

**Development of Advanced Drill Components for BHA Using Microwave
Technology Incorporating Carbide, Diamond Composites and
Functionally Graded Materials**

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ABSTRACT

The main objective of this program was to develop an efficient and economically viable microwave processing technique to process cobalt cemented tungsten carbide with improved properties for drill-bits for advanced drilling operations for oil, gas, geothermal and excavation industries. The program was completed in three years and successfully accomplished all the states goals in the original proposal.

In three years of the program, we designed and built several laboratory scale microwave sintering systems for conducting experiments on Tungsten carbide (WC) based composites in controlled atmosphere. The processing conditions were optimized and various properties were measured. The design of the system was then modified to enable it to process large commercial parts of WC/Co and in large quantities. Two high power (3-6 kW) microwave systems of 2.45 GHz were built for multi samples runs in a batch process. Once the process was optimized for best results, the technology was successfully transferred to our industrial partner, Dennis Tool Co. We helped them to built couple of prototype microwave sintering systems for carbide tool manufacturing.

It was found that the microwave processed WC/Co tools are not only cost effective but also exhibited much better overall performance than the standard tools. The results of the field tests performed by Dennis Tool Co. showed remarkable advantage and improvement in their overall performance. For example: wear test shows an increase of 20-30%, corrosion test showed much higher resistance to the acid attack, erosion test exhibited about 15% better resistance than standard sinter-HIP parts. This proves the success of microwave technology for WC/Co based drilling tools.

While we have successfully transferred the technology to our industrial partner Dennis Tool Co., they have signed an agreement with Valenite, a world leading WC producer of cutting and drilling tools and wear parts, to push aggressively the new microwave technology in to the marketplace.

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1. EXECUTIVE SUMMARY

New developments and innovative ideas in the area of materials processing have often led to the discovery of new materials and/or new technologies which could be energy-efficient and cost-effective. The use of microwave energy in the area of materials processing is one such new discovery. The advantages of using microwave technology for various ceramic systems has already been demonstrated and proven. The recent developments at Penn State including the work performed under this program have succeeded in applying the microwave technology for the commercialization of WC/Co based cutting and drilling tools and fabrication of transparent ceramics for advanced applications.

Microwave heating is fundamentally different from conventional heating. In the microwave process, the heat is generated internally within the material instead of originating from external sources. It is a specific function of the material being processed, and there is nearly 100% conversion of electromagnetic energy into heat largely within the sample itself unlike in conventional heating where considerable energy is wasted. Due to the volumetric heating and energy conversion, microwave sintering is very rapid and grain growth is minimized. The total process cycle time is very short.

Tungsten carbide (WC) based composites due to their unique combination of hardness, toughness and strength, are universally used in cutting tools and drills, machining of wear resistant metals, mining, and geothermal drilling. Conventional methods for sintering WC with Co as binder involve high temperature and lengthy processing cycle (about 24 hours), and make the production cost quite high. Furthermore, such conventional conditions favor an undesired growth of WC grains during sintering.

Consequently, the mechanical strength and hardness of the tool are diminished. However, microwave process does not require long processing times and produces better performed products.

The microwave processed WC/Co tools are not only cost effective but also have exhibited much better performance. The results of the field tests performed on these parts by Dennis Tool Co. showed remarkable advantage and improvement in their overall performance. This proves the success of microwave technology for WC/Co based drilling tools. Some of the highlights of these tests are as the following:

- Wear test of microwave parts shows 20-30% increase in the performance over conventional part.
- Cobalt leaching test by acid treatment shows microwave processed part was 3-4 times more resistant than the conventional counterpart.
- Microstructures of microwave sintered sample is much finer in grain size compared to the Sinter-HIP samples.
- Corrosion test performed on WC/13%Co substrates using nitric acid clearly showed superior resistance to the attack of the acid on the microwave sintered samples to conventional sinter-HIP sample. The conventional sinter-HIP sample was extensively damaged after two days including chipping off the material, while microwave material remained intact and hardly any damage was observed. The weight loss of the microwave sintered parts was more than 7 times less than the sinter-HIP parts even including the chips that fell off the parts. The weight loss was over 19 times less when weighing only the main piece without the chips.
- Erosion test was performed using dry blasting as the erosive medium and a blast spray gun with air pressure of 100 psi. The test results showed that the microwave sintered part had about 15% better erosion resistance than the corresponding sinter-HIP parts.

