in the diamond structure is important to ensure that polycrystalline diamond compact inserts will perform as expected. By changing the diamond crystals' size or the amount of cobalt content in the diamond layer, the performance of the PDC insert can be proportionally altered. Increasing the cobalt content in the diamond layer provides toughness to the structure, but decreases its thermal-stability as well. Reducing the average crystal size of the diamond layer increases the abrasion resistance of the insert, but it also makes it less resistant to impact fractures.

In order to prove a difference between the two cube cell methods, images from the samples at the diamond-substrate interface, at 500 μm from the top edge and 500μm from the outer edges, and from the middle of the sample were taken.

The samples were EDM cut perpendicularly to the diamond surface, mounted on a Bakelite fixture, and polished using an Ewag diamond polisher with diamond wheel. A strip of copper tape, as shown in Figure 4-12, was applied over the sample to reduce charging at high vacuum.

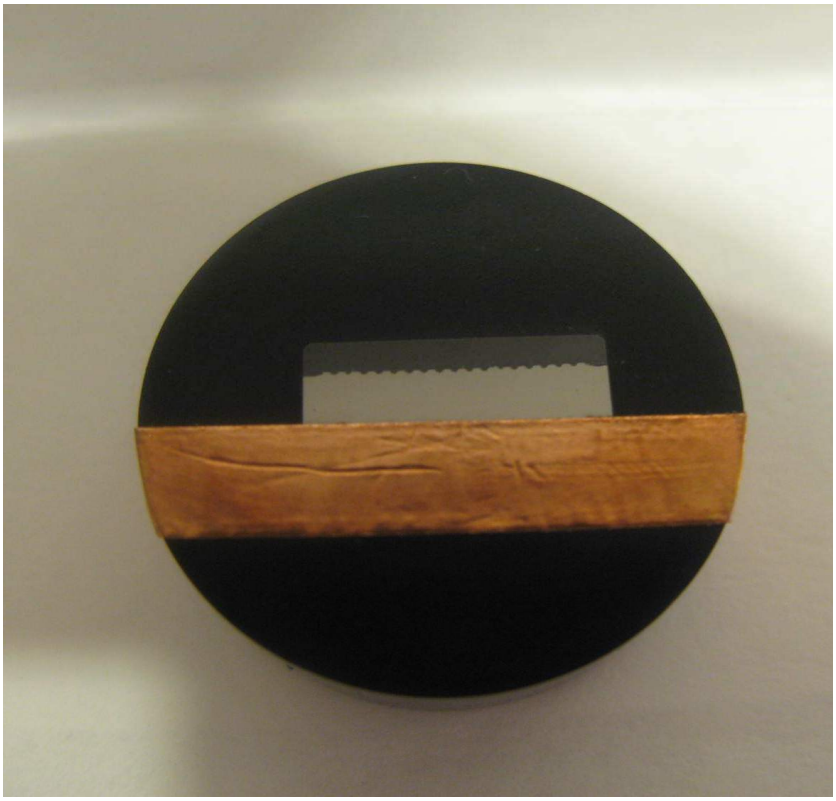

Figure 4-12: Sample prepared for SEM analysis.

The surface crystals were measured in the horizontal direction. The area image was divided in five rows. The crystal size data was collected by manually taking dimension from the crystals, therefore a measurement error should be considered. The sample measurements were collected, and the information was analyzed. From the resulting data we can determine that the proposed cell and the current cell design provide the same uniform crystal growth at the interface and through the diamond layer maintaining consistent crystals sizes.

Table 4-5: Diamond crystal size and material analysis comparison for both manufacturing methods

Proposed Cell Design Sample						Current Cell Sample				
	Crystal Size (in microns)		Material Analysis in %			Crystal Size (in microns)		Material Analysis in %		
Sample	Average	Median	Tungsten (W)	Cobalt (Co)	Carbon (C)	Average	Median	Tungsten (W)	Cobalt (Co)	Carbon (C)
Right Side										
Top	15.73	14.16	2.58	9.12	88.29	16.87	15.00	2.95	8.92	88.13
Bottom			2.99	8.68	87.33			3.14	8.79	88.07
Middle										
Top	14.14	13.52	2.68	9.29	88.03	15.83	14.79	3.22	8.80	87.98
Bottom			3.61	9.22	87.16			2.62	9.58	87.80
Left Side										
Top	15.55	13.94	3.26	8.74	88.01	19.11	18.80	3.13	8.25	88.62
Bottom			3.30	8.87	87.83			3.53	8.65	87.82
Average	15.14	13.87	3.07	8.99	87.78	17.27	16.20	3.10	8.83	88.07
Standard Dev	0.87	0.33	0.39	0.26	0.44	1.68	2.26	0.30	0.43	0.30

There is not a substantial difference between the two samples; the amount of tungsten and cobalt found in the diamond layer was within the allowed parameter for the diamond mix and the tungsten carbide utilized to make the samples.

