DEVELOPMENT OF CUBIC BORON NITRIDE (CBN) COATING PROCESS FOR CUTTING TOOLS

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Approval of Thesis

DEVELOPMENT OF CUBIC BORON NITRIDE COATING PROCESS FOR CUTTING TOOLS

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I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

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ABSTRACT

DEVELOPMENT OF CUBIC BORON NITRIDE (CBN) COATING PROCESS FOR CUTTING TOOLS

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In today's market conditions, higher tool life and durable cutting tools which can stand high cutting speeds are required in chip removal process. In order to improve the performance of cutting tools, coatings are employed extensively. Cubic boron nitride (cBN) is a new kind of coating material for cutting tools due to its outstanding properties and testing of cBN as a hard coating for machining have been increasing in recent years. However, there are some challenges such as compressive residual stress, poor adhesion and limiting coating thickness during the deposition of cBN on substrates.

In this study, cubic boron nitride (cBN) coatings are formed on cutting tools from hexagonal boron nitride (hBN) target plates. For this purpose, a physical vapor deposition (PVD) system is utilized. PVD system works on magnetron sputtering technique in which material transfer takes place from target plate to substrate surface. Firstly, cBN coatings are deposited on steel and silicon wafer substrates for measurements and analyses. Compositional, structural and mechanical measurements and analysis are performed for the characterization of coatings. Next, several types of cutting tools are coated by cBN and the effects of cBN coatings on cutting performance are investigated. Finally, it can be said that cubic boron nitride coatings are successfully formed on substrates and the improvement of wear resistance and machining performance of cBN coated cutting tools are observed.

Keywords: Cubic Boron Nitride, Cutting Tools, Physical Vapor Deposition Technique, Machining

ÖZ

KESİCİ TAKIMLAR İÇİN KÜBİK BOR NİTRÜR (CBN) KAPLAMA SÜRECİNİN GELİŞTİRİLMESİ

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Talaşlı imalat işleminde bugün gelinen noktada daha uzun ömürlü ve daha yüksek hızlarda kesim yapabilen kesici takımlara ihtiyaç vardır. Kesici takımların performanslarının artırılması için yaygın olarak farklı kaplamalar kullanılmaktadır. Kübik bor nitrür (cBN) malzemesi de sahip olduğu üstün özelliklerinden dolayı kesici takımların kaplanmasında son yıllarda yaygın olarak denenmektedir. Ancak cBN kaplamaların alttaşlar üstünde büyütülmesi sırasında artık basma gerilmeler, düşük yapışkanlık ve dolayısıyla sınırlı kaplama kalınlığı gibi zorluklar ile karşılaşılmaktadır.

Bu projede heksagonal bor nitrür (hBN) hedef plakalar kullanılarak kesici takımların üzerinde kübik bor nitrür (cBN) kaplamalar elde edilmiştir. Bunun için fiziksel buharlaştırma yöntemiyle büyütme (FBYB) sistemi kullanılmıştır. FBYB sistemi magnetron saçtırma tekniğiyle çalışmakta olup, hedef plakadan alttaşa doğru bir malzeme transferi esasına dayanır.

CBN kaplamalar öncelikle analizler ve ölçümler için çelik ve silikon yonga plakası numuneleri üzerinde büyütülmüştür. Kaplamaların nitelendirilmesi için birçok yapısal, birleşimsel ve mekanik karakterizasyon teknikleri kullanılmıştır. Karakterizasyon aşamasından olumlu sonuçlar alınınca farklı türde kesici takımlar kaplanmış ve kesim performans analizleri yapılmıştır. Sonuç olarak kübik bor nitrür kaplamalar alttaşlar üzerinde başarıyla oluşturulmuştur ve kesici takımlar üzerinde oluşturulan cBN kaplamaların da aşınma direncine ve kesim performansına katkısı gözlemlenmiştir.

Anahtar Kelimeler: Kübik Bor Nitrür, Kesici Takımlar, Fiziksel Buharlaştırma Yöntemiyle Büyütme, Talaşlı İmalat

To My Family

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CHAPTER 1

INTRODUCTION

1.1 Background Information

Machining is a material removal process in which a cutting tool is utilized to remove excess material from a work piece in the form of chips so that the remaining material is the desired shape part [1]. The predominant action in machining is the relative motion between the work piece and cutting tool which results in shear deformation of the work material to form a chip [2].

The demand of the machining market is to increase the productivity or the material removal rate while decreasing the cost. Stephenson and Agapiou [3] claim that on the average, the cutting tool cost represents about 3% the total component cost in high volume production. Therefore, a 50% increase in tool life reduces the total cost per component by 1 to 2%. On the other hand, a 20% increase in the material removal rate could reduce the total cost per component by 15%. For this reason, tools having longer life and working at higher cutting speeds are desired in today's market condition. In order to obtain better machining performance, cutting tool materials and cutting tool geometries should be improved.

A recent trend in the machining industry and a new way to minimize the economic and environmental pressure placed on manufacturers today is to eliminate the use of coolants. Dry machining has become increasingly popular as a means of reducing production costs while protecting the environment. Dry machining is the machining without using a coolant or using minimum quantity of lubricant (MQL) which consists of drops or droplets of oil suspended in compressed air [10]. Dry machining offers significant decrease in the coolant usage, cleaner shop environment, less machine maintenance, longer tool life and long term cost saving. According to the study conducted by DaimlerChrysler, it is concluded that 16% of the cost of a machine part is directly attributable to the coolant used. By comparison, tooling accounts for only 4% of the part cost [11].

Cutting tool is the most significant part of the machining system. Therefore, selection of the proper cutting tool for a given work material brings success in metal cutting. A cutting tool is any tool that is used to remove metal from the work piece by means of shear deformation. It has one or more sharp cutting edges. There are two surfaces of the tool connected to the cutting edge; rake face and flank [2]. Rake face directs the flow of the newly formed chip whereas flank provides a clearance between the tool and work surface, thus protecting the surface from abrasion.

The performance of a cutting tool is concerned in terms of tool life and cutting speed. There are two failure mechanisms which determine the life of a tool. Sudden-death mechanism is a rapid, usually unpredictable and often catastrophic failure resulting from cutting forces or temperature. On the other hand, slow-death mechanism is a gradual tool wear on the rake face and flank face of the tool [1, 2].

There is a wide range of cutting tool materials available in the market depending on the machining process, work piece material and the cost. The most popular ones are high carbon steels and low/medium alloy steels, high speed steels, cast cobalt alloys, cemented carbides, cast carbides, coated carbides, coated high speed steels, ceramics, cermets, cubic boron nitride (cBN), polycrystalline diamond and single crystal natural diamond [4].

During machining the cutting tool is subjected to severe conditions such as high temperature, high cutting forces, and severe friction. Therefore, in order to withstand this harsh environment in machining process, a cutting tool should have the following characteristics; high hardness, resistance to abrasion, resistance to wear and chipping of the cutting edge, high toughness, high strength to resist bulk deformation, good chemical stability, adequate thermal properties, high stiffness, consistent tool life, correct geometry and surface finish [1].

Hard coatings are employed on cutting tool surfaces extensively to improve mechanical properties of the tool in the recent years. Main reasons to apply coatings on cutting tools are to provide longer tool life, improve wear resistance, increase hardness, prevent chemical reactions between the tool and work piece material, reduce built-up edge formation, decrease friction between the tool and work piece and prevent deformation of cutting tool edge due to excessive heating [3]. The coatings must be thick enough to prolong tool life, however; thin enough to prevent brittleness. Depending on the coating method and type of substrate, coating thickness can vary between 0,2 to 15 µm. Mainly, two most common coating techniques are chemical vapor deposition (CVD) and physical vapor deposition (PVD). Detailed information will be given about these techniques in the following chapters. Some examples of coatings that are used on cutting tools are; TiN, TiC, TiCN, TiAlN, CRN, Al_2O_3. Moreover, new materials and multi-layer composite coatings have been tested in last years.

Cubic boron nitride (cBN) has been tested as a coating material for cutting tool applications in recent years due to its excellent mechanical and chemical properties. Its high hardness, low friction coefficient, good thermal conductivity, high electrical resistivity, high wear resistance and chemical inertness at high temperatures can be stated as some of the outstanding properties of cubic boron nitride [5, 6]. Owing to these properties, the use of cubic boron nitride in cutting tool applications such as dry cutting, high speed machining and cutting of hard materials, has increased within the last years [7, 8]. Sintered cubic boron nitride cutting tools have already been used in defense, automotive, aerospace and manufacturing industries extensively. Major drawbacks of sintered cBN cutting tools are their high cost, poor ductility and difficulty of forming them into various cutting tool shapes [9]. Therefore, demand for thin or thick film deposition of boron nitride for cutting purposes has grown up rapidly. Consequently, many researchers have attempted to synthesize cubic boron nitride coatings. However, there are some challenges such as compressive residual stress, poor adhesion and limiting coating thickness during the deposition of cBN. One important fact about cubic boron nitride is that Turkey has the largest amount of boron reserve in the world. Therefore, production of boron related products should be encouraged and increased.

CHAPTER 2

SURVEY OF LITERATURE

Many studies have been conducted about dry machining, performance of cutting tools, effect of coatings on cutting performance, different coating materials, formation of cubic boron nitride on various substrates, parameters effecting the deposition process and application of cubic boron nitride coatings on cutting tools. Literature survey can be grouped as; studies related with cutting forces and dry machining, studies related with coating materials and coating techniques, studies related with deposition of cubic boron nitride films and studies related with cubic boron nitride coated cutting tools.

2.1 Cutting Forces and Dry Machining

Dölen et al. [12] developed cross reference models for estimating unknown principle force components in end-milling process. They suggested two compact cutting process models for end-milling and estimation of an unknown principal cutting force component was possible by these models. The estimation accuracy was investigated experimentally and limitations of the method were discussed. They also improved a cutting force estimator for CNC machining centers [13]. The estimator was desired to compute the machining forces accurately and also to filter the noise components in the measurements by using recursive discrete Fourier transform. The performance, accuracy and limitations of the estimator were also presented in their study.

Rincon et al. [14] discussed the effects of drill vibrations on cutting forces and torque. They considered the resultant effect of drill vibration and developed an analytical model to predict torque, thrust and radial forces in drilling. They commented that predicted mean drill torque and thrust values are in good agreement with experimental data and previously published models.

Weinert et al. [15] claimed that the elimination or significant reduction of cooling lubricant completely affected the production system. They recommended that a detailed analysis and adaptation of cutting parameters, cutting tools, machine tools and the production environment should be done to decide dry machining. Finally, they concluded that dry machining leads to reduction of lubrication cost and improve the overall performance of cutting tools according to the study (Figure 2.1). Klocke and Eisenblatter [10] presented the most recent developments in dry cutting. They analyzed the dry machining of cast iron, steel, aluminum, super alloys and titanium. However, it was stated that dry cutting may be preferable if the part and machining times in wet machining were equaled or suppressed.

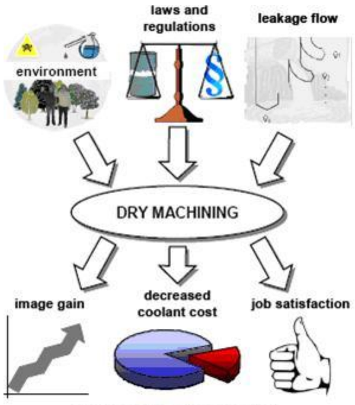


Figure 2.1 Dry Machining [15]

Aoyama [16] performed a study about development of a mixture supply system for machining with minimal quantity lubrication. To reduce the environmental pollution caused by cooling lubricants, the minimal quantity lubrication (MQL) technique should be used. However, Aoyama argued that the MQL technique may cause some problems in high-speed machining. The centrifugal force from the high-speed rotation of the spindle causes the oil mist to separate and that causes decrease in the oil mist supplied to the cutting area. The performance of that system was tested experimentally and was numerically evaluated. Bhowmick and Alpas [17] investigated the minimum quantity lubrication (MQL) drilling of aluminum-silicon alloys in water using diamond-like carbon coated drills. According to the researchers, it was seen that there was a significant decrease in the cutting forces and cutting torques in comparison to conventional flooded cooling.

2.2 Coating Materials and Coating Techniques

As mentioned in the first part, various coating materials have been utilized depending on cutting conditions of the machining process. Researchers have tested several materials and several coating techniques to improve the properties of coatings on cutting tools.

Hauert and Patscheider [18] tried to improve the performance of hard coatings used in dry machining. They said that expectations from hard coatings are lower friction, longer tool life, thermal stability in various environments and adaptability to different applications. These demands can be achieved by alloying with suitable materials (Figure 2.2). That’s why multilayer and nano-composite coatings have been developed in recent years. As a consequence of this study, researchers pointed out that overall performance of hard coatings can be upgraded by alloying, development of multilayer systems and nano-composites.

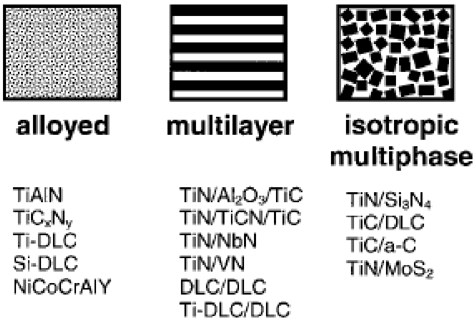


Figure 2.2 Schematic views of different coatings [18]

Attia and Kops [19] found a new approach to cutting temperature prediction considering thermal constriction phenomenon in multi-layer coated tools. In their approach, they considered the contact mechanics at asperity level and thermal constriction resistance. They developed a micro-contact model and obtained a correlation between contact pressure and thermal constriction resistance of uncoated and multi-layer coated tools. Moreover, they analyzed the thermal interaction and heat distribution between the chip and the tool and constructed a finite element model. As a result of their study, it is proven that the tool coating reduces the heat flowing into the tool significantly. By applying proper coating to the tool, stresses generated in the cutting process may be overcome and the wear resistance can be improved.

Klocke et al [20] carried out a useful study about improved cutting processes with adapted coating systems. They tried to improve the cutting tools by coating with multilayer hard thin films or composite hard/soft coatings for the conditions like interrupted cutting, machining of adhesive material and hard and dry machining. They performed many experiments and investigated how coatings affect contact conditions and wear mechanism during the machining of different ferrous and non-ferrous metals. According to their experiments, it is observed that TiAl-based or Al_2O_3 -containing film systems provided the best protection against wear.

Venkatesh et al. [21] compared the performance of uncoated, CVD-coated and PVD-coated carbides in turning and milling. According to their experiments, CVD-coated carbide tools gave the brightest surface in turning and milling. Moreover, when looked by optical microscopy, it was seen that irregular thermal cracks occurred in CVD coatings whereas no cracks were seen in PVD coatings (Figure 2.3, 2.4). Furthermore, high residual compressive stress in PVD coatings and low tensile residual stress in CVD coatings were observed at the end of the experiments. Also, cutting forces were lower and secondary shear zone was thinner in PVD coatings than in CVD coatings.

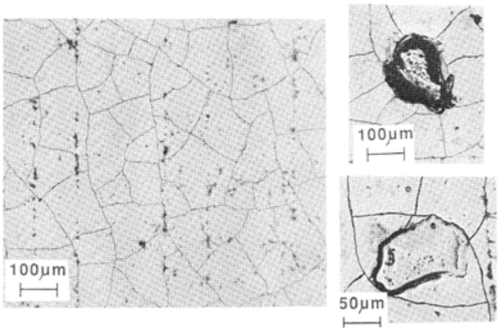


Figure 2.3 Plan view of a CVD coated insert shows cracks, pores and asperities

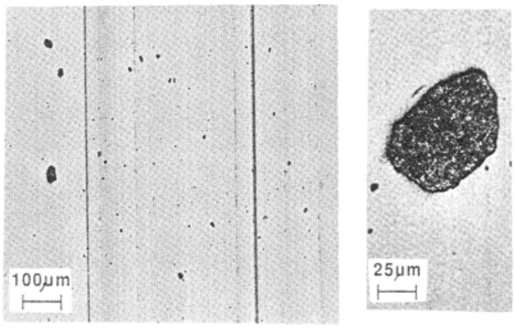


Figure 2.4Plan view of a PVD coated insert shows no cracks, a few pores and asperities

Bouzakis et al. [22] investigated experimental and FEM (Finite Element Modeling) analysis of the fatigue behavior of PVD coatings on HSS (High Speed Steel) substrate in milling operation. They examined the coating failure by means of Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) micro spectral analyses. They explored the necessary parameters for FEM analysis experimentally. As a result of their study, researchers declared that performance of coatings can be predicted before and their suitability for cutting operations can be decided. Ducros et al. [23] performed a study about deposition, characterization and machining performance of multi-layer PVD coatings on cemented carbides cutting tools. Within this study, CrN/TiN and TiN/AlTiN multi-layer coatings were formed on cemented carbide tools by PVD arc evaporation device. Nano layer coatings were obtained and performance of these coatings was evaluated by cutting of Inconel 718 super alloy. Results showed that TiN/TiAlN coatings have better performance at high cutting speeds.