Prototype microwave systems were designed, built and tested for their capability to produce parts with reproducible and consistent properties, and now this technology is well on its way to commercialization. While we have successfully transferred the technology to our industrial partner Dennis Tool Co., they have signed an agreement with Valenite, a world leading WC producer of cutting and drilling tools and wear parts, to push aggressively the new microwave technology in to the marketplace.

The research and development of diamond composite area has progressed quite satisfactorily and in our exploratory experiments significant successes have been made in the development of laboratory type diamond composites.

It is recommended that further systematic investigation should be conducted to take this technology also to commercial scaling-up. Another area that should also be explored is the development of compositional and functional gradient materials using WC/Co, metals/steels and diamond using the microwave technology.

2. PROJECT SUMMARY

2.1. Introduction

New developments and innovative ideas in the area of materials processing have often led to the discovery of new materials, with interesting and useful properties, and/or new technologies which are *faster and better* (improvement in product performance), *cheaper* (energy-efficient and cost-effective), and *greener* (environmentally friendly). The use of microwave energy in the area of materials processing is one such new discovery, which shows great potential for improving product performance and reducing the cost of materials consolidation substantially. This field is now has been accepted as commercially viable and several relevant industries have initiated commercial ventures using recent developments in microwave technology. The most recent example is the signing of a partnership agreement between Dennis Tool Company (our industrial sponsor) and Valenite, a world leading tungsten carbide producer of cutting and drilling tools, in pushing the Penn State developed microwave technology to the marketplace (a copy of the announcement is enclosed).

The Materials Research Laboratory (MRL) at The Pennsylvania State University (PSU) has been a pioneer in the microwave processing of white (oxides) ceramics and recently expanded its activities to the processing of hard metal (tungsten carbide based tools) composites and powder metals. We have also developed a second-generation microwave sintering apparatus and technique in which green parts of typical commercial sizes can be continuously processed with high energy efficiency and cost effectiveness to yield perfectly formed and fully dense alumina, WC/Co and metal components. The product performance of microwave parts has evidently increased 30-40% in case of

WC/Co composites. In this program, Dennis Tool Company (DTC) of Houston, a leader in specialty cutting tools for the petroleum drilling industry, has been our partner and has developed a higher temperature brazing system for hard-metal based drilling/cutting tools, and a lower frictional and wear polycrystalline diamond compact (PDC) cutter. This development in combination with the utilization of microwave processing offers great potential in the development of new advanced drilling tools with greatly improved performance and substantially longer life than the existing tools and reduction in processing cost.

2.2. Results and Discussion

2.2.1. Microwave Processing Technology

Microwave processing of materials, which includes heating and sintering, is fundamentally different from conventional processing which involves radiant/resistance heating followed by transfer of thermal energy via conduction to the inside of the body under process. In case of microwave process it is the absorption of the microwave energy followed by volumetric heating involving a conversion of electromagnetic energy into thermal energy, making the process instantaneous and rapid. Microwaves are a small portion of the electromagnetic spectrum with wavelengths ranging from 1 mm to 1 m in free space and frequency between 300 GHz to 300 MHz, respectively. It is well recognized that the bulk metals are opaque to microwave and are good reflectors. However, recently it has been discovered (1) that metallic materials in powder and porous form are very good absorbers of microwaves and get heated very effectively and rapidly.

In the microwave process if the material couples in microwave field, the heat is generated internally within the material instead of originating from external sources, and flows towards outside. Hence, there is an inside-out heating profile unlike in conventional

process where heat flow occurs from outside to inside. In general, the microwave heating is very rapid as the material is heated via energy conversion rather than energy transfer as in the conventional method. Microwave heating is a function of the material being processed. There are major potential and real advantages using microwave energy for material processing over conventional heating [2-4]. Some of these advantages include: time and energy saving, very rapid heating rates ($>400^{\circ}\text{C}/\text{min}$), considerably reduced processing time and temperature, fine microstructures and hence improved mechanical properties, better product performance, environment friendly, etc.