It can be concluded that the two cell designs are quite similar with regard to the results obtained through the diamond synthesis process. Normal amounts of exaggerated tungsten-carbide grain growth with cobalt precipitations, as shown in Figure 4-13, were found at the interface of the diamond and the WC substrate in the samples from both processes. The crystal size difference between the samples was within the error of the test. It can not be determined with this process that there is a physical difference between the samples.

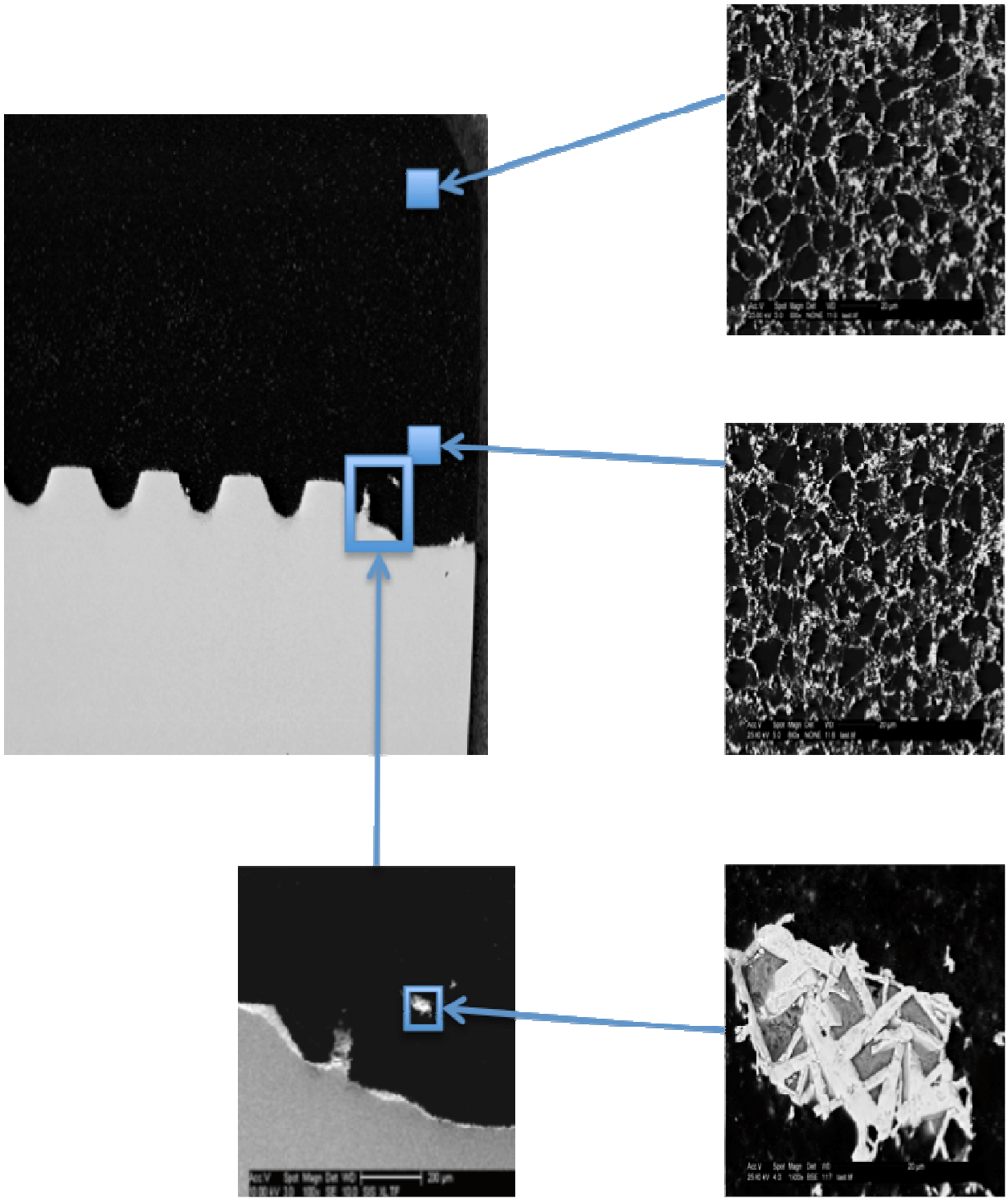

Figure 4-13: SEM images of an insert made with the current process. The top left corner is a 50X magnification image of the right side interface area of the insert. The two top right images are 800X magnifications of the top and bottom areas marked on the right image. The bottom two images are a magnification of an exaggerated WC grain growth at the diamond-to-substrate interface.