2.3 Deposition of Cubic Boron Nitride Films

The number of studies related with cubic boron nitride deposition has been especially increasing in the last years. Many researchers attempted to form cubic boron nitride (cBN) as thin or thick film on several substrates. Klages et al. [24] discussed deposition processes for diamond and cBN and also the textured diamond films on cutting tool inserts. In their study, they stated the difficulties such as not so high coating adhesion and restricted film thickness standing in front of cBN coatings. However, with the increasing number of research & development work, these main problems can be overcome.

Audronis et al. [25] presented recent technological developments and improvements in deposition of cBN coatings. It is reported that cBN does not react with ferrous metals, can be deposited in thin film form under standard conditions (1 bar, 298 K) and has a high resistance to oxidation in air at high temperatures. Furthermore, the handicaps of cBN films, like high compressive stresses in cBN thin films and poor adhesion strength of the interface due to high stresses occurring in strong ion bombardment were discussed in their study. At the end, they have also mentioned the technological developments in deposition of cBN coatings.

Cubic boron nitride films have been used in industrial applications in recent years. However, cBN films should have enough industrial quality which is affected by kinetic energy, chemical nature of substrates and temperature during the deposition. Bello et al. [26] indicated that carbon including amorphous carbon, nano crystalline and polycrystalline diamond provides deposition of stable, thick and adherent cBN films. They also said that as the deposition temperature increases the quality of the cBN films also increases. Bewilogua et al. [27] analyzed growth and characterization of thick cBN coatings on silicon and tool substrates. In their study, thick cBN coatings were sputter deposited on silicon substrates using a boron nitride target. After boron nitride interlayer, nitrogen content was increased gradually and cBN films up to 2 µm were grown. However, for different substrates like cemented carbides, some modifications in the process should be required. Good adhesion and stability in humid air were observed on both coatings. Coatings were tested in turning operation and comparisons with other cutting materials were carried out in terms of wear.

Gimeno et al. [28] performed experiments about the adhesion of cBN films. They employed hexagonal boron nitride (hBN) plate as a target material and deposited cBN films by RF (Radio Frequency) magnetron sputtering technique. During the deposition process, pure Argon %85, Ar + %15 N_2 and Ar + %10 < H_2 < %30 gases were supplied to the system. In order to improve the stability of the films, H_2 was added. The best results were obtained at %18

H_2 ratio. Moreover, they also tried % 50 Ar + % 50 N_2 gas ratio for the cBN film formation. When they analyzed the films, it was seen that films exhibited longer lifetimes and less delamination.

Keunecke et al. [29] attempted to form boron including coatings on cutting tools by reactive sputtering technique. B-C-N and Ti-B-N films were deposited from Ti, TiB_2/BN and B_4C target materials. The power applied to the target materials were varied between 3 and 8 KW. N_2 and C_2H_2 were utilized as reactive gases. As a result a 3 µm multilayer coating system with a 0.5 to 0.8 µm cBN top layer was formed (Figure 2.5).

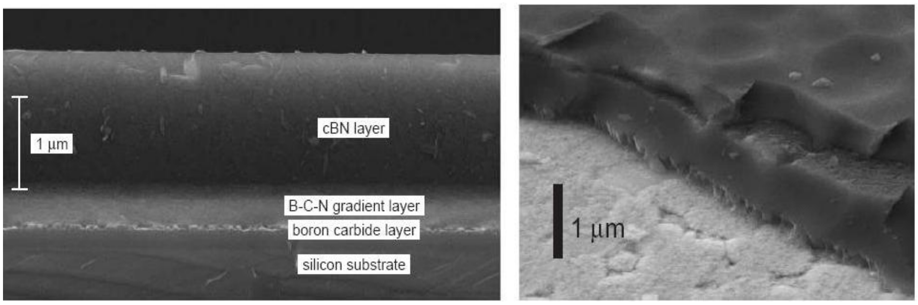


Figure 2.5 SEM images of cBN layer system on silicon (left), cBN layer system on cemented carbide substrate (right) [29]

Ye et al. [30] synthesized cBN films from a hBN target material on silicon substrates by RF magnetron sputtering. They observed the formation of cBN films after the ion bombardment of substrate at 300 ºC. Furthermore, they thought that if deposition parameters were attentively controlled, above %90 formation of cBN can be seen. Yamamoto et al. [31] investigated the deposition of cBN films up to 2 µm thickness in the B-C-N gradient system. Films were deposited by RF diot sputtering system with an auxiliary magnetic field and boron carbide (B_4C) target plate was employed. In order to obtain thick and stable films, inter layers including B, C, and N were utilized. Tzeng and Zu [32] deposited cBN films on silicon substrates in argon environment by RF magnetron sputtering. Deposition process was supported by electron bombardment. They claimed that cBN films are started to form at electron bombardment rate of 140 $mAcm^{-3}$ and at temperatures of 750 ºC and above. In addition, they discussed the effect of electron current density and argon pressure on cBN formation. Jiang et al. [33] also obtained cBN films on silicon substrates from sintered hBN target by RF magnetron sputtering. They adjusted gas ratio as %90 argon and %10 N_2. In the end, they stated that a two-step process was required for cBN formation at a lower substrate voltage and this two-step process reduced residual stress of the films significantly. Bewilouga et al. [34] tried to deposit thick cBN films on silicon using boron carbide target material. However, they claimed that for different substrates such as cemented carbide, some modifications in the deposition process would be required. They observed that coatings deposited on both silicon and cemented carbide exhibited high adhesion and stability in humid air (Figure 2.6).

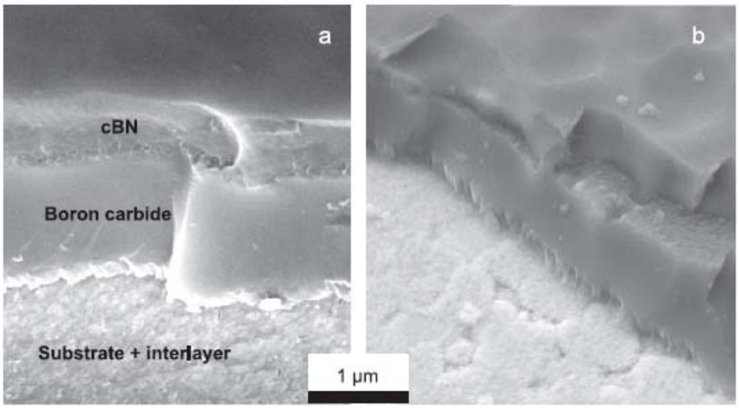


Figure 2.6 SEM images of cross sections of boron carbide-cBN on high speed steel (a), cemented carbide(b) substrates [34]

Jiang et al. [35] developed cBN-TiN nano composite coatings on cutting inserts for hard turning operation. They used a two-stage process; electrostatic spray coating (ESC) and chemical vapor infiltration (CVI). They discussed aspects of the coating on chip-breaker inserts, repeatability and process optimization. According to machining tests, tool life for each cutting edge is found to be 20 min against AISI 4340 steel material at cutting conditions; 150 m/min cutting speed, 0.15 mm/rev feed and 0.25 depth of cut.

Similarly, Russel et al. [36] synthesized cBN-TiN using ESC-CVI novel combinatorial method. Electrostatic spray coating is used to deposit cBN regularly on cutting tool surface. On the other hand, chemical vapor infiltration is used to keep particles together on the tool surface. They optimized process parameters so that dense cBN particles dispersed in TiN matrix. Metal cutting tests proved that tool life increased three to seven times in comparison with CVD coated TiN films in turning of 4330 steel material. Djouadi et al. [37] considered growth of boron nitride films by PVD method. Transition from hexagonal to cubic phase by PVD method was carried out and boron nitride films are deposited successfully on substrates. High internal stress because of ion bombarding in the formation of cBN films by PVD cause less adhesion between the substrate and film. [25] Therefore, there are many attempts to produce low stress cBN film. Zhu et al. [38] succeeded to deposit cBN films on metallic substrates by tuned substrate r.f. magnetron sputtering. When the films were characterized by transmission electron microscopy, it was seen that the surface of the film was pure cBN which has low stress and excellent adhesion.

Bello et al. [39] studied the deposition of thick cubic boron nitride films. They claimed that the interfacial soft hexagonal/amorphous boron nitride layers and large stress associated with cBN deposition can be overcome by reduction of ion energy, deposition at elevated temperatures, post-deposition treatment, use of buffer layers and introduction of halogen chemistry. As a result of their research, they obtained thick, epitaxial cBN films that already meet the requirements for many tools and tribological applications.

Deng et al. [40] tried to grow cBN films from hot-pressed hexagonal boron nitride target by RF sputtering. With the appropriate deposition conditions, boron nitride films containing nearly pure cBN were obtained in their experiment. As a conclusion of their study, they said that appropriate energy flow density of positive ions impinging on substrate for formation of cBN should be necessary. Zhou et al. [41] mentioned the formation of the cubic boron nitride films on nickel substrates by RF magnetron sputtering. According to experiments, nickel can be used as a substrate material for the high quality and well-adherent cBN films.

Cubic boron nitride films can also be formed by different techniques such as chemical vapor deposition. Nonogaki et al. [42] investigated about preparation of boron nitride films by multi-source plasma CVD method. In this study, BF_3 and NH_3 gases were used to deposit boron nitride films in the multi-source CVD technique. In this technique the gases were first excited and introduced to the front of the substrate. At the same time above the substrate, spread sheet-like plasma which was generated by the arc-plasma gun under magnetic field and excited gases is formed. At the end, boron nitride film was deposited on the substrate. Moreover, it was aimed to investigate the effect of substrate self-bias to accelerate ions towards the growing surface on the structure of BN films. Furthermore, Yang et al. [43] studied high-quality cBN thin films prepared by plasma chemical vapor deposition with time-dependent biasing technique. They investigated the influences of argon flow rate and the time-dependent biasing condition on the composition and the transition layer thickness of cBN films in their study.

They also proved that by using proper techniques high quality cBN films can be prepared by chemical vapor deposition similar to prepared by physical vapor deposition.

2.4 Cubic Boron Coated Cutting Tools

Uhlmann et al. [44] discussed the application of cBN coated cutting tools. They tried various layers of boron coating on TiAlN and varied the thicknesses in order to reach the highest film adhesion and wear resistance (Figure 2.7). Performances of the coatings were tested by interrupted and uninterrupted turning and milling operations.

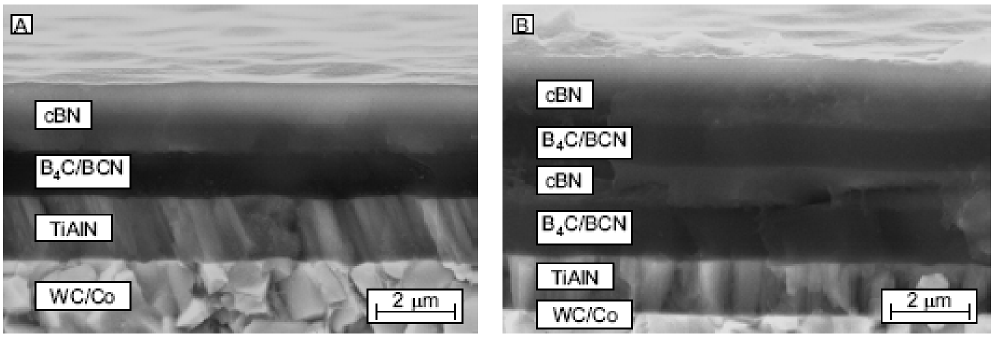


Figure 2.7 SEM images of cBN layer system [44]

Next, wear mechanisms of the coatings were compared with TiAlN coated tools. According to the test results, it was seen that cBN coated tools exhibited twice hardness in comparison to TiAlN coated tools which results an increase of tool life about %70. Furthermore, cBN coatings diminished cutting forces and improved the surface roughness. Jiang et al. [45] investigated the performance of cBN-TiN coated composite carbide tools in hard turning operation. In order to obtain this composite coating system, they used electrostatic spray coating (ESC) and chemical vapor infiltration

(CVI) techniques. Surface morphology, chemical composition, micro hardness, adhesion and wear of the films are examined. Characterization analysis indicated that relatively uniform coating with continuous cBN particle dispersion in an adherent TiN matrix was formed. Next, cBN-TiN coated tools are utilized in machining of AISI 4340 steel material which has a hardness of 52 HRC under machining parameters of $V = 100$ m/min., $f = 0.2$ mm/rev, DoC = 0.5 mm, and dry conditions. They claimed that cBN–TiN coated tools demonstrated a tool life of about 10 min per cutting edge and cBN-TiN coated tools can be complementary to PcBN tools. Jedave et al. [46] used the same technique which was stated above to obtain cBN-TiN composite coatings. They showed that electrostatic spray coating (ESC) and chemical vapor infiltration (CVI) techniques can be applied on tungsten carbide cutting tools to obtain multilayer coatings with a thickness in excess of 10 µm. Moreover, they tested an industrial electrostatic spray coating unit, which can coat a batch of about 50 inserts in a couple of minutes. Arunachalam et al. [47] examined residual stresses and surface roughness of cBN and ceramic cutting tools while machining Inconel 718 material. They also investigated the effect of cutting speed, depth of cut, tool nose geometry and coolant usage on residual stresses and surface roughness. According to the results, it is concluded that cBN inserts exhibit minimum residual stress and better surface roughness in machining of Inconel 718 material under cutting conditions of $V = 150$ m/min, depth of cut of 0.05 mm and wet machining. Keunecke et al. [48] analyzed cutting performance of thick cBN films on cemented carbide tools in turning and milling operations. Initially, they formed cBN on TiN and TiAlN cemented carbide cutting tools and they determined mechanical and tribological properties of the coating system. After that, they tested coated tools in turning and milling. They found that cBN coating have lower flank wear which resulted an increase in tool life in turning operation (Figure 2.9). However, they claimed that tool life is reduced in milling operation because of insufficient adhesion.

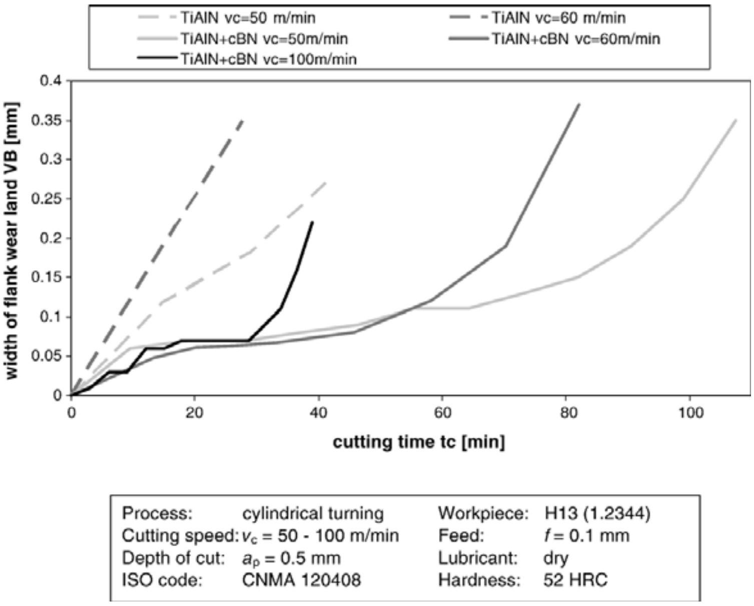


Figure 2.8 Flank wear of various coated cutting tool in turning operation [48]

More et al. [49] compared cBN-TiN coated carbide tools with PcBN tools in terms of tool wear and cutting performance in machining of hardened AISI 4340 steel. They analyzed the effect of cutting speed and feed on tool life, surface roughness and cutting forces using ANOVA technique. They determined the optimum values as depth of cut of 0.25 mm, cutting speed of 100-125 m/min and feed of 0.15-0.20 mm/rev for cBN-TiN coated carbide tools. When tool lives are compared, it is observed that cBN-TiN coated carbide inserts have 18-20 min tool life whereas PcBN tools have 32 min per tool. Neo et al. [50] examined the performance of pure cBN cutting tools in machining steel. Pure cBN and binderless cBN are compared in terms of surface roughness and tool wear. They made a feasibility analysis about direct utilization of pure cBN tools in ultra-precision machining. According to the results of their investigation, pure cBN tools are better than binderless cBN tools in terms of wear resistance and surface roughness.

CHAPTER 3

OBJECT OF PRESENT INVESTIGATION

In this thesis, an experimental study about cubic boron nitride coating formation for cutting tools is proposed. One of the three primary objectives of this study is to establish a new laboratory for research and development. In this laboratory, the experimental set up which consists of a physical vapor deposition system and some auxiliary devices are located. The important point here is that, the physical vapor deposition system is completely designed and manufactured by a local company in Turkey. Detailed information about the experimental setup will be given in the following chapters. Second objective is to form cubic boron nitride coatings on sample substrate surfaces and cutting tool surfaces for applications such as dry machining and high speed machining. Upon achieving the second objective, as a third objective further studies were carried out to implement that process to industrial applications. For that purpose, collaborations not only from universities but also from companies in industry have been attempted.

Despite the fact that Turkey has the largest amount of boron reserve in the world, number of studies related with boron or boron related products, is not so high. Therefore, researches about production of boron and boron related products should be encouraged and increased.

This graduate study is a multi disciplinary study in which primary branches such as mechanical engineering, material & metallurgical engineering, surface engineering, tool engineering, chemistry and physics are included.