Microwave Processing of Ceramics: Microwave energy has been in use since the late 1940s in ceramic processing with a big push in the eighties. Ceramic processes where microwaves had been applied include: process control, drying of ceramic sanitary wares, calcination, decomposition of gaseous species by microwave plasma, and sintering of oxide ceramics by microwave plasma. However, except drying of ceramic wares there is hardly any other area where microwave technology has been commercially exploited. Only, recently some success has been achieved in commercializing the microwave sintering of tungsten carbide based cutting tools [5] using the technology developed at Penn State.

Microwave sintering of WC/Co Composites: WC/Co composites due to their unique combination of hardness, toughness and strength, are universally used for metal and rock cutting and drilling operations. Conventional methods for sintering WC with Co as a binder phase involve high temperature (up to 1500°C) and lengthy sintering cycles of the order of one day in order to achieve a high degree of sintering [6]. Such conditions unfortunately favor undesirable WC grain growth. Consequently, the mechanical strength and hardness of the tools are diminished. It is generally recognized that finer

microstructures provide superior mechanical properties and longer life of the product. Often, additives such as titanium carbide (TiC), vanadium carbide (VC) and tantalum carbide (TaC) are used to prevent grain growth of WC grains. Unfortunately such additives deleteriously affect the mechanical properties of the product. Since microwave heating requires very little time to obtain nearly full sintering, the grain growth is relatively suppressed and finer microstructure is generally obtained. In 1991, J. P. Cheng in a Ph.D. thesis [7] first showed that WC/Co composites could be sintered in a microwave field. Gerdes and Willert-Porada [8] also reported the sintering of similar WC objects from normal size powders, but they followed reactive sintering route using a mixture of pure W, C and Co instead of normal sintering. In another work [9], Cheng et al. at the Penn State University using a newly designed microwave apparatus (Figure 1) were able to fully sinter WC commercial green bodies containing 12% and 6% Co. They observed that microwave processed WC/Co bodies exhibited better mechanical properties than the conventional parts, fine and uniform microstructure (\simeq 1 micron size grains) with very little grain growth, and nearly full density without adding any grain-growth inhibitors when sintered at 1250°-1320°C for only 10-30 minutes [4,10,11]. Figure 2 shows some of the WC/Co commercial tools processed in microwave system under the program.

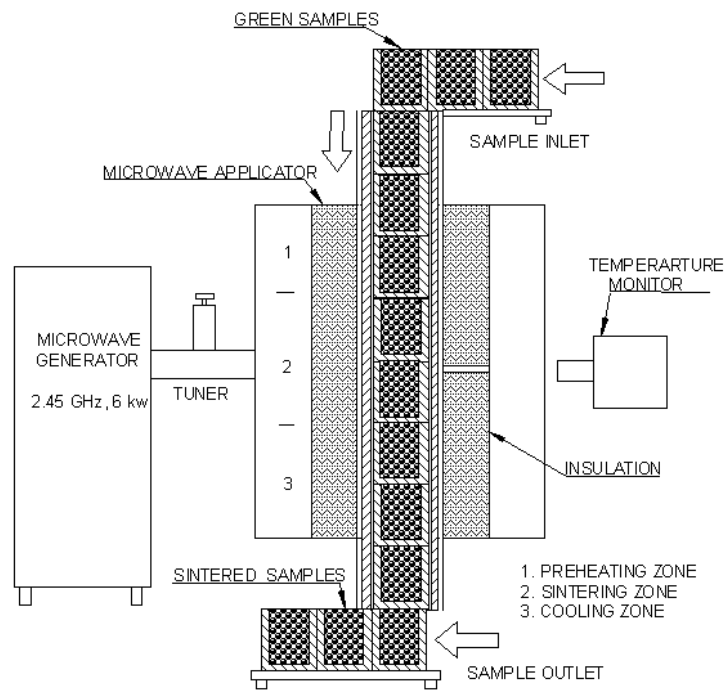


Figure 1. Schematic of a continuous microwave system for sintering of WC/Co composites