5 Conclusions and Recommendations

5.1 Summary

The goal for this research was to reduce the current manufacturing cost of the polycrystalline diamond compact inserts utilized in the natural gas and oil drilling industry while not reducing their current performance.

With the current cube cell design, it is necessary to bond an extra WC substrate to the polycrystalline diamond insert to achieve the sizes commonly ordered by the customers.

The researcher was able to achieve the goal by increasing the operating volume of the cube cell assembly.

A new cell design was proposed, and tested through this thesis. The next two sections were developed to express the researcher's conclusion and recommendations for future study.

5.2 Conclusion

The objective of the thesis stated that modifications could be made to the cube cell design to allow two PDC inserts to be manufactured to size, eliminating the need for bonding. The challenge on this research was that in order to preserve the performance of the cutters it was necessary to maintain the same internal pressure and temperature utilized in the current diamond sintering process.

The alternative proposed in this thesis consisted in changing the heat distribution in the cube cell assembly allowing larger samples to fit in the cell while achieving the same internal pressure.

The data for the first hypothesis were provided by the polycrystalline diamond compact inserts manufactured to test the second hypothesis.

The results from the material cost analysis showed that the proposed cell design would reduce the total manufacturing cost of polycrystalline diamond compact inserts by 27.58%. Most of the cost difference was gained by eliminating the bonding components accounting for 22.54% of the total manufacturing cost. The rest of the gain pertained to the end-heating components. The proposed method did not add any significant machine supply cost to the manufacturing process.

A reduction in the operation cost was also achieved by the proposed method. Through time studies it was possible to prove that the proposed method took 21.58% less time to produce a polycrystalline diamond compact insert than the current method. The finishing process of the inserts was abridged to less than 50% of the current method. The proposed method also reduced rejects, by eliminating four post-press processes. Four machines and two operators could also be relocated to form a new manufacturing cell if demands required.

The subsequent information supported the second thesis hypothesis. By internal insert testing it was possible to confirm that the performance of the product manufactured with the proposed cell design did not differ significantly from that of the inserts made with the current method.

The t-test results done on the G-Ratio data obtained through the heavy wear test supports the hypothesis that the performance of the inserts manufactured with the proposed cell design is

not less than that of the inserts manufactured with the current process. With a confidence level of 95% it can be stated that the data from the two sample populations are not significantly different.

Two other tests were done on the polycrystalline diamond inserts to determine if the new process altered the structure of the inserts in any way. The results from the exaggerated tungsten-carbide t-test showed once again that the means of the two populations are not significantly different. When the samples were observed at high magnification and their material composition analyzed, there were no significant differences between the samples manufactured with the current and the proposed methods.

Therefore, it has been confirmed that by modifying the current cube cell design used in the high-pressure/high-temperature apparatus it is possible to reduce manufacturing cost of polycrystalline diamond compact inserts with out diminishing their current performance.

5.3 Recommendations for Future Study

This thesis shows that it is possible to alter the cube cell assembly utilized in the high-pressure/high-temperature apparatus for the manufacturing of polycrystalline diamond compact inserts without negatively impacting the performance of the inserts.

The current cell has not been substantially modified since Dr. Hall's first design in the late 1950's, and there are many more improvements that can be done to the cell.

The following are some of the items that can be revised in future work:

1- Reduce the amount or modify the refractory metal utilized in the can assembly to reduce cost and minimize possible contamination.

2- Convert the cube cell assembly to a true center-heating assembly where the heat can be centralized only in the area where it is required for the sintering process. This

will reduce the number of components and also be less stressful for the WC substrate.

3- Develop a pre-gasketed cube cell assembly for the cubic HP/HT apparatus to eliminate the gasket material around the insert. This will lead to a higher internal pressure, therefore a better performing cutter.

4- Optimize the current pre-press dimensions to reduce the post press finishing operations, thus reducing processing cost.

These activities would continue improving the manufacturing of polycrystalline diamond compact inserts, helping the American companies to maintain their competitive advantage by reducing cost and improving their productivity and product performance. We can consider these thesis results as the first change of many more to come in the super-abrasive industry.

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