CHAPTER 4

THEORY

4.1 Cubic Boron Nitride (cBN)

Cubic boron nitride was discovered in 1957 and it is very popular due to its mechanical and chemical properties in the market. CBN is a ceramic material and is composed of boron and nitrogen elements which make covalent bond. Cubic word indicates the phase of the crystal structure of the boron nitride. The most two common crystal forms of boron nitride can be seen in Figure 4.1:

- Hexagonal (graphitic) — a soft, white, lubricious powder
- Cubic (diamond structure) — hard, amber or gray colored, abrasive

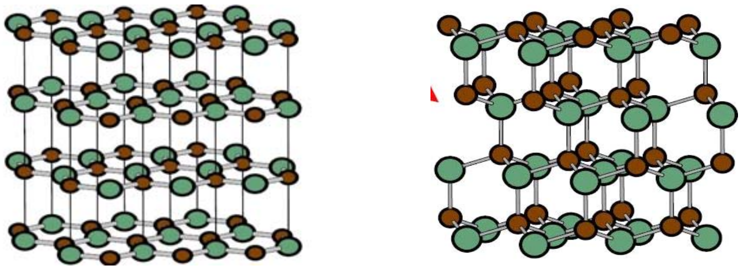
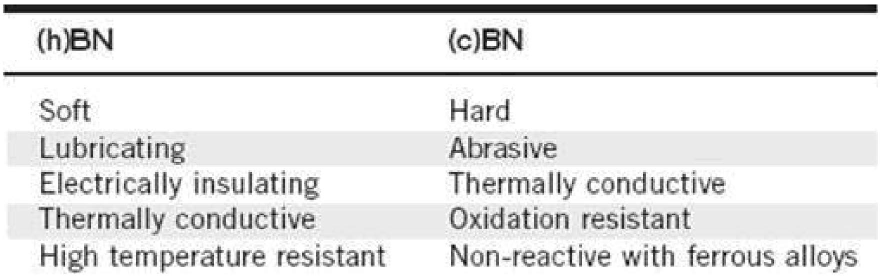


Figure 4.1 Lattice structures of hBN and cBN [51]

Boron and nitrogen are adjacent to carbon in the periodic table of elements. In combination B and N have many similarities to carbon. Both carbon (C) and boron nitride (BN) have hexagonal (graphitic) and cubic (diamond) crystal structures. Hexagonal boron nitride (hBN) is a soft lubricious material while cubic boron nitride (cBN) is second only to diamond in hardness and is commonly used as an abrasive. Two most common phases of the boron nitride are compared in Figure 4.2.



(h)BN	(c)BN
Soft	Hard
Lubricating	Abrasive
Electrically insulating	Thermally conductive
Thermally conductive	Oxidation resistant
High temperature resistant	Non-reactive with ferrous alloys

Figure 4.2 Comparison of hexagonal and cubic boron nitride [52]

4.1.1 Physical and Chemical Properties of Cubic Boron Nitride

Cubic boron nitride (CBN) is the second hardest known material, only diamond being harder (Figure 4.4). Moreover, in comparison to diamond it is more stable at the high temperatures (Table 4.1). CBN has the same crystalloid structure with diamond; therefore it also has similar mechanical properties to diamond. The high mechanical strength of cBN is because of its excellent internal structure and isometric material grains. The elastic modulus of cBN is almost twice of the other abrasive materials but its thermal expansion coefficient is about half of the others materials. Thus, cBN has more resistance to thermal deformation.

CBN has very stable characteristics compared to other machining metals at high temperatures. CBN crystals are able to maintain their mechanical properties, phase structures and hardness up to 1200-1300 C°. Another property of cBN is that, it is not active to chemical reactions especially with metals. Its abrasive and reaction resistance properties are very useful and it does not wear out as diamond does at high speeds. Unlike diamond and SiC which are popular in metal cutting, cBN does not react with ferrous metals and it can cut ferrous metals having hardness of 50RC and high content nickel-cobalt alloys. Some examples of cBN products can be seen in Figure 4.3. Main properties of cubic boron nitride are listed below [5];

- High Hardness
- Fair thermal conductivity
- High Wear Resistance
- Stable at high temperatures (up to 1200-1300 °C)
- Does not react with metals
- Resistance to oxidation
- Low friction coefficient

Figure 4.3 Some examples of cBN products

Cubic boron nitride (CBN) has been used as a cutting tool material recently due to its excellent mechanical and chemical properties in applications such as dry cutting, high speed machining and cutting of hard materials [7,8]. Main drawback of cubic boron is its high cost. Sintered cubic boron nitride cutting tools are available in the market and these tools have already been used in defense, automotive, aerospace and manufacturing industries extensively. However, there are geometrical constraints in sintering operation and it is not possible to form sintered tools into various tool shapes. In addition, poor ductility and high cost of sintered tools are stated as their disadvantages [9].

Figure 4.4 Comparison of materials in terms of Knoop hardness [52]

Table 4.1 Comparison of cBN with other tool materials

Property	Steel	Cast Alloy	WC	TiC	C-BN	Diamond
Hardness (Gpa)	8.5	8	14-24	18-32	40-50	70-80
Strength (Mpa)	4100-4500	1500-2300	4100-5850	3100-3850	6900	6900
Elastic Modulus (Gpa)	200	200	520-600	310-450	850	820-1050
Density (g/cm^3)	8.6	8-8.7	10-15	5.5-5.8	3.48	3.5
Melting Temperature(°C)	1300	-	1400	1400	1300	700
Thermal Conductivity (W/m°C)	-	-	42-125	17	13	70
Thermal Expansion Coefficient (x10^{-6}/°C)	12	-	4-6.5	7.5-9	4.8	1.2

4.1.2 Synthesis of Cubic Boron Nitride Material

4.1.2.1 Synthesis of cBN as a bulk material

The chemical combination of boron and nitrogen does not occur in nature. The fabrication of boron nitride as a bulk material is possible at high temperatures and pressure. Synthesis of cBN can be done at nearly a temperature of 1470-1720 K and pressure of 6 GPa. [53] Under these conditions, atomic structure changes from hexagonal to cubic and the transformation of hBN to cBN can be achieved. If hBN is of small particle size and poorly crystallized, polycrystalline and fine-grained cBN is obtained. This method has been performed on laboratory scale and produces cBN of highest purity and quality. However, catalyzed transformation should be carried out for industrial scale by inserting additives which results in cBN powder of small size with crystals of tetrahedral or octahedral. Another alternative method called static high pressure uses a different temperature (1890 K to 1540 K) in a powder filled reactor to grow crystals from cBN seeds. This process is very slow but larger crystals of cBN can be synthesized.

Hexagonal boron nitride is required as a starting material for the production of cBN. The first synthesis of hBN was made in 1842 by W. H. Balmain using the action of molten boric acid on potassium cyanide (Figure 4.5). Unfortunately, the new compound was unstable. Researchers were able to create more stable material in the form of powders and hot-pressed shapes in 1950s. By new processing technology boron nitride has become an economically affordable advanced material and is now used in various forms in a multitude of applications [52].

Aydogdu [54] tried to form boron nitride by carbothermic formation method in his dissertation. In this method, formation of boron nitride by reaction of boric oxide with carbon and nitrogen was obtained. B_2O_3 and activated carbon mixtures were held under flowing nitrogen atmosphere in the experiments. Then, the formation of boron nitride was observed in 120 min at 1500 °C. As a result of their experiment, they found that reaction proceeds through a gaseous boron containing B_2O_3.

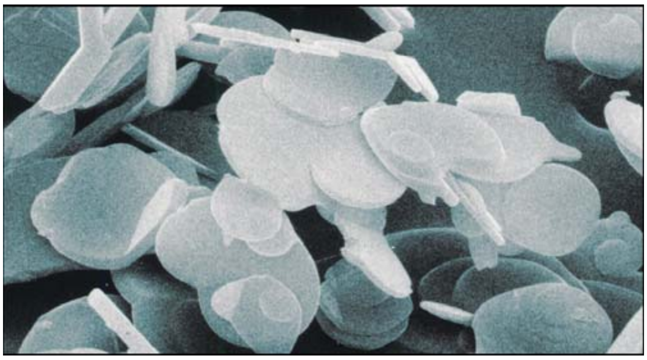


Figure 4.5 hBN typical crystal view about 10μm

Another method of formation of boron nitride is the plasma process. Kostic et al. [55] investigated optimal plasma routes for boron nitride (BN) powder production from boric acid. In this process, firstly boric acid powder is vaporized and then decomposed in the nitrogen plasma and B-O-H-N system is formed. Next, methane is sprayed on this system and boron nitride powders are obtained between suitable temperatures. (1200°K<T<2600°K) In the industrial formation of boron nitride powder, borates like boric acid or borax are heated with compound that includes ammonium or urea and form B-N bond. In order to obtain hexagonal boron nitride processes should be followed in the order of mixing, nitriding, grinding, washing, drying, crystallizing and re-grinding.

4.1.2.2 Synthesis of cBN as a thin film

Demand to thin or thick film deposition of boron nitride has grown up rapidly and many researchers have attempted to synthesize boron nitride coatings in cubic phase. CBN films have been used as a wear resistant coating on cutting tools, a protective coating, an optical coating and an electrical insulating layer. There are several ways to synthesize cBN as a thin film. Basically, thin film deposition techniques can be described as any technique for depositing a thin film of material ranging from fractions of a nanometer to several micrometers in thickness onto a substrate or onto previously deposited layers.

Deposition techniques can be classified as physical vapor deposition (PVD) and chemical vapor deposition (CVD). CVD is based on chemical reactions whereas PVD processes (often just called thin film processes) are atomistic deposition processes in which material is vaporized from a solid or liquid source in the form of atoms or molecules. The evaporated material is transported in a low pressure or plasma environment to the substrate where it condenses and forms thin layers of films. Comparison of two processes is given in the Table 4.2.

Table 4.2 Comparison of PVD and CVD

Physical Vapor Deposition (PVD)	**Chemical Vapor Deposition (CVD)**
Performed in a vacuum chamber (10-2 to 10-4 Torr)	Can be performed in controlled atmosphere or vacuum
Relatively low process temperature (90° to 450° C)	High temperature process (1050°C)
Line of sight process: will coat areas directly exposed to ion source	Reactive gas process coats wherever atmosphere contacts tool surface
Coating exhibits a physical bond to the substrate's surface	Coating exhibits a chemical & metallurgical bond to the substrate
Average thickness: 2-5 µm	Average thickness: 6-10 µm
Suitable for a wide range of substrates	More limited range of substrates than for PVD
Ideal for closely toleranced components (+/- 2,5 µm is appropriate)	Requires relatively loose tolerances (ex:+/- 13 µm per 25 mm diameter)
No heat-treating required after coating due to low process temperature	Post-coating heat-treating required on steel parts due to high process temperature
Good for sharp edges: no excessive coating build-up	Requires hone on sharp edges due to heavier coating build-up
Coating will generally replicate existing surface finish - mirror finishes can be maintained	Difficult to maintain mirror finish (post-coating polishing will improve finish)

There exist various types of PVD and CVD methods depending on the application and coating material. They are listed below.

Physical Vapor Deposition

- Evaporation
    - Thermal Evaporation
    - E-beam Evaporation
- Sputtering
    - DC Sputtering
    - RF Sputtering
- Ion Plating

Chemical Vapor Deposition

- Low-Pressure CVD (LPCVD)
- Plasma-Enhanced CVD (PECVD)
- Atmosphere-Pressure CVD (APCVD)
- Metal-Organic CVD (MOCVD)

PVD has more advantages over CVD like lower deposition temperature, possibility to deposit thinner coatings and possibility to deposit sharp edges and complex forms. Therefore, PVD is a more suitable process for coating steel and some other metallic substrates since lower coating temperature is required. In the next part, detailed information about physical vapor deposition process is given.

4.2 Physical Vapor Deposition (PVD)

Physical vapor deposition is fundamentally a vaporization coating process in which the basic mechanism is an atom by atom transfer of material from the solid phase to the vapor phase and back to the solid phase, gradually building a film on the surface to be coated (Figure 4.6). In the case of reactive deposition, the depositing material reacts with a gaseous environment of co-deposited material to form a film of compound material. PVD coatings are widely used in applications such as aerospace, surgical/medical components, die and moulds for all manner of material processing, cutting tools, fire arms and decorative coating [53, 56].

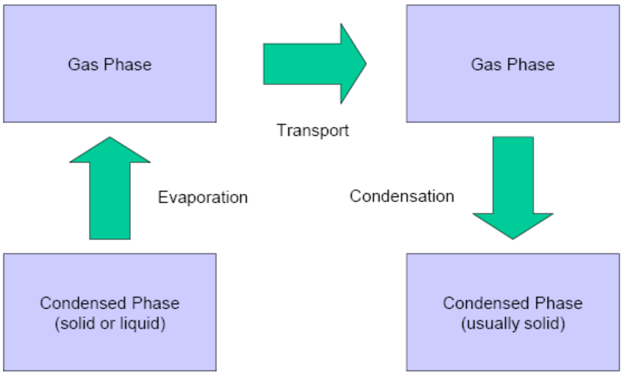


Figure 4.6 Schematic diagram of Physical Vapor Deposition process

Main types of PVD techniques are evaporation, sputtering and ion plating and all types utilize the same three fundamental steps to develop a coating. These steps are evaporation (vapor phase generation from source material), transportation (transfer of the vapor phase from source to substrate) and condensation (deposition and growth of the film on the substrate). For reactive deposition, source material reacts with the supplied gas before the last step and forms the thin film/coating.

Advantages of Physical Vapor Deposition

- Materials can be deposited with improved properties compared to the substrate material.

- Almost any type of inorganic material can be used as well as some kinds of organic materials.
- The process is more environmentally friendly than traditional coating processes such as electroplating.
- PVD coatings are generally harder and more corrosion resistant than coatings applied by the electroplating process. Most coatings have high temperature and good impact strength, excellent abrasion resistance and are so durable that protective topcoats are almost never necessary.

Disadvantages of Physical Vapor Deposition

- It is a line of sight technique meaning that it is extremely difficult to coat undercuts and similar surface features.
- Some PVD technologies typically operate at very high temperatures and vacuum requiring special attention by skilled operators.
- Requires a cooling water system to dissipate large amount of heat.
- The rate of coating deposition is usually quite slow.
- High capital costs.

4.2.1 PVD System

Basically, there are four elements in a PVD system. These are listed below and shown in Figure 4.7.

a) A vacuum chamber: Evacuated with a pumping unit (i.e., roughing pump and diffusion or turbo-molecular pump).

b) A vapour source: (i.e., target).

c) A medium: Usually an argon based plasma (except for thermal evaporation techniques).

d) A receiving area: (i.e., substrates). Generally, and because of the directivity of the vapour flux, the parts / substrates to be treated are located opposite to the vapour source.

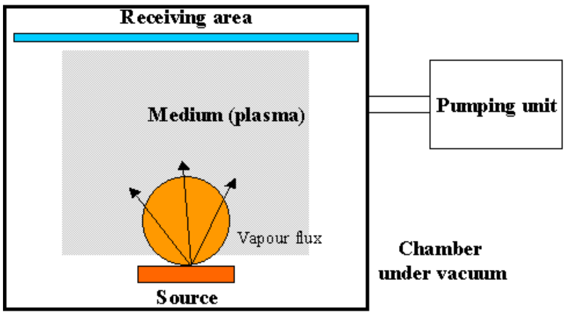


Figure 4.7 Schematic representation of a PVD Device

4.2.2 PVD Techniques

For the whole PVD processes, the vacuum chamber is firstly evacuated to a pressure as low as 10-4 / 10-6 Pa, and then backfilled with the appropriate partial pressure of a neutral gas, usually argon. Several reactive gases, such as oxygen, nitrogen or acetylene can be supplied to the chamber during the deposition process to allow the synthesis of ceramic compound (i.e., oxides, nitrides or carbides and all the possible combinations). According to the way of production of the material vapour, PVD techniques can be classified as follows.

4.2.2.1 Evaporation

In this technique, vapours are produced in a vacuum environment from the source material which is heated by various methods. Resistance, induction, arc, electron beam, or lasers are the possible heat sources for evaporation [53]. Evaporation takes place in vacuum, in a pressure range of 10^{-5}-10^{-10} Torr and in this pressure range, the mean free path (MFP) is very large ($5x10^2$-10^7 cm) which means that evaporated particles can travel directly to the deposition target without colliding with the background gas. By evaporation, very high purity coatings can be produced with a very low deposition rate. However, according to the low energy of the particles impinging the substrates, the coatings are usually porous and poorly adhesive. Post treatments of the coatings are generally needed to improve their properties.

4.2.2.2 Sputtering

A schematic picture of a sputter system can be seen in Figure 4.8. The system consists of high vacuum chamber to ensure a contamination free environment, a pumping system to reach a low pressure, a working gas inlet (generally argon), a substrate holder with a heater and target consisting of the material to be deposited. In the basic process a negative potential is applied to the targets, which results in ionisation of argon. The positive argon ions are then accelerated towards the target, where material is removed through momentum transfer. This phenomenon can be expressed as atomic billiards and shown in Figure 4.9.