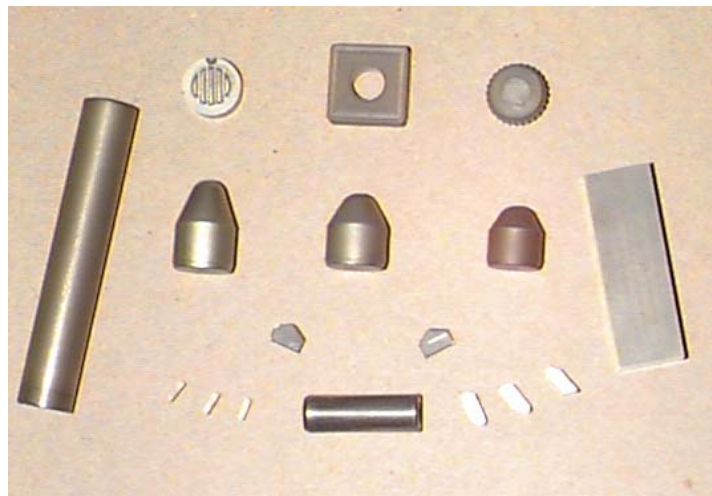


Figure 2. Microwave sintered commercial parts of WC/Co.

Microwave sintering of powdered metals: Metal powders are used in industry for diversity of products and applications. Traditional powder metallurgy is a process whereby a metal or alloy powder is compacted in to a green body and then sintered to net shape at elevated temperatures. The most important metal powders in use are: iron and steel, copper, aluminum, nickel, Mo, W, WC, Sn and their alloys. It has been well recognized by the researchers that microwave heating does not work in metals and is good only to oxide ceramics and semi-metals like carbides and nitrides. We found that the microwave sintering can also be applied as efficiently and effectively to powdered metals as to many ceramics [1]. Bulk metals are excellent reflectors in microwaves at room temperature and in general are not heated significantly. But in powdered and unsintered form virtually all metals, alloys, and intermetallics couple/heat in a microwave field very effectively to produce highly sintered bodies with improved mechanical properties. The microwave sintering of PM green bodies comprising various metals and metal alloys (for example: Fe-Ni-C and Fe-Cu-C systems) produced highly sintered bodies in a very short period of time [16]. The mechanical properties such as the modulus of rupture (MOR) and hardness of microwave processed samples were much higher than the conventional samples. The densities of microwave processed samples were also better than conventional samples. Figure 3 shows some of typical powder metal steel parts which have been microwave sintered at Penn State.



Figure 3. Microwave sintered powder metal commercial parts.

Microwave sintering of transparent ceramics: Transparency is a valuable optical property of materials. The nature of the material including grain size, density, crystal structure, porosity and the grain boundary chemistry which influence the degree of transparency. Glasses are optically isotropic and have no grain boundaries, and therefore possess excellent transparency. Most non-cubic ceramics are anisotropic and polycrystalline. The grain boundaries in the ceramic strongly scatter light. Therefore, to convert a non-cubic ceramic having grains larger than the wavelength of light, into a transparent ceramic, one must have very low grain boundary volume and no inter- or intra-granular porosity. However, if the grain size is smaller than the wavelength of the light ($0.4 - 0.7 \mu$), the light can transmit through the ceramic. Cubic ceramic materials such as spinels and AlON can

be made into transparent ceramics even if the grain size is larger than the wavelength of light. To achieve transparency in a ceramic, one must control the grain growth, eliminate porosity and achieve a fully dense body.

The conventional methods to fabricate fully dense and reasonably transparent ceramics involve high temperatures, lengthy sintering conditions, and various complex processing steps, which not only make the processing of transparent ceramics uneconomical but often the desired properties are not achieved. However, microwave method has been successfully used to fabricate transparent ceramics due to its ability to minimize the grain growth and produce a fully dense ceramic in a very short period of time without utilizing high pressure conditions [12,13]. Fang *et al.* [14] prepared a fully sintered transparent ceramic by microwave processing in less than 15 minutes. Related work [15,16] also demonstrated that one could make transparent ceramics of spinel and alumina ceramics as well. Fully dense alumina and spinel ceramics using high purity and submicron size powders were developed with reasonable degree of transparencies on laboratory type small samples at 1700°C sintered for 15 minutes in the microwave system. Figure 4 shows some of the microwave processed transparent ceramics of hydroxyapatite, alumina, spinel phases and AlN.