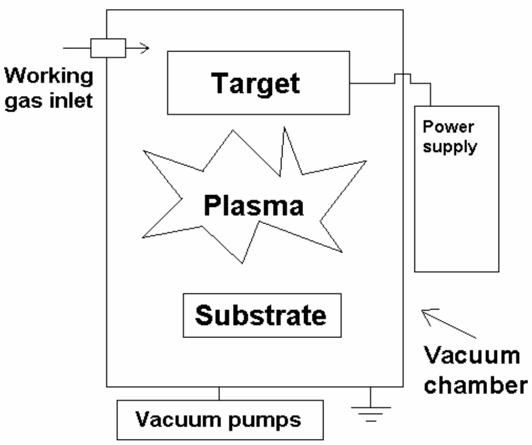


Figure 4.8 Schematic view of a sputter system

For all of the sputtering techniques, argon based plasma must be formed by means of a discharge initiated between two electrodes: the source or target (negative) and the chamber wall (grounded). The argon ions (Ar^+) created in the plasma impact the source material (target) with energy proportional to the potential difference between the plasma and the target. Since the energy of the argon ions is usually superior to 100 eV, the target starts to be sputtered and atoms, clusters or molecules are ejected from its surface. These particles cross the plasma with more or less collisions and reach the substrates with a residual energy to form the coating. As more than 80% of the incident kinetic energy of the argon ions is turned into heat during sputtering, the target has to be water cooled. In sputtering techniques, the target can be powered by DC or RF (Radio Frequency) current in order to initiate plasma. The RF power is usually implemented to sputter dielectric materials without growing defects. Additionally, substrates to be coated can also be powered in order to increase the density of the growing film (Figure 4.10).

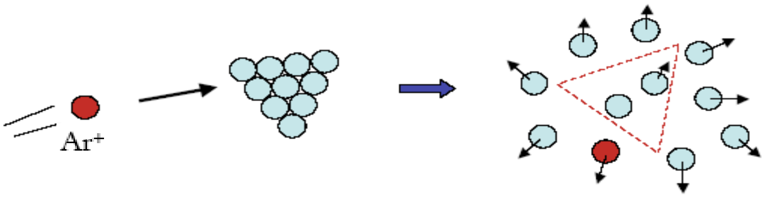


Figure 4.9 Atomic billiards

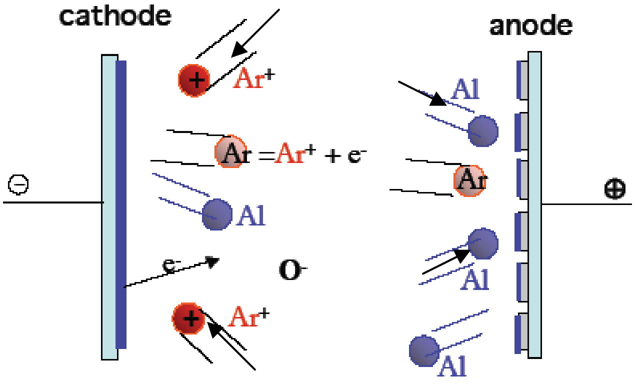


Figure 4.10 Sputtering process

Types of sputtering techniques can be categorized as diode, triode and magnetron sputtering [53].

a) Diode Sputtering

In this technique, relatively high argon pressure (4 to 15 Pa) is required to initiate and sustain the ionisation and plasma formation between the two electrodes. This high pressure plasma also increases collision probability and scattering of the sputtered atoms during their travel between the target and the substrate. The deposition rate associated to the diode sputtering technique is fairly low and in the range of 1 mm per hour for metallic coatings.

b) Triode Sputtering

In order to increase the electron density of the plasma, a thermo-emissive tungsten (or refractory metal) filament coupled with water cooled anode is utilized in the sputtering chamber. As a result, these additional electrons will increase the ionisation probability of argon and its pressure can significantly be reduced and higher quality coatings can be synthesized. Furthermore, the density, the adhesion and the deposition rate of the coatings are improved in triode sputtering method.

c) Magnetron Sputtering

In this technique, a magnetic field, mainly parallel to the target surface, is superimposed to the applied electric field so that the secondary electrons (emitted by the target during its bombardment) are trapped near the target surface. By employing magnetic fields, ionization efficiency at the target surface and the deposition rate can be greatly improved. As the plasma can be sustained at low pressure, magnetron sputtering also produces dense, adherent and high quality coatings. Cylindrical or planar magnetrons can be used. Even though magnetron sputtering technique has advantages such as ionization efficiency and high deposition rate, erosion of the target is not uniform due to magnetic field applied and that leads to a utilization of the target in the range of 30 %. Moreover, there is a decrease of the deposition rate with time. Additionally, target should be prepared without voids or bubbles to avoid local melting and spitting since high power rate is applied to the magnetrons [53, 57].

CHAPTER 5

EXPERIMENTAL SETUP

5.1 BOR-N PVD SYSTEM

Experimental setup consists of a physical vapor deposition system (PVD) which works on magnetron sputtering principle and some auxiliary components such as an ultrasonic cleaning machine, a generator, a cooling system an uninterruptible power supply and a compressor. The physical vapor deposition system is completely designed and manufactured by a local company in Turkey (Figure 5.1 & 5.2).

Figure 5.1 BOR-N PVD System -View 1

The experimental system is utilized to obtain mono or multi layered coatings on different substrates. It can work under high vacuum. It has two magnetrons so that both metallic and reactive sputtering can be done. Substrates can be biased by a RF power supply and they can also be exposed to microwave for surface modification if necessary. Substrates are mounted on a spindle mechanism and can be rotated by an electric motor. Temperature inside the vacuum chamber can be also controlled.

Figure 5.2 BOR-N PVD System -View 2

PVD System Components are vacuum chamber, 19” control panel, gases and computer. Auxiliary components are generator, compressor, cooling system, ultrasonic cleaning machine, and uninterruptible power supply.

5.1.1 PVD SYSTEM COMPONENTS

5.1.1.1 Vacuum Chamber

The most significant part of the PVD system is the vacuum chamber in which the deposition process takes place (Figure 5.3). In order to withstand high vacuum and to have less damage from sputtering, walls of the chamber are made of stainless steel. One DC and one RF magnetron are located inside of the chamber and different target plates can be attached to these magnetrons. DC magnetron is used for metallic sputtering whereas RF magnetron is used for reactive sputtering. The pumping unit which is composed of a mechanical and turbo molecular pump, is used to evacuate the chamber to very low pressures. Initially, mechanical pump is operated and pressure is reduced from atmospheric pressure to 10^{-3} Torr. After that, turbo molecular pump is started. Turbo molecular pump can rotate at a speed of 35 000 rpm and pressure is decreased to 10^{-7} Torr in a few minutes.

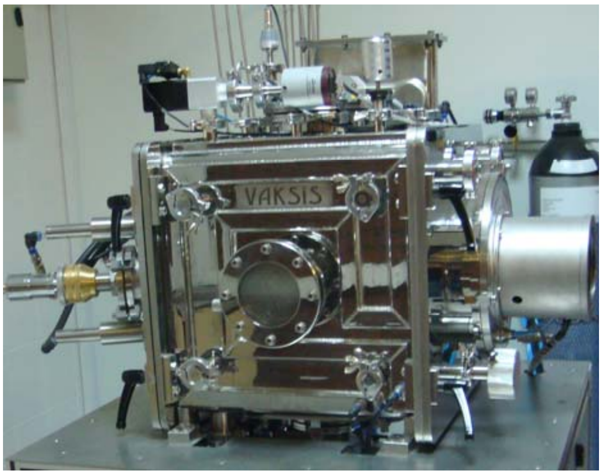


Figure 5.3 Vacuum Chamber

Substrates are mounted on a rotary spindle mechanism inside of the vacuum chamber and can be rotated by a PC controlled electric motor (Figure 5.4). Substrates can make both planetary rotation and axial rotation. Rotation is not needed for flat surfaces but it is necessary to obtain homogenous thickness on the surface of cylindrical cutters. Three different power supplies; DC, RF and microwave are used in the system. DC and one RF power supplies are employed for sputtering process. Second RF power unit is connected to spindle and substrates can be excited during plasma cleaning. Alternatively, there is a microwave power supply which may be used for plasma cleaning also. DC power supply is 300 W, RF1 is 600 W, RF2 is 1200 W and micro wave power supply is 1400 W.

Figure 5.4 Rotary Spindle Mechanisms

Two vertically located heaters are utilized to keep system at desired temperatures up to 400°C. Heater temperature is measured by a thermocouple which is attached to heaters and temperature can be set by a PID controller (Figure 5.5). Water cooling is applied to magnetrons because of excessive heating during the experiment. For that purpose, a new cooling system which works with pure distilled water is designed and produced also. During the experiments, various gases are supplied to the vacuum chamber to generate plasma and to deposit coatings in desired thicknesses.

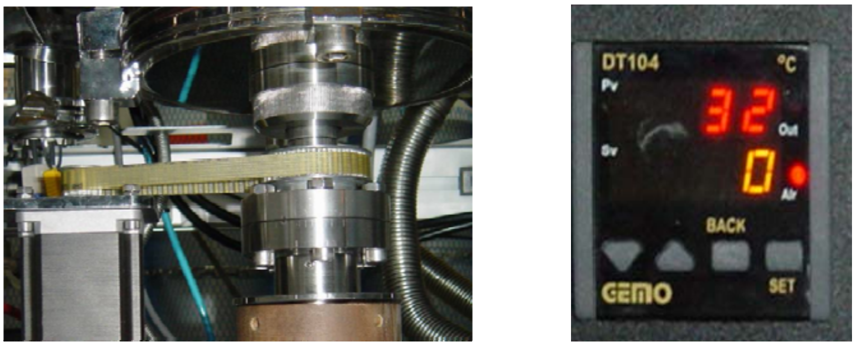


Figure 5.5 Electric Motor & Temperature Controller

5.1.1.2 Control Panel

Next component of the PVD system is the control panel on which many fuses, on/off buttons, pressure gauges, valves, gas flow control unit, computer and several power supplies are mounted. Pumps, heaters, valves, gas flow and power supplies are controlled and experiment parameters are adjusted to desired values from the control panel to get better results. Detailed view of the control panel can be seen in the next photograph.

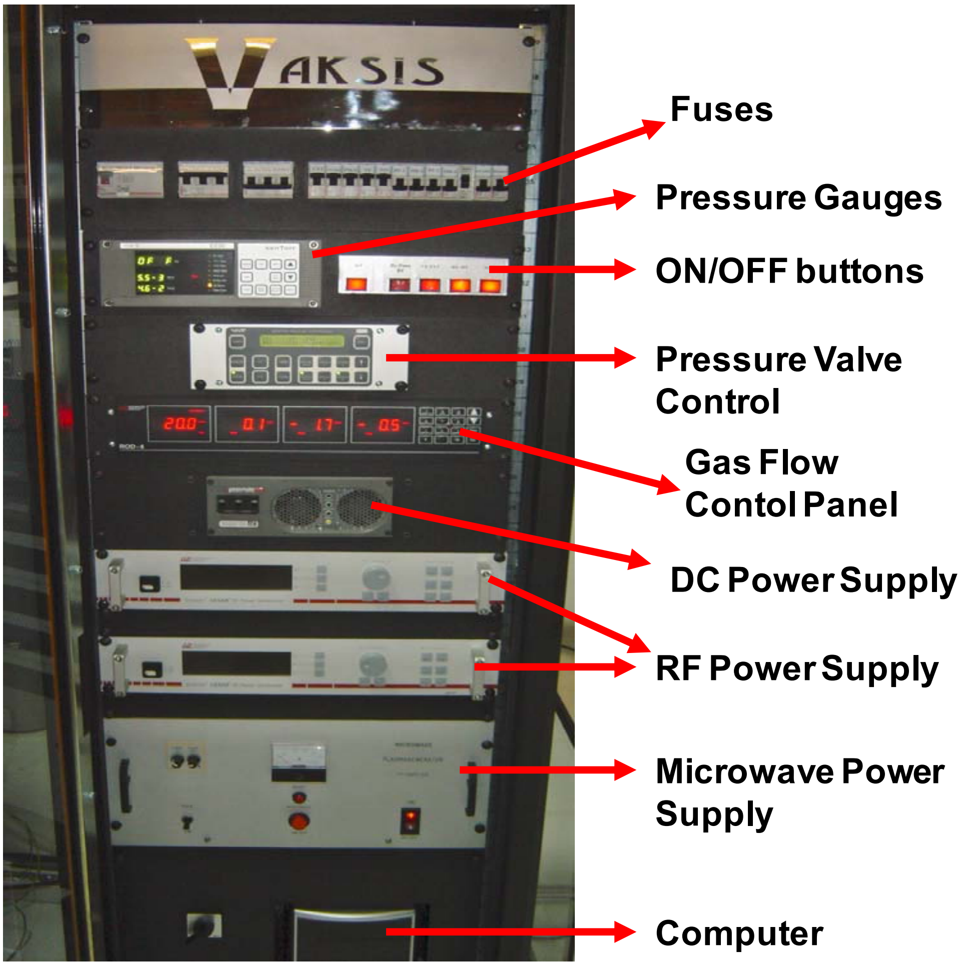


Figure 5.6 Control Panel

5.1.1.3 Gases

In the PVD laboratory, five different gases are utilized. They are Argon (Ar), Oxygen (O_2), Nitrogen (N_2), Hydrogen (H_2) and Acetylene (C_2H_2) gases. Flow of the gases is controlled by very sensitive valves and flow rate values can be adjusted from the control panel. Five gases are supplied to vacuum chamber through four inlet valves. Hydrogen and Acetylene gases use the same inlet. As a result, only one of them can be used in the same experiment. Acetylene gas is very flammable; therefore, particular attention should be paid while using it. Moreover, all gas tubes are securely fixed to wall by chains.

Figure 5.7 Gases

5.1.1.4 Computer

Computer is the last component of the PVD system which is used for controlling electric motor speed and direction to rotate the spindle mechanism. DC power unit is also run from the computer and DC power, voltage, current and other properties can be adjusted. Lastly, it is used for preparing documents such as experiment protocols which includes parameters, results, etc.

5.1.1.5 Auxiliary Components

Besides the main components which mentioned above, some auxiliary devices are also necessary for the PVD system to work properly. These auxiliary components are; uninterruptible power supply, generator, compressor, cooling system and ultrasonic cleaning machine.

a) Uninterruptible Power Supply

Experiments generally continue for a few hours and during the experiments electricity should not be interrupted. For that reason, a 20 kVA uninterruptible power supply (UPS) is utilized and electricity is fed to the system from the UPS (Figure 5.8). In case of an electricity loss, system can work for a few minutes until the generator is put into use.

b) Generator

When the electricity goes off, generator starts to work automatically. It is located in a different room and its base is isolated very well not to affect the building. Generator is 45 kVA in power and has diesel engine (Figure 5.8). The exhaust gas is discharged securely to the outside.

Figure 5.8 Generator & UPS

c) Compressor

Some of the valves in the PVD system are pneumatic. Therefore, air pressure is required to open and close these valves. Compressor is employed for that reason and can be started manually if needed.

d) Cooling System

Heaters and applied high power may damage magnetrons during the experiments. Therefore, water cooling is applied to protect magnetrons from excessive heating. At first, city water is used for cooling but the amount of lime in city water is high and that causes problems in magnetrons. For this reason, a cooling system which works by distilled water is designed and produced (Figure 5.9).The cooling system is necessary to keep the temperature of the magnetrons at safe values.

Figure 5.9 Cooling System

e) Ultrasonic Cleaning Machine

Before the experiment, each of the substrates has to be cleaned very well to get rid of dirt, rust and other surface impurities. For that reason, an ultrasonic cleaning machine is installed in the laboratory. This cleaning process is necessary to increase adhesion, improve surface quality and obtain dense and high quality coatings.

5.2 TARGET PLATES

Target plates are the plates that are attached to the magnetrons and are one of most critical parts of the system. In order to obtain cubic boron nitride (cBN) coatings, hexagonal boron nitride (hBN) target plate is utilized. Since there are two magnetrons in the system, both mono or multi layered coatings can be deposited. DC magnetron is used for metallic sputtering, so that titanium target plate is mounted. However, RF magnetron is used for reactive sputtering, therefore; target plates such as aluminum and boron nitride can be attached. Magnetrons are 100 x 250 mm in dimension. Thus, target plate dimensions should not exceed these values.

Figure 5.10 Titanium and Aluminum Target Plates

Target plates in Figure 5.10 are used to obtain TiN and TiAlN coatings. There is a path formed on the surface of the plates because of sputtering. The shape of the path is due to the magnetic field effect generated by the magnets inside of the magnetron. High powers are applied to the plates during the experiments which may cause target plates to crack or fracture. Therefore, cooling of the plates is essential. After every experiment, plate surfaces are cleaned by alcohol to prepare them for the next experiment.

5.2.1 Boron Nitride Target Plates

One of the key points of this study is to develop boron included target materials. Therefore, boron nitride target plates are produced in Middle East Technical University and Eskişehir Anadolu University. Moreover, one target plate is purchased from abroad. Unfortunately, comparison of the target plates could not be made in this study. As a future work, target plates performances will be compared.

a) Production of alloyed boron nitride target plates in METU facilities

Firstly, target plates are planned to be produced in dimensions of 50 x 100 mm and a die is designed and produced according to that dimension as can be seen in Figure 5.11. After that, Prof. Dr. Ali Kalkanlı developed alloyed Al-B-Ti-Mg-hBN material. For that purpose, hBN powders obtained from 6 th Element company in South Africa and Anadolu University are used. Moreover, titanium particles and magnesium are also included in this alloyed material. Composition of the alloyed material is as follows; % 1 of boron, % 5 of titanium and remaining is aluminum.

Then, alloyed mixture is put into the die in Figure 5.11 and squeezed in a vertical press at 45 tons of loading in the facilities of METU Material & Metallurgical Engineering Department (Figure 5.12). Target plates are produced from that alloyed material in the press. After the first trials, it is realized that the alloyed material could not fill the rectangular die region due to tight tolerances. As a result, only cylindrical samples can be produced but they could not be used in the magnetrons. When the matrix form of the alloy is examined, it is seen that hBN particles agglomerated. Therefore, this agglomeration is overcome by rolling.

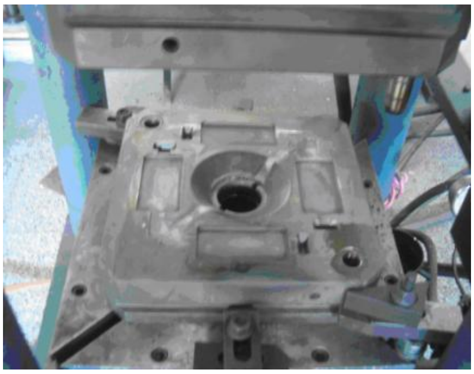


Figure 5.11 View of the bottom part of die for target plates production

Figure 5.12 Closed view of the die for target plate production

Since cylindrical target material could not be used in magnetrons, single piece die which can be seen in Appendix VI is designed and manufactured in METU-BILTIR Center. Then, Al-B-Ti-Mg-hBN alloyed material is used to obtain the target material in one piece as shown in the Figure 5.14.

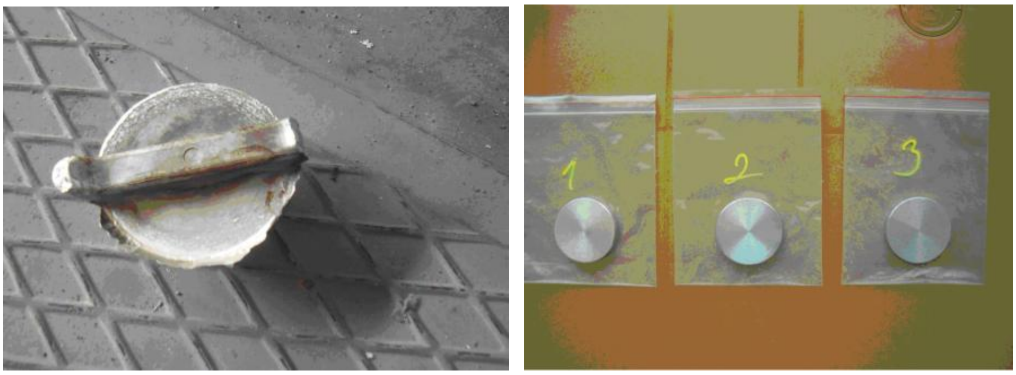


Figure 5.13 Al-B-Ti-Mg-hBN alloyed target material in cylindrical shape, a) After ejecting form the die b) After turning operation

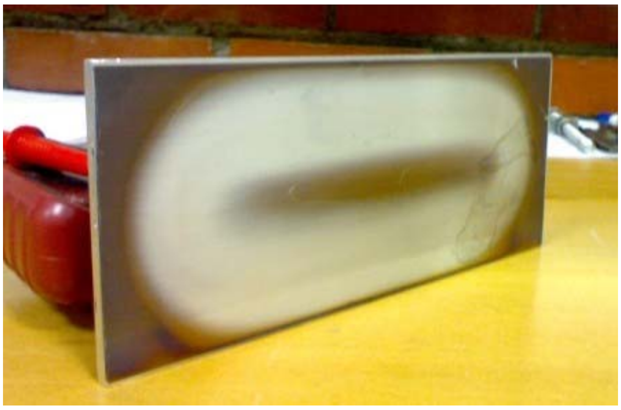


Figure 5.14 Al-B-Ti-Mg-hBN alloyed target plate

b) Production of hBN target plates in Eskişehir Anadolu University

Prof. Dr. Nuran Ay from Eskişehir Anadolu University sintered hexagonal boron nitride powder at 1800 ºC to form the target plate. The plate has the dimensions of 100x50x5 mm, so that five plates can be attached to the magnetron. The properties of the hBN powder and hBN target plate are given below:

<u>hBN Powder:</u>

- High purity hBN,
- Only hBN peaks exist in XRD and FTIR analyses
- Surface area: 20,75 m2/g
- Particle Size: d10: 2,270 µm, d50: 6,939 µm d90: 12,959 µm.

<u>hBN target plates</u>:

Sintering temperature: 1800 °C.
Density: 1,58 g/cm3
XRD analyses is in *Appendix V.*
Overall porosity in Mercury porosimeter measurements: %26,8.

Figure 5.15 hBN target plates produced in Eskişehir Anadolu University

c) Purchasing of hBN target plates from abroad

As a third case, hBN target plate is purchased from abroad and used for experiments so far. The plate is one piece and 250x100x5 mm in dimensions. HBN plate is mounted on a Molybdenum plate to prolong its use as seen the below figure.

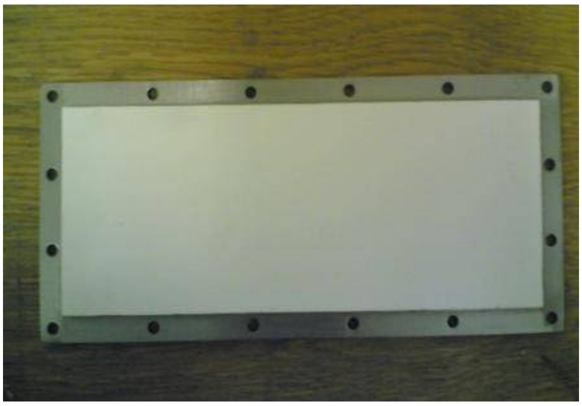


Figure 5.16 hBN target plate (mounted on Molybdenum plate)

5.3 SUBSTRATES

Boron nitride coatings are firstly deposited on steel and silicon samples, and then cutting tools are utilized as substrates. Initially, experiments are performed on steel disc samples and silicon wafers for characterization purpose. Silicon wafers are 20x20 mm in dimension and steel samples are 30 mm in diameter and 5 mm in thickness.

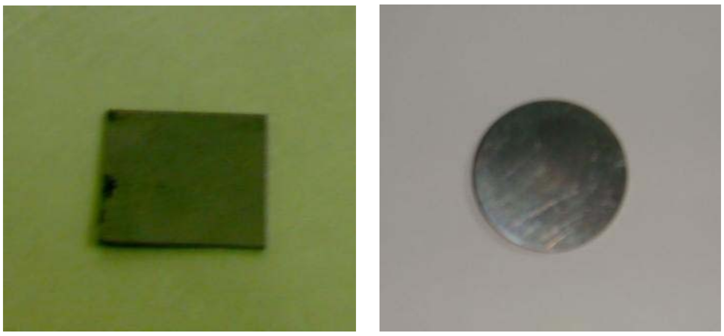


Figure 5.17 Silicon wafer & Steel disc samples

Steel disc samples are polished to improve the surface quality and cleaned ultrasonically. For silicon wafers, ultrasonic cleaning is done in the order of acetone, ethanol and distilled water. After cleaning operation, substrates are immediately located on the special holders in the vacuum chamber.

After structural and mechanical characterization of these samples, experiments continued with cutting tools. Several types of cutting tool are coated and machining tests are carried out in the facilities of universities and industries (Figure 5.18 to Figure 5.21). Moreover, die and machine elements parts are also used as substrates.

Figure 5.18 Drill & Milling cutters

Figure 5.19 Turning Insert & Screw maker cutters

Figure 5.20 Gears & Gear Teeth Making Cutters

Figure 5.21 Die parts

CHAPTER 6

EXPERIMENTS

6.1 SAMPLE PREPARATION

Initially, samples having flat surfaces are coated with boron nitride. AISI 2379 high speed steel is utilized as substrate material. Circular samples are cut from steel rods in the dimensions of 30 mm diameter and 5 mm thickness. Steps of sample preparation are given below and flow chart is provided in Appendix VII.

6.1.1 Heat Treatment

Hardness of a coating strongly depends on the hardness of its substrate. Therefore, steel samples are heated and quenched to increase their hardness which results an increase of coating hardness. Facilities of METU Material & Metallurgical Engineering Department are used for that purpose (Figure 6.1). AISI 2379 steel rod is heated up to 1040 °C in the furnaces and then quenched rapidly in oil. After tempering at 540 °C for one hour, hardness is measured as 54 HRC.

After heat treatment operation, surface of the steel rod is machined in a lathe and ground in order to remove the oxide layer and obtain a smooth surface. Next, steel rod is cut into small pieces by electrical discharge machine (EDM) in the facilities of METU-BİLTİR Center in Mechanical Engineering Department. As a result, 30 mm diameter and 5 mm thick hardened steel samples are obtained.

Figure 6.1 Heat treatment furnace

6.1.2 Polishing

Surface of the substrates have to be polished very well for better adhesion between substrate surface and coating. Polishing is a significant process and directly determines the quality of the coating. Firstly, prior to the polishing, surface of the steel samples are improved by an emery paper. After that, samples are loaded onto the polishing machine in METU Material & Metallurgical Engineering Department as shown in Figure 6.2. After adding the abrasive solution, polishing process takes place for a few minutes. Samples are pressed to the polishing disc and rotated in the reverse direction to the disc. As a last step, surface is cleaned ultrasonically.

Figure 6.2 Polishing Machine

6.1.3 Ultrasonic Cleaning

Ultrasonic cleaning is the last step of sample preparation. An ultrasonic cleaning machine is installed in the cleaning room as can be seen in Figure 6.3. After polishing, the surface of the samples has to be cleaned for the deposition process. Procedure of the cleaning is as follows; samples are put into the basket of the machine and detergent is added in the ratio of 5% of water volume. Washing temperature is set to 65 °C and duration is adjusted to 20 minutes. Cleaning process starts and continues until the adjusted time. Next, samples are taken out with the help of forceps. Samples are passed through the distilled water and rinsed in the last stage. Finally, they are mounted onto the holder inside the vacuum chamber and are ready for the deposition.

Heat treatment and polishing are not applied to cutting tools and silicon wafers. They are only cleaned in ultrasonic machine and loaded into the system.

Figure 6.3 Ultrasonic Cleaning Machine

6.2 COATING EXPERIMENTS

Experiments can be classified into four groups; TiN (Titanium nitride) experiments, TiAlN (Titanium-aluminum nitride experiments), cBN (Cubic Boron Nitride) experiments and cBN-TiN multilayer experiments. Experimental procedure and parameters are also discussed in the following parts.

6.2.1 TiN Coating Experiments

In the first experiments, only metallic sputtering is performed and titanium target plate is utilized to form titanium nitride coatings. Titanium nitride coated cutting tools are widely used in manufacturing industry and especially in chip removal processes. TiN coatings improve tool lives significantly and due to lower friction coefficient, lower cutting forces are required and chips can be removed more easily.

Titanium plate is mounted on the DC magnetron and argon gas is supplied to the chamber to generate plasma. After applying power to the DC magnetron, nitrogen gas is also supplied. As a result, sputtered titanium ions react with nitrogen gas molecules and form the compound TiN on substrate surface. The critical parameter here is the flow rate of nitrogen which determines the color and the quality of the TiN coating. When the flow rate is high, the color of coating becomes darker. Depending on the duration of the experiment, thickness of TiN coatings ranges from 0.5 to 4 μm as seen in Table 6.1.

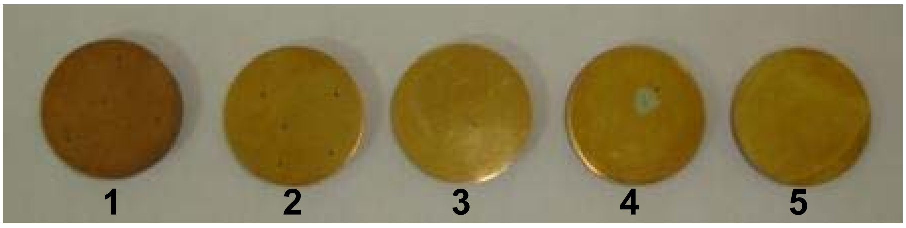


Figure 6.4 TiN coated steel samples

Table 6.1 TiN experiment parameters

Sample #	Target material	Substrate Hardness (Rockwell C)	Argon Flow (sccm)	N_2 Flow (sccm)	Duration (min)	Thickness (μm)
1	Ti	52.4	20	30	30	0.5
2	Ti	53.2	20	14.1	30	0.7
3	Ti	52.9	20	12.3	15	0.4
4	Ti	53.3	20	12.3	45	2
5	Ti	53.5	20	12.3	120	3.8

6.2.2 TiAlN Coating Experiments

After the success in TiN coatings, reactive sputtering is experimented. In reactive sputtering both DC and RF magnetrons are employed to form the desired coatings. Titanium target plate is attached to DC magnetron and aluminum target plate is attached to RF magnetron. By applying power to the magnetrons, titanium and aluminum materials are sputtered from targets and deposited onto the substrates. The resultant coating is named as titanium-aluminum nitride. TiAlN coatings are preferred in dry and high speed machining and drilling operation especially because of high oxidation resistance. Addition of aluminum increases resistance to oxidation up to temperatures of 800 ^{o}C. TiAlN coatings are observed to be purplish in color.

6.2.3 CBN Coating Experiments

Boron nitride films are deposited on Si (100) wafers and steel substrates by RF magnetron sputtering at a frequency of 13.56 MHz. Substrates are attached to a substrate holder connected to a spindle mechanism which can be biased by a second rf power supply. Substrates can make either axial or planetary motion. The spacing between samples and the target hBN plate is kept at 50 mm for all deposition processes.

The vacuum chamber is evacuated to a base pressure lower than $5x10^{-6}$ Torr using a turbo molecular pump. Afterwards, system is filled with argon gas leading a deposition pressure of $3x10^{-3}$ Torr. Prior to the deposition, plasma cleaning is performed in argon environment at a negative bias of 400 V for 10 min. Then, reactive gas nitrogen is supplied to the system so that the gas ratio of Ar to N_2 gases becomes 5 to 1. During the deposition, magnetron power applied to the target plate is increased up to 900 W gradually and maintained at that value till the end of deposition. Heater temperature is varied between 100-250 °C for different bias voltage values ranging from 50 to 200 V. The growth of the films continued from 1 to 4 hours for every experiment. The deposition parameters are summarized in Appendix II.

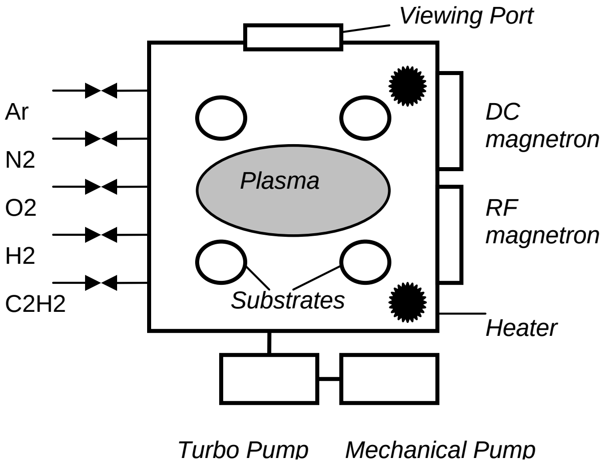


Figure 6.4 Schematic view of vacuum chamber

6.2.4 cBN-TiN Multilayered Coating Experiments

Previously reported challenges in physical vapor deposition such as compressive residual stress, poor adhesion and limiting coating thickness during cBN formation can be eliminated by carefully controlling deposition parameters and by applying multi-step deposition processes [35,36,45,46,47].

Besides cBN coatings, composite or multi-layer coatings are also deposited in this research. cBN-TiN multi-layer coatings are formed by a two step process.

Firstly, DC magnetron is employed with a titanium target plate and TiN layer is initially coated. Later, without interrupting the experiment, RF magnetron is employed with a hBN target plate. The transition from DC to RF magnetron should be slowly so that between TiN and cBN layer, a TiN-cBN matrix should be formed. This is necessary to gain sufficient adhesion between layers. As a result, TiN becomes the bottom layer and provides adhesion with the substrate. On the other hand, top layer is formed from cBN so that desirable hardness on the top surface is achieved. After the experiments, coatings are characterized by several methods which will be discussed in the next chapter.

CHAPTER 7

DISCUSSION OF RESULTS

Deposited boron nitride coatings are investigated by several characterization techniques. These techniques are divided into three main groups such as structural and compositional characterization, mechanical characterization and machining tests.