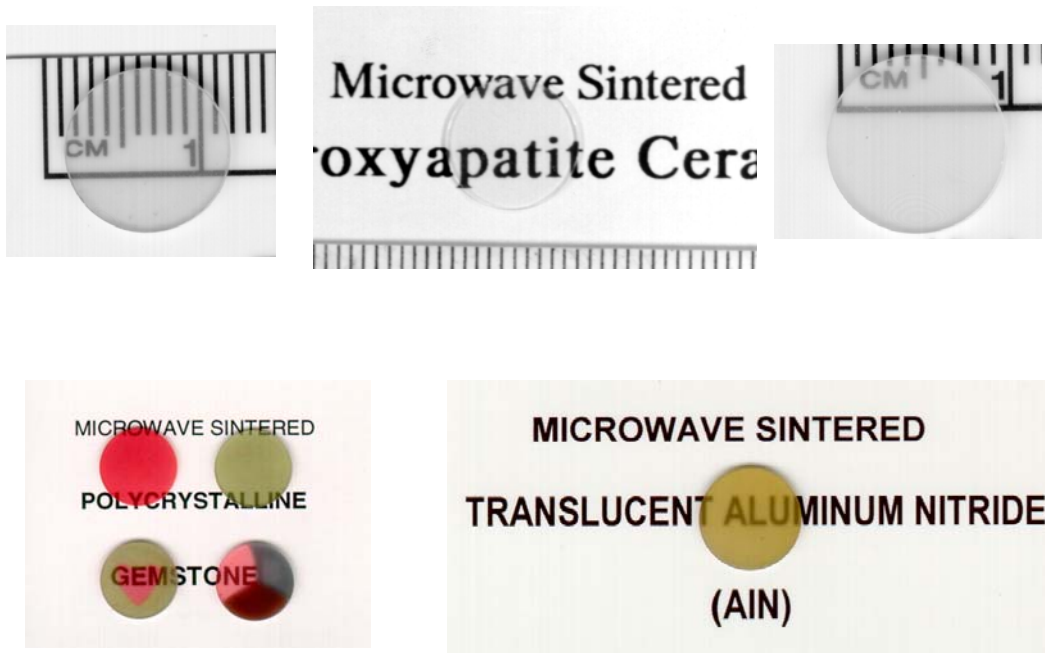


Figure 4: Microwave Processed Transparent Ceramics

2.2.2. Development of a Cost Effective New Advanced Drill Bit

The drilling systems in the exploration of oil, gas, geothermal, mineral, water well, and other natural resources have seen constant innovations and improvements. New advanced drilling technology has been developed which is safer, cheaper and faster. Many research programs throughout the country have been aimed at achieving short-term and long-term benefits. Improvements in individual system components could be incorporated in conventional rotary drills to provide short term payoffs. Our proposal focuses on such short-term gains by making substantial improvement in the performance of the WC/Co based components used in the drill-bits of all categories.

Drilling operation on rocks involves a set of processes for breaking and removing rock to produce boreholes, tunnels, and excavations. In general, the object of drilling is to reach a subsurface target in order to explore natural resources like oil, gas, mineral etc. or make tunnels for building infrastructures. The paramount objectives of a drilling operation are to reach the target safely in the shortest possible time and at the lowest possible cost. The principal component of a drilling system consists of a drill bit that comminutes rock on the bottom of the borehole. In most cases, a drill bit is made of steel and dozens of WC/Co/diamond cutters of special designs attached to it. These cutters remove rock by impact or shearing processes. New wear-resistant, diamond-coated cutters are finding increased use in hard abrasive rocks. Currently, their availability and performance/cost ratios are serious concerns of the drilling industry. The performance of the drill bit greatly depends upon the wear qualities, toughness, and hardness of the WC/Co components, and the overall performance depends upon their (i) design, (ii) composition, (iii) microstructure, and (iv) any coating material.