1. Structural and Compositional Characterization
    - Fourier Transform Infrared Spectroscopy (FTIR)
    - X-Ray Photoelectron Spectroscopy (XPS)
    - X-Ray Diffraction (XRD)
    - Scanning Electron Microscopy (SEM)

2. Mechanical Characterization
    - Thickness Measurement
    - Hardness Measurement
    - Friction Coefficient Measurement
    - Adhesion Measurements

3. Machining Tests
    - Cutting Force Measurements
    - Wear Measurements
    - Tool Life Determination

7.1 STRUCTURAL AND COMPOSITIONAL CHARACTERIZATION

7.1.1 Fourier Transform Infrared Spectroscopy (FTIR)

The first tool for the structural and compositional characterization is Fourier Transform Infrared Spectroscopy (FTIR). In this analysis type, peaks are seen at certain wave numbers depending on the phase of the film. Considering the phases of boron nitride, these peak values are seen at 780 and 1380 cm^{-1} for hexagonal phase and 1065 cm^{-1} for cubic phase. Therefore, by examining the FTIR results of deposited boron nitride coatings, one can determine the ratio of the phases easily. In other words, volume fraction of the boron nitride coating is calculated from FTIR plots by evaluating the area under the peaks. Determination of the ratio of the phase of the coating is calculated by a simple formulation below and *I* symbol in the formulation represents the area under the peaks of boron nitride phases;

$$cBN\% = \frac{I_{cBN}}{I_{cBN} + I_{hBN}} \times 100\% \tag{1}$$

FTIR results of boron coatings are plotted in Figures 7.1 and 7.2 respectively. As can be understood from the graphs, phase of the coating differs with respect to deposition temperature and bias voltage. According to the graphs, highest volume fraction of cubic phase is calculated as 75 % at 200 °C deposition temperature and 200 V bias voltage. This ratio may be increased by modifying the experiment parameters. Other peaks observed at different wave numbers may be belong substrate material. FTIR results indicate that boron nitride films are successfully deposited on steel substrates.

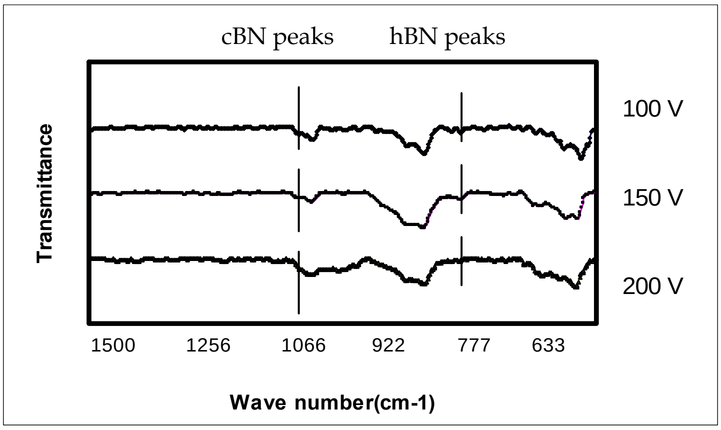


Figure 7.1 FTIR spectrum of BN films deposited at 200 ºC

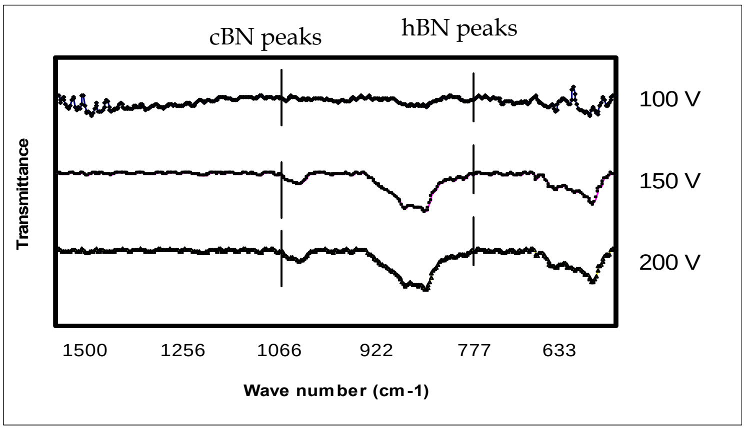


Figure 7.2 FTIR spectrum of BN films deposited at 250 ºC

7.1.2 X-Ray Photoelectron Spectroscopy (XPS)

In order to examine the composition of the coating X-Ray photoelectron spectroscopy (XPS) method is utilized. Elemental composition of the BN coating deposited at 200 ºC and under 100V bias voltage is determined by XPS device which is located in METU Central Laboratory.

Figure 7.3 and Figure 7.4 are showing that the binding energies of single chemical states of boron and nitrogen (B_{1s} and N_{1s}) are revealed at 190.3 and 397.5 eV, respectively. Moreover, B/N ratio is found to be 1.1 which means that the films are deposited stoichiometrically.

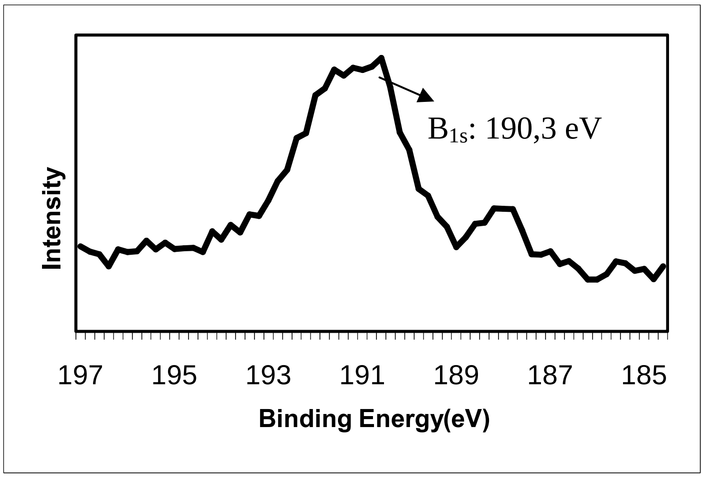


Figure 7.3 XPS results of BN film (expressing boron binding energy)

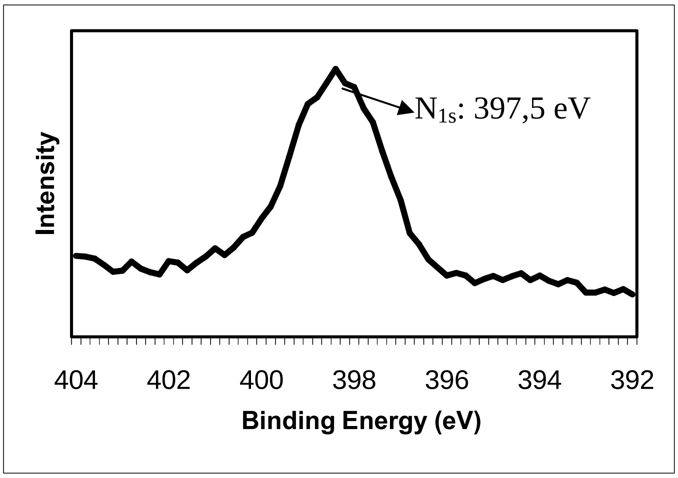


Figure 7.4 XPS results of BN film (expressing nitrogen binding energy)

7.1.3 X-Ray Diffraction (XRD)

X-ray diffraction analyses of the boron nitride films which deposited on silicon wafer substrates are performed in the facilities of METU Metallurgical and Material Department. 2° theta angle is used during the analysis. The plot in Figure 7.5 demonstrates that silicon single crystal peak is seen at 69.3° intensively. Peaks belong to cubic phase of boron nitride are observed at 45.5°, 49.4° and 73.2° respectively. These values are reported previously in the literature so that they are reliable.

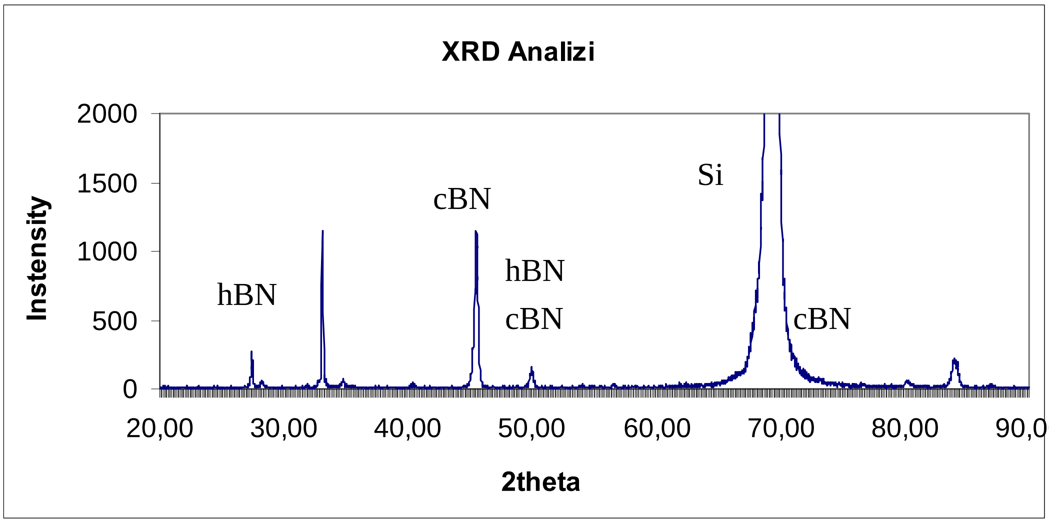


Figure 7.5 XRD results of BN film (deposited at 200°C and 150 V bias voltage)

7.1.4 Scanning Electron Microscopy (SEM)

In order to investigate the surface morphology of the films, scanning electron microscopy (SEM) is utilized. Many photographs are taken to observe the surface of the coatings and defects, cracks and impurities are carefully investigated. Furthermore, it is possible to perform EDX (Energy Dispersive X-ray) analysis by SEM which gives the elemental proportion of the components of the coating and it is employed to verify the composition of the coating. Results of EDX analysis are tabulated in Table 7.1.

Initially, TiAlN coated samples are put into the scanning electron microscope and surface images are taken which is seen in Figure 7.6. Cracks, scratches, delamination and other defects on the surfaces can be seen in the below figures. These defects may occur due to high residual stresses during the deposition process at high power values.

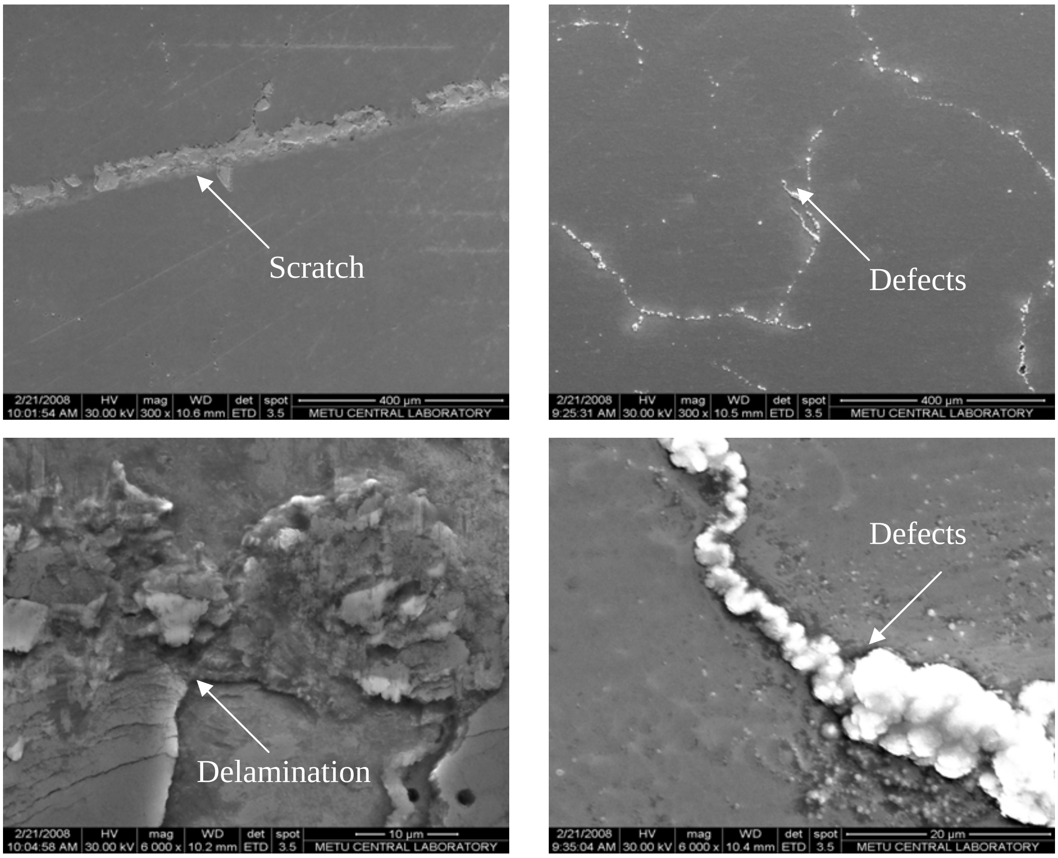


Figure 7.6 SEM images of TiAlN films deposited on steel samples

In case of boron nitride coatings, micro cracks are seen in Figure 7.7 due to high residual stress which was reported many times in the literature [25,31,34,37,39]. High ion bombardment rate during the sputtering leads to high stresses in the films. That residual stress also limits the thickness of the boron nitride films. However, many researchers reported synthesis of thick cBN films by multi-step processes in recent years [26,27,29,31,39]. Moreover, internal stresses of the films may be reduced by modifying the experiment parameters and multi-step deposition. Boron nitride films deposited on both steel samples and silicon wafers are investigated by SEM to observe the affect of the substrate. It seems that films deposited on silicon wafers have fewer defects because of very good surface quality. Besides micro cracks, some defects, voids are also observed in Figure 7.7 and 7.8. Possible reasons for these defects may be impurities on the substrate surface, defects from substrates, insufficient cleaning or problems in the deposition process. Optimization of deposition parameters and care on sample preparation may reduce these defects.

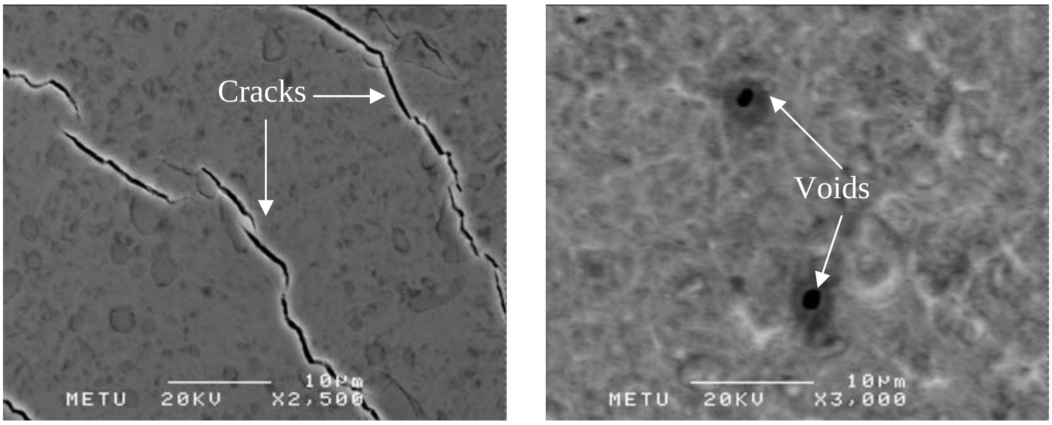


Figure 7.7 SEM images of cBN films deposited on steel samples

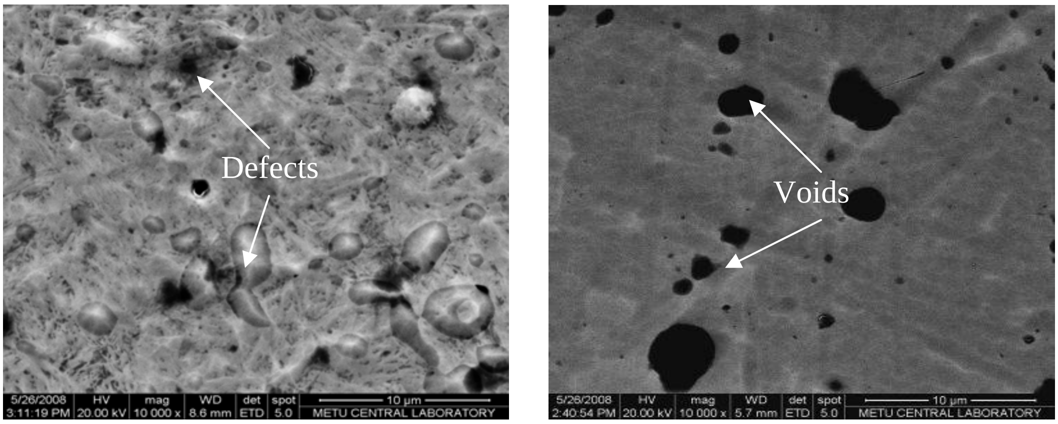


Figure 7.7 SEM images of cBN films deposited on steel samples

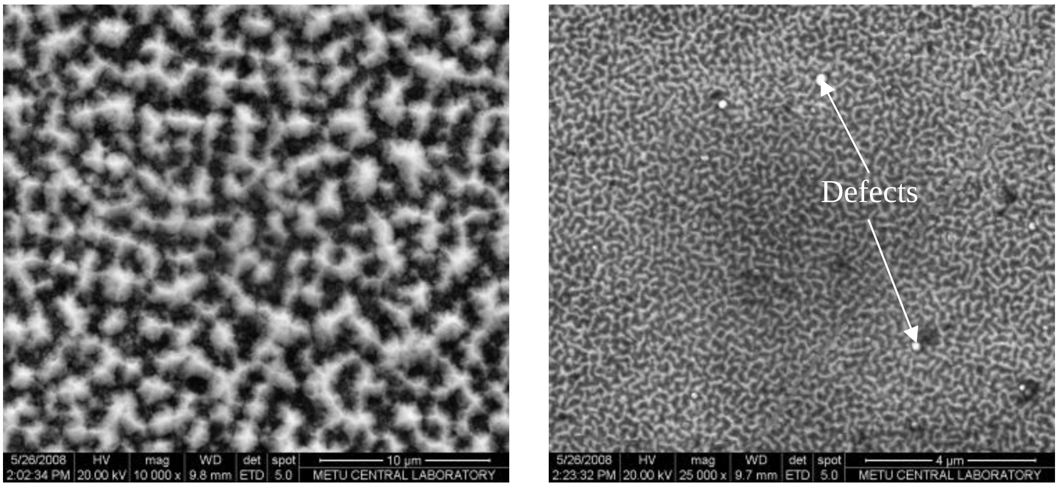


Figure 7.8 SEM images of cBN films deposited on silicon wafers

Table 7.1 EDX analysis results showing elemental proportion of BN coating

Element	Weight Ratio (Wt %)	Atomic Ratio (At%)
Boron (B)	14.05	33.52
Nitrogen (N)	19.00	34.99
Argon (Ar)	1.01	0.65
Chrome (Cr)	11.92	5.91
Iron (Fe)	51.78	23.92
Nickel (Ni)	1.99	0.87

7.2 MECHANICAL CHARACTERIZATION

7.2.1 Thickness Measurement

Thickness of the boron nitride films are measured by a calotest device in a destructive manner. Steel samples are clamped by vise and are in touch with the spherical ball. After the addition of abrasive solution, the ball is rotated until a sufficient crater is observed at the sample surface. Diameters of the crater on coating surface and substrate surface are measured by optical microscope and thickness of the coating is calculated. Film thickness of 0.5 µm is achieved at deposition conditions of 900 W RF power, $3x10^{-3}$ Torr deposition pressure, Ar/N2=5/1 ratio for 2 hours. Thickness measurements for different deposition durations at constant parameters are listed in Table 7.2.

Figure 7.9 Calotest Device & Optical Microscope

Table 7.2 Thickness variation of BN coatings depending on deposition time

Duration	1 hr	2 hr	3 hr
Thickness	0.2 µm	0.5 µm	0.8 µm

7.2.2 Hardness Measurement

7.2.2.1 Substrate Hardness Measurement

Hardness of the substrate directly affects the hardness of the coating. In other words, harder the substrate, harder the coating is. For that reason, substrate hardness should be determined before coating the substrate. Rockwell C hardness measurement device is utilized to determine bulk hardness of the substrates. After heat treatment process, steel rod is cut by EDM to many pieces and hardness values of surfaces of the steel samples are measured on three different points and average value is calculated. According to the measurements of 14 steel samples, hardness values of steel samples are found around 55 HRc and shown in the *Appendix III*.

Figure 7.10 Rockwell C hardness measurement device

7.2.2.2 Coating Hardness Measurement

One of the significant mechanical properties of a coating is its hardness. In hardness measurement of thin coatings, to avoid the substrate effect on coating hardness, indenter tip should stay inside of the coating and should not pass to the substrate. Since thickness of the coating is very low, effect of substrate is more dominant. In other words, very small penetration depth and loads should be accurately controlled. For example, if a micro hardness device is used to determine the coating hardness, the obtained data would not be correct because the indenter penetrates not only into the coating but also into the substrate under loading. That's why a nano scale hardness device is required to determine hardness of cBN coating. However, nanoindentation devices in Turkey could not be used. Thus, it is requested to measure coating hardness by a private company in Switzerland. CSM Instruments open platform with a nano indenter head is utilized to evaluate the hardness of the coatings (Figure 7.11). Basically, a nanoindentation device can measure hardness of thin coatings very accurately. Penetration depth and loading can be controlled by very sensitive sensors so that the indenter does not pass through the substrate. Very small loads can be applied in the range of mN's. Penetration depth is in nano meter scale.

Figure 7.11 CSM Instruments Nanoindentation (Nano hardness) device

Unfortunately, company accepted to test only two cBN coated and one cBN-TiN coated steel sample. Tests are performed at room temperature (23 ⁰C) and in humid air of %40. Other test parameters are listed below:

- Indenter Type: Berkovich
- Approach Speed: 2000 nm/min
- Loading Type: Linear
- Loading Speed: 60 mN/min
- Maximum Load: 30 mN
- Pause at Maximum Load: 0 s
- Unloading Speed: 60 mN/min

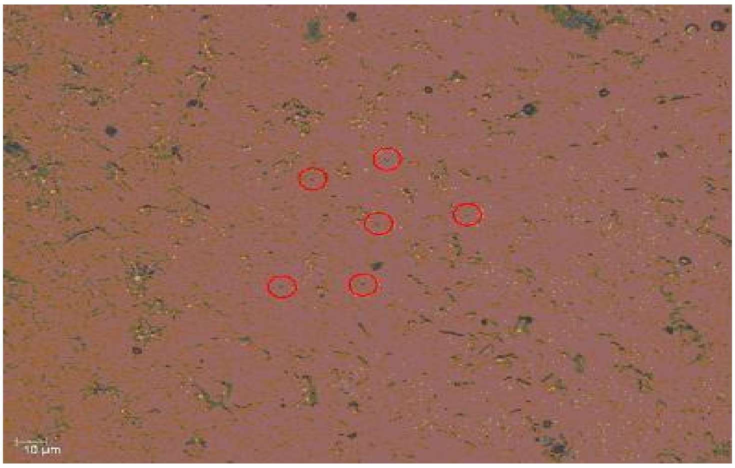


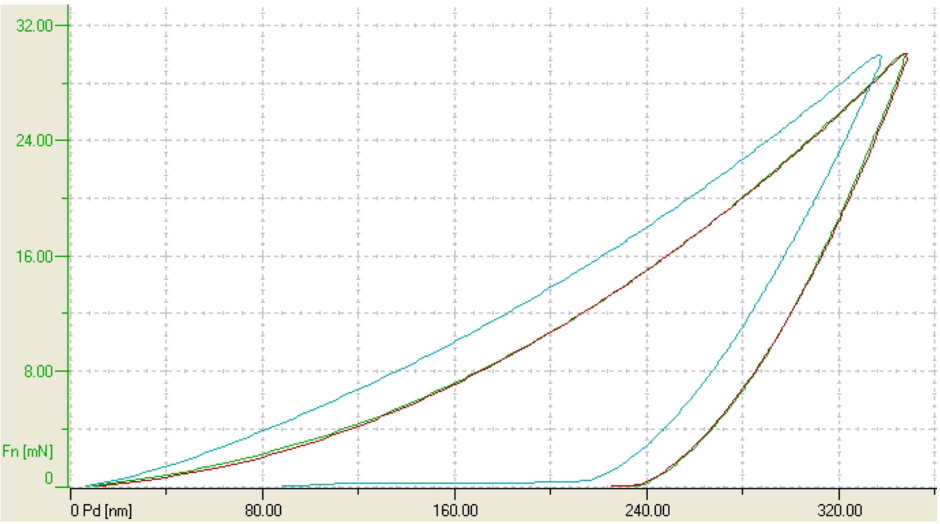
Figure 7.12 Indentation marks

The above photo shows the marks of indenter on sample surface in red circles. Surface roughness and impurities on the surface may influence the real coating hardness values. For that reason, six measurements are carried out and an average hardness value is calculated for each sample (Figure

7.12). Besides hardness values, Young's Modulus of the coatings are evaluated with a Poisson's ratio assumption of 0.3 using the load versus penetration depth curve in Figure 7.13. Results are shown in Figure 7.14.

In order to minimize substrate effect, penetration depth is limited to 340 nm. If the values are examined, it is seen that cBN films exhibit about 12.5 GPa hardness. On the other hand, hardness of cBN-TiN multi-layer coating is observed as 14 GPa which is higher than monolayer cBN coatings. However, hardness values are low in comparison with the literature [29,31,58]. The reason may be because of surface quality, surface roughness and surface porosity which are seen in SEM photographs. Similarly, elastic modulus values are consistent in CBN coatings and evaluated as 276 and 271 GPa. Hardness and elastic modulus values are given in Appendix IV. Sample 1 is cBN coated steel (at 200 °C), sample 2 is cBN coated steel (at 250 °C) and sample 12 is cBN-TiN coated steel sample.

Improvement of hardness values can be possible if cubic phase of the film will be increased. In the first experiments, the most used deposition parameters are selected among the several studies in the literature. For that reason, optimization of the deposition parameters and improvement of the surface quality will be done as a future work.



🟩	🟥	🟦
Sample #1: cBN coated hardened steel (deposited at 200 °C)	Sample #2: cBN coated hardened steel (deposited at 250 °C)	Sample #12: cBN–TiN coated hardened steel

Figure 7.13 Load vs Penetration Depth curve of samples

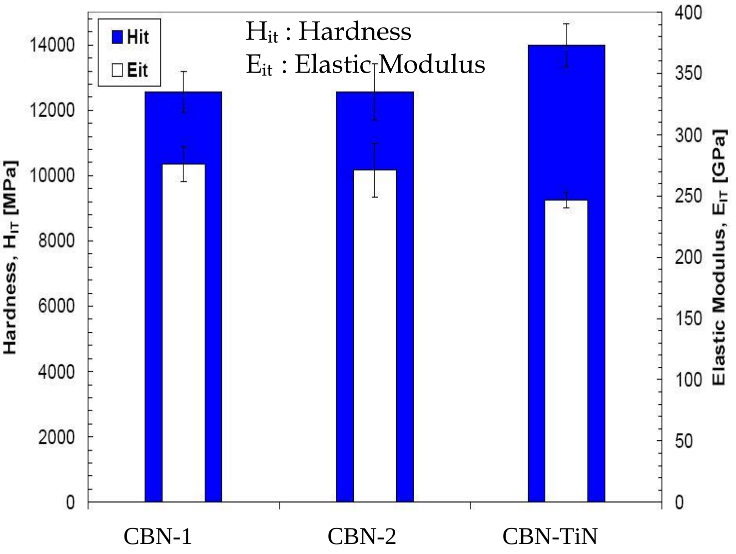


Figure 7.14 Hardness and Elastic Modulus of BN coatings

7.2.3 Friction Coefficient Measurement

Data on friction coefficient of steel samples is obtained using a tribometer device in the facilities of Material & Metallurgical Engineering Department of Istanbul Technical University (Figure 7.15). Analyses are implemented without lubrication in air environment at room temperature. Alumina ball having diameter of 6 mm is utilized as a pin material. Tests are performed under conditions of normal load of 1 N, linear speed of 5 cm/s and radius of 5 mm for 50 m distance. As shown in Figure 7.16, mean coefficient of friction against alumina is found to be around 0.5 at that test conditions. It was reported that friction coefficient of cubic boron nitride against steel was found to be 0.4 which makes our result meaningful [48]. Wear of the film on the steel samples are tried to be observed by a profilemeter; however, it is really difficult to distinguish wear marks and surface roughness which indicates that boron nitride coating deposited on steel has quite high resistance to wear.

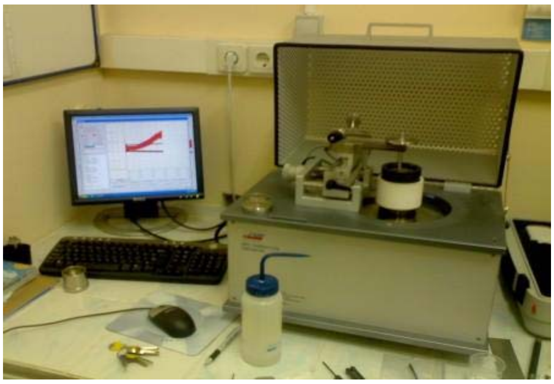


Figure 7.15 Tribometer device in Material & Metallurgical Engineering Department of Istanbul Technical University

Same tests are also repeated in the facilities of Mechanical Engineering Department of Erzurum Ataturk University. Same parameters are used to verify previous test results and mean friction coefficient is measured as 0.5 against alumina material (Figure 7.17)

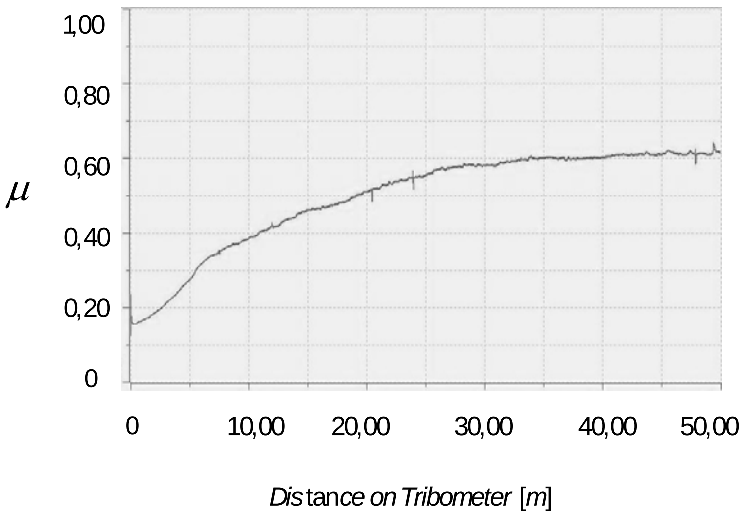


Figure 7.16 Tribometer Test Results performed in Istanbul Technical University

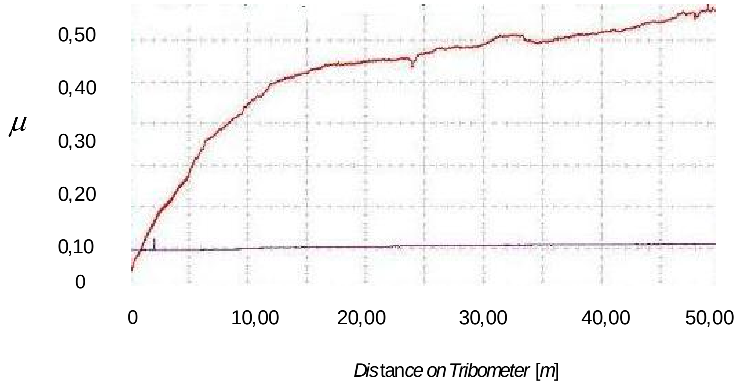


Figure 7.17 Tribometer Test Results performed in Istanbul Technical University

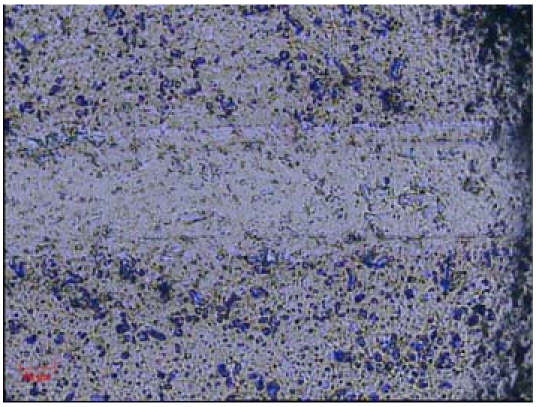
7.2.4 Adhesion Measurements

7.2.4.1 Micro Scratch Tests

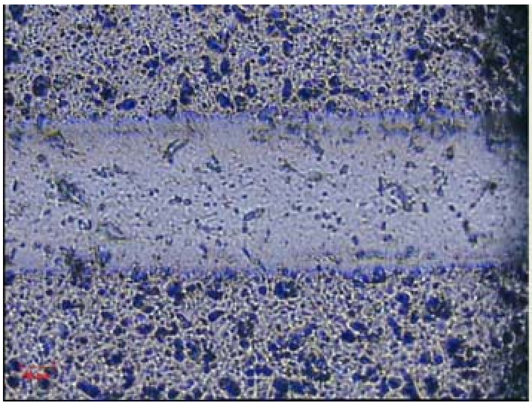
Adhesion of the cBN coatings is determined firstly by a micro-scratch tester device in the facilities of Mechanical Engineering Department of Erzurum Ataturk University (Figure 7.18). CBN coated steel samples are tested under progressive loading at a rate of 100N/min. Scratches are formed on cBN coated surfaces by a diamond indenter which has a nose radius of 200 µm and moves at a speed of 10 mm/min. Starting load is adjusted to 0.2 N and final load at the end of the scratch is 80 N. Length of the scratch is 8 mm. At the end of test, scratches are examined under microscope and failure or fracture of the cBN coatings are tried to be observed.