2.3. Conclusions

At the conclusion of this program, we have succeeded in accomplishing almost all the goals as described in the original proposal. Some of the accomplishments and the milestones achieved are listed below:

- 1 Successful design and building of prototype microwave sintering systemz for the sintering of WC/Co based drilling and cutting tools.
- 2 Successful transfer of the technology to the Dennis Tool Company for the manufacturing of carbide tools.
- 3 Completion of the laboratory and field tests on selected carbide samples. The test results show the following:
 - Wear test of microwave parts shows 20-30% increase in the performance over conventional part.
 - Cobalt leaching test by acid treatment shows microwave processed part was 3-4 times more resistant than the conventional counterpart.
 - Microstructures of microwave sintered sample is much finer in grain size compared to the Sinter-HIP samples.
 - Corrosion test performed on WC/13%Co substrates using nitric acid clearly showed superior resistance to the attack of the acid on the microwave sintered samples to conventional sinter-HIP sample. The conventional sinter-HIP sample was extensively damaged after two days including chipping off the material, while microwave material remained intact and hardly any damage was observed. The weight loss of the microwave sintered parts was more than 7 times less than the sinter-HIP parts even including the chips that fell off the parts. The weight loss was over 19 times less when weighing only the main piece without the chips.
 - Erosion test was performed using dry blasting as the erosive medium and a blast spray gun with air pressure of 100 psi. The test results showed that the microwave sintered part had about 15% better erosion resistance than the corresponding sinter-HIP parts.

- 4 Preliminary experiments on the diamond-carbide composites indicate that microwave process can sinter diamond composite in 30 minutes at temperature without causing any damage to the diamond.
- 5 Preliminary experiments of the development of compositionally gradient system of carbide and steel were successfully conducted.

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3. APPENDIX

3.1. Current Success and Future Research

The cutting and drilling operations underground are performed usually by tool-bits made of tungsten carbide with cobalt as binder. It is well recognized that the performance of these tools can be drastically improved if diamond is incorporated as either (a) coating material, or (b) as an active phase in WC/Co matrix, or (c) as TSD (thermally stable diamond) compact or PDC (polycrystalline diamond compact) imbedded on to WC/Co surface. Conventional processes for diamond composite development which involve high temperature and pressure conditions have not produced much success so far, and secondly they are very expensive and time consuming. As demonstrated in our ongoing program of WC/Co tools, microwave processing provides rapid sintering and single step process to achieve full sintering without using high pressure and high temperature conditions. In microwave process the time for sintering is kept to minimum and temperature relatively lower than required in a conventional heating. This means that in microwaves not only the diamond can be prevented from graphitization but can also be bonded in the matrix. In our exploratory research we have adopted many approaches to develop diamond composites with WC/Co as the matrix. Our results are positive so far and highly encouraging. However, this research needs to be pursued further vigorously so that we can eventually develop diamond composites based commercial tools using microwave technology.

Currently, we are exploring the following approaches, as illustrated in Figure 5 and 6.

1. Metal (Si, Co, WC/Co) and diamond composites
2. Diamond + WC/Co on steel substrates
3. WC/Co + TSP (Thermally Stable Polycrystalline Diamond Compact)
4. Diamond ceramics

5. Encapsulation of steel with diamond composite
6. *In-Situ* brazing between diamond composite and steel

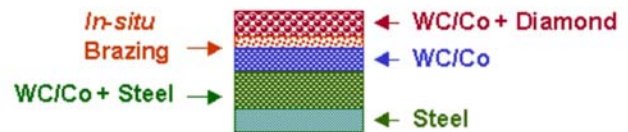
We are using metal coated diamond powder either prepared in the laboratory or acquired from the commercial source. Pure diamond powder is sensitive to Co attack and also does not form any bonding with matrix. Ni, Ti and Cr coated diamond powders are being used in this study. The highlight of this research so far is that we have been able to successfully obtain about 96% density of the diamond composites at ambient pressure and without damaging the diamond. This has been achieved on small laboratory type samples. Another area in which we have achieved some success is *in-situ* brazing of carbide with steel body without using in fact any brazing material. This is an innovative approach to the brazing problem. Figure 7 illustrates the success obtained in this area in which we developed a compositionally graded material system. The same idea can further be expanded to develop a complete drilling tool of WC/Co and diamond composite including PSD/TSD.

DIAMOND COMPOSITES DEVELOPMENT

1. Metal + Diamond

- Si, Co, WC/Co

2. (Diamond + WC/Co) + WC/Co Substrate + Steel



3. WC/Co + TSP (Thermally Stable Polycrystalline Diamond)



4. Diamond Ceramics

Figure 5: Various Approaches for Diamond Composites Development Using Microwave Technology

METAL AND DIAMOND COATINGS AND ENCAPSULATIONS

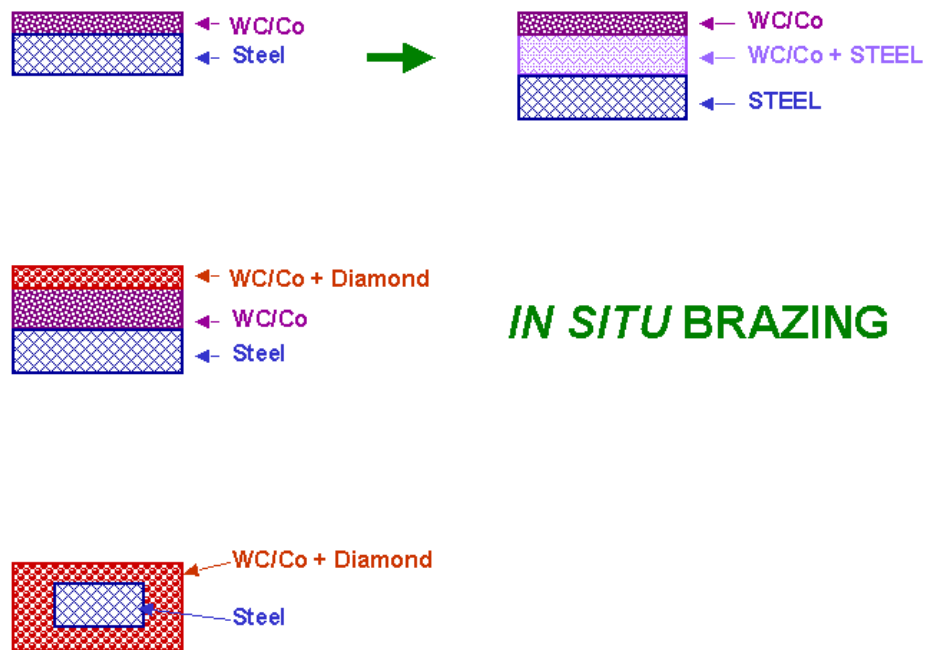


Figure 6: Development of Metal and Diamond composites and Encapsulation: In-Situ Brazing using Microwave Process

Development of Compositionally Gradient Material Using Microwave Technology

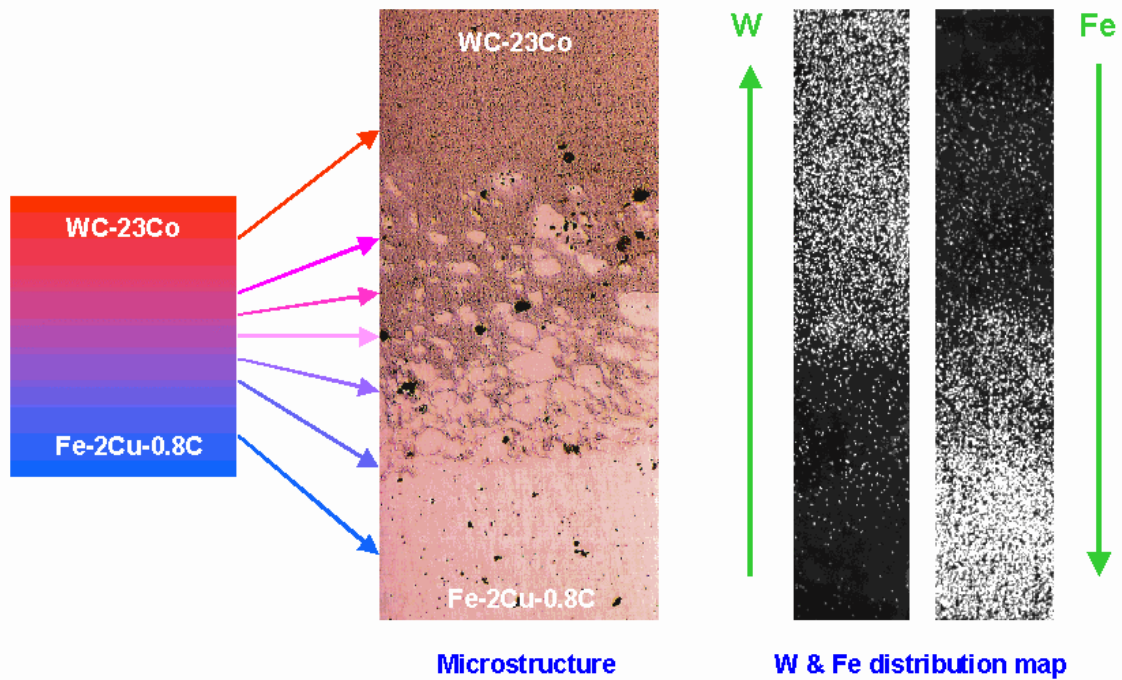


Figure 7: Development of Compositionally Gradient Material using Microwave Technology: A Microwave Solution to Brazing Problem.

3.2. Development of Diamond Composites Using Microwave Process

Diamond is a unique material with respect to a host of properties. It is the hardest material known, with highest thermal conductivity and very high corrosion resistance in hostile atmosphere. This makes diamond very attractive and unique in major cutting and grinding industries. Composites based on hard-metals (such as WC) and metal - alloys are commonly used as cutting tools for metal and rock cutting applications. Carbides, nitrides, diamond and other hard coatings are often applied on these cutting tools to enhance the performance and the durability of the system. It is generally recognized in the drilling and mining industry that the performance and durability of WC/Co based tools can further be improved many times if diamond is incorporated in WC/Co matrix rather than a thin coating on the surface, and if a fully dense composite is developed. Efforts to develop a diamond coating on to WC/Co surface encountered many problems such as adherence and mismatch of thermal expansion causing cracks and peeling off. Existing diamond tools including PDC with high performance are basically composites of 2 - 35 volume % natural or synthetic diamond embedded in a metal, alloy or WC matrix. The conventional methods to make PDC involve very high temperature and pressure conditions, and hence the processing cost is very high. If the fabrication is performed without resorting to high pressure and high temperature conditions, it would have tremendous advantages in terms of cost and overall commercialization.

Numerous attempts involving high temperature, hot press (HP) and hot isostatic press (HIP) conditions have been made to develop cutting tools with diamond as an active phase in the composite instead of as a coating material, but all these efforts have met with little success. The recent developments in the microwave technology have enabled us to process diamond-metal composites at lower sintering temperatures and at a room pressure

without causing graphitization of the diamond. This opens an entirely new opportunity and challenge for the development of diamond and WC/Co based products for host of applications. In the proposed program we will utilize the microwave techniques to develop diamond composites without resorting to high pressure and temperature conditions.

Building on the success of microwave sintering of WC/Co parts and initial positive results in the exploratory experiments conducted so far in the diamond composites area, we recommend that in future research one should adopt several approaches for the development of diamond composites by exploiting the inherent advantages of microwave technology, e.g.: (i) design and develop improved PDCs and new WC/Co - TSD composites (ii) develop WC/Co - diamond composites using industrial and metal coated diamond, (iii) develop (WC/Co + diamond) composites on to steel body without brazing, and (iv) encapsulation of steel with diamond composites.