Figure 7.18 Micro Scratch Test Device

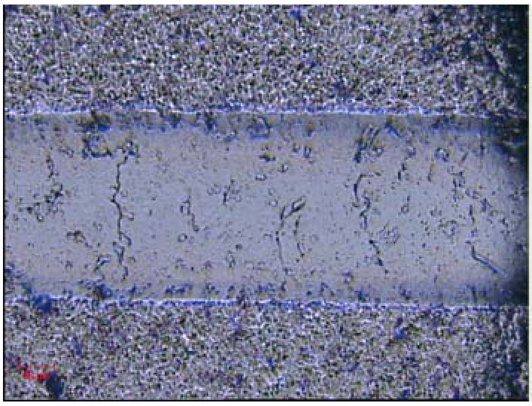
As the load increases, penetration depth also increases and at the end of 8 mm scratch load becomes 80 N whereas penetration depth is 50 µm. This means that diamond indenter passes the coating and penetrates into the substrate material. Therefore, a nano scratch will give better results about the coating adhesion. In micro scratch tests, loads are quite high and it is seen that coatings exhibit quite good adhesion such that no cracks, fractures or failures on the coatings are observed (Figure 7.19).



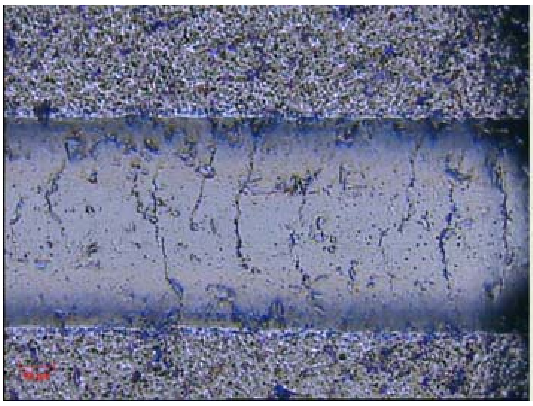
Under 20 N loading



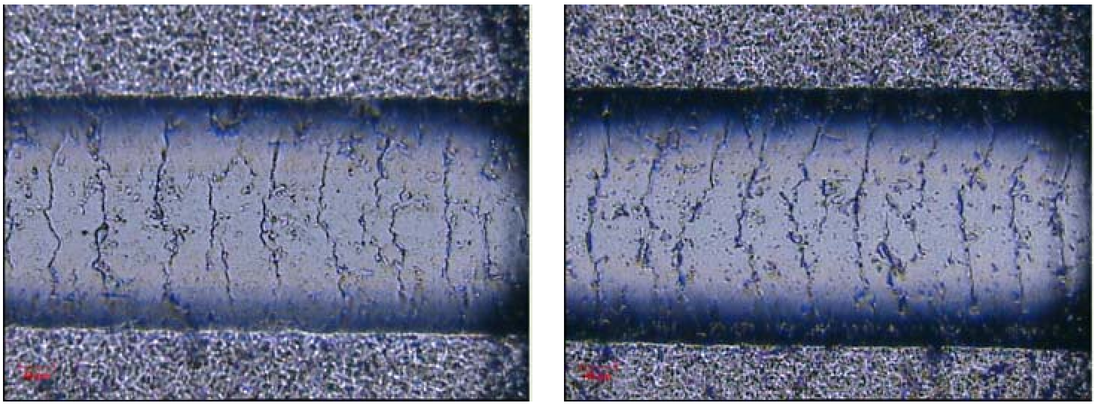
Under 30 N loading



Under 40 N loading



Under 50 N loading



Under 60 N loading Under 70 N loading

Figure 7.19 Micro Scratch Test results of boron nitride coatings

7.2.4.2 Nano Scratch Tests

Only cBN-TiN coated steel sample can be tested by CSM nano scratch device (Figure 7.20). Spherical-conical 90° indenter type with a radius of 5 µm is used. Loading is started from 3 mN and progressively increased to 200 mN. 2 mm scratch is formed while indenter is moving at a speed of 4 mm/min.

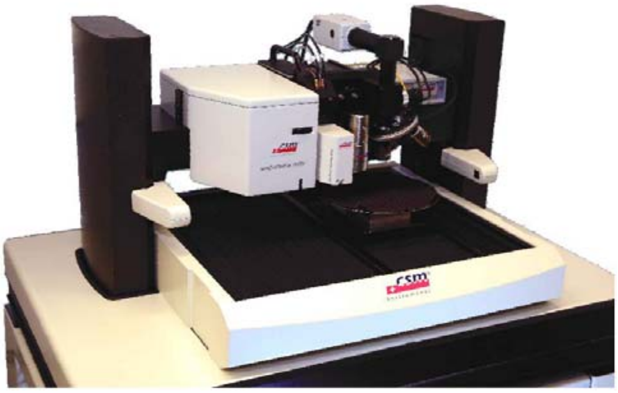


Figure 7.20 CSM Instruments Nano Scratch device

Tests are performed at room temperature and humid air of % 40. Penetration depth is increasing due to progressive loading. Besides, load and penetration depth, friction coefficient and acoustic emission can be measured by accurate sensors. In a scratch test, two critical loads should be observed which is indicated by vertical lines in the graph. First critical load is the normal load at which the first failure appears on the film. In Figure 7.21, a fracture of the coating is observed at the side of the scratch track and simultaneously the first delamination of the coating appears. An increase of the friction coefficient is also observed after the first critical load. Second critical load is the normal load at which the full delamination of the film occurs which is seen in Figure 7.22. Eventually, critical loads are seen at 108 mN and 173 mN, respectively.

At the end of 2 mm scratch, the friction coefficient is found to be about 0.5 which is matching with the tribometer result reported before. The nano scratch system has a force feedback loop control, so that the force applied on the sample is not affected by the surface topography.

Moreover, graph in Figure 7.21 also gives us thickness of the coating. After second critical load, substrate surface comes on the scene and if we look at the penetration depth at this value, it is seen that the thickness of the coating system is between 700 and 900 nm in this case.

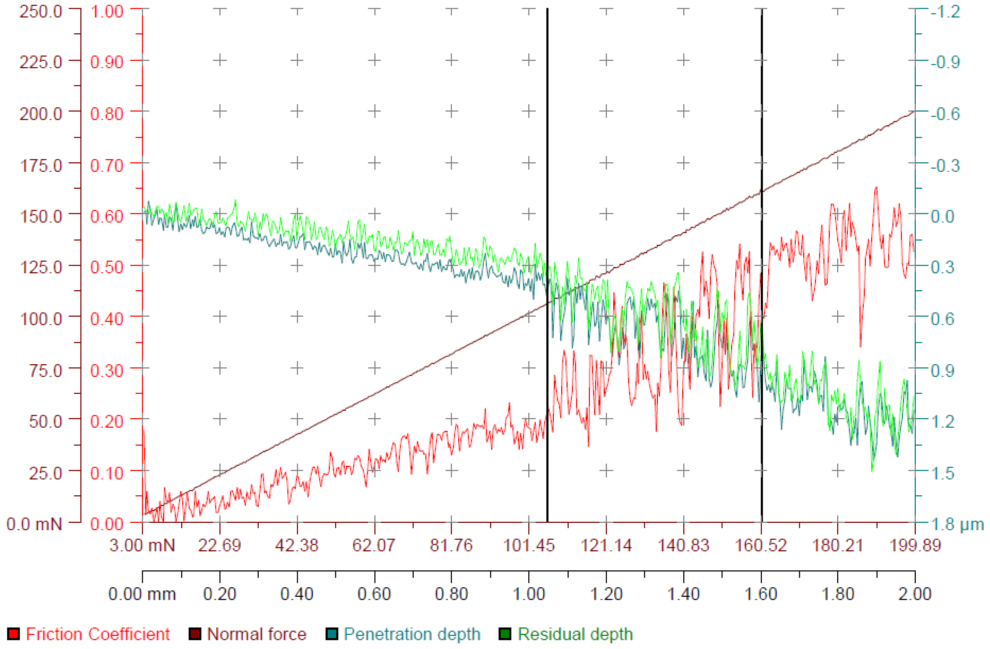


Figure 7.21 Nano-scratch test results for CBN-TiN multilayer coating

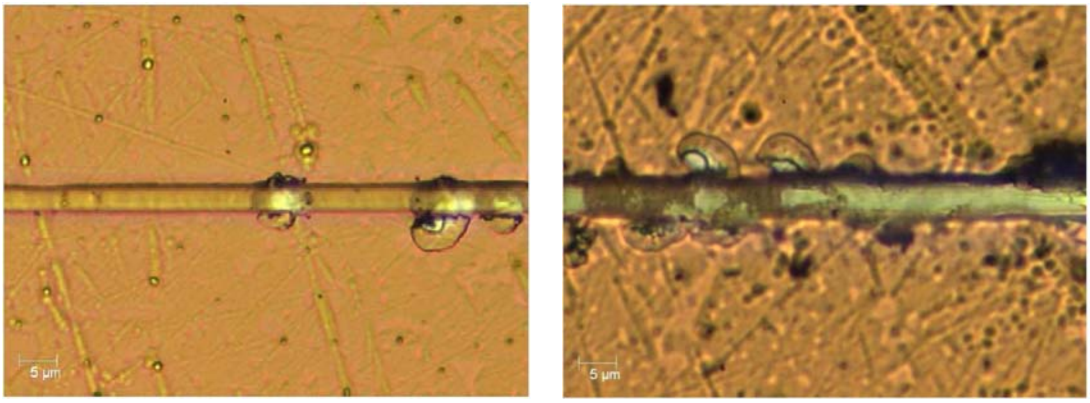


Figure 7.22 Failure at the first and second critical loads. (108 mN and 183 mN, respectively)

7.3 MACHINING TESTS

After obtaining satisfactory results on samples, cutting tools are utilized as substrate material and coated with cubic boron nitride. For that purpose, collaborations from universities and industry have been attempted and cutting tools are provided for experiments. After coating them with cBN, analysis and measurements are carried out to investigate the machining performance. Analysis of cBN coatings on cutting tools are divided into three topics.

7.3.1 Cutting Force Measurements

Tools are exposed to high cutting forces and temperatures during machining. Determination of cutting forces in a machining operation is essential and gives significant information about the behavior of cutting tools during machining. Many studies have been performed to determine cutting forces and torques before the machining operation [12,13,14].

Initially, turning inserts are coated by cubic boron nitride using a physical vapor deposition system. Assoc. Prof. Dr. Tuğrul Özel from Industrial & System Engineering Department of Rutgers University in USA sent four different turning inserts (Figure 7.23). Surface of the two of these inserts are made of PcBN (Pollycrystalline Cubic Boron Nitride). One of the inserts is coated with TiAlN and other one is uncoated tungsten carbide. Properties and photographs of the inserts are given in *Appendix VIII* in detail.

One uncoated and one TiAlN coated tungsten carbide (WC) inserts are cleaned ultrasonically and located inside the system carefully. Inserts are mounted on tool holder from the middle of cutting side so that insert corners can be deposited effectively. Then, inserts are coated by cubic boron nitride for three hours. Experiments are performed under deposition pressure of 3×10^{-3} Torr and heater temperature of 100 °C. Applied magnetron power is fixed at 900 W and argon to nitrogen gas ratio is adjusted to 5/1. After the first set of experiments, it is realized that effect of bias voltage or substrate voltage is significant. In other words, cutting edges could not be deposited well when bias voltage is high (Figure 7.24). Therefore, bias voltage is kept as minimum value as possible in the following experiments.

Figure 7.23 Turning Inserts

Figure 7.24 CBN coated WC turning insert

After coating experiments, machining tests are performed using the facilities of Rutgers University. Inserts are tested in cylindrical turning of titanium alloyed Ti-6Al-4V material which is known as a difficult-to-cut material. Four inserts; uncoated WC, CBN coated WC, TiAlN coated WC and CBN-TiAlN coated WC are used for comparison. Edge radius of the inserts is 2 μm. Machining tests are done without using a coolant. In other words, dry machining is applied. Depth of cut is adjusted to 2 mm and feed is set to 0.1 mm/rev. In order to observe the performance of coatings at different cutting speeds, two tests are done at cutting speeds of 50 and 100 m/min, respectively.

Cutting tools

- Uncoated Tungsten Carbide (WC) insert
- TiAlN coated WC insert
- CBN coated WC insert
- CBN-TiAlN coated WC insert
    - Insert nose radius r_ε= 0.8 mm
    - Insert edge radius $r_\beta \approx$ 2 μm

Cutting conditions

- Dry machining
- Depth of cut: a_p=2mm
- Cutting speed: V=50 m/min and 100 m/min
- Feed: f=0.1 mm/rev
- Tool holder: 0° lead,
  -5° side rake,
  -5° back rake angles
- Cutting time: 7 minutes at 50 m/min
  3 minutes at 100 m/min

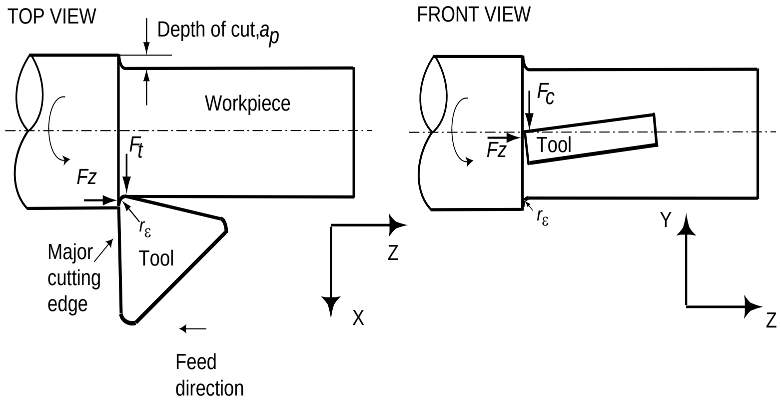


Figure 7.25 Cutting Forces in cylindrical turning

Forces in cylindrical turning operation are shown in Figure 7.25. Mainly, there are three different forces acting on the tool. Tangential or cutting force (F_c) acts in a direction tangential to the revolving work piece and represents the resistance to the rotation of the work piece. Longitudinal or axial force (F_z) acts in the direction parallel to the axis of the work and represents the resistance to the longitudinal feed of the tool. Radial or thrust force (F_t) acts in a radial direction from the center line of the work piece.

These forces are measured during turning process at two different speeds for each cutting tool and results are plotted in Figure 7.26.

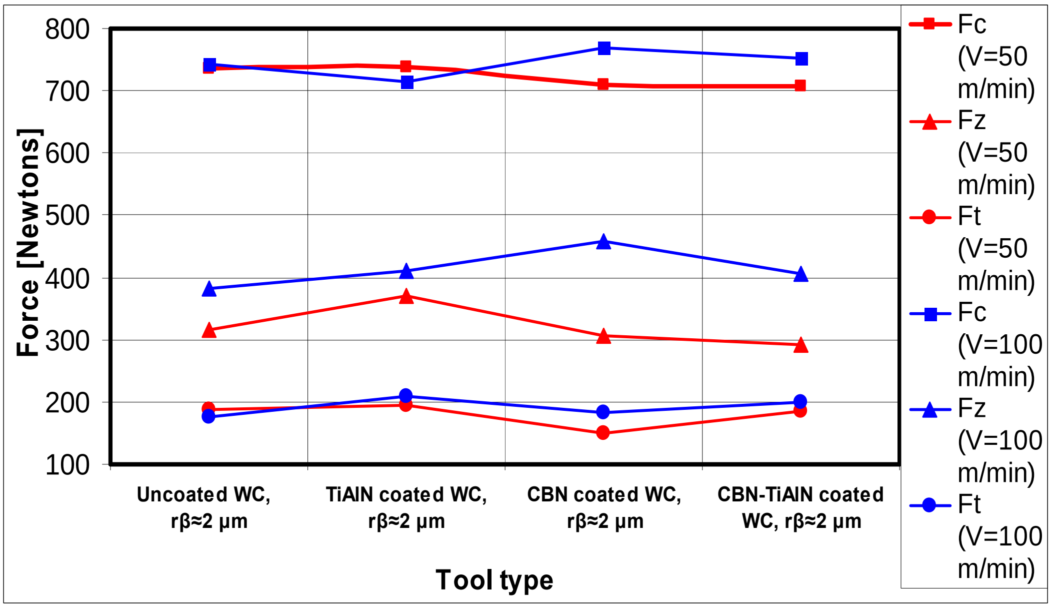


Figure 7.26 Comparison of cutting forces at different cutting speeds

According to the above graph, CBN and CBN-TiAlN coated inserts exhibit lowest cutting forces at low cutting speeds. On the other hand, TiAlN coated WC inserts has the lowest cutting force when cutting speed is high. Furthermore, if thrust forces are compared, highest thrust force is measured on CBN coated WC insert at high cutting speed. Thrust forces of TiAlN and CBN-TiAlN coated inserts are almost same. At low cutting speed, TiAlN coating has the highest thrust force. Additionally, CBN and CBN-TiAlN coatings have similar thrust forces at low cutting speeds. Radial forces do not show much difference at low and high speeds. Finally, it can be claimed that CBN coated WC inserts has better performance at low cutting speeds.

7.3.2 Wear Measurements

Tool life is generally determined with respect to wear on the tool surface. Among the wear types, crater and flank wear are types of gradual wear which is actually a slow degrading mechanism of a tool surface [1,2]. Flank wear is the wear on the flank of the cutting tool which is caused by friction between the newly machined work piece surface and the contact of the tool flank. However, crater wear is often the factor which determines the life of cutting tool. If the crater becomes so severe, then the tool edge is weakened and eventually fractures (Figure 7.27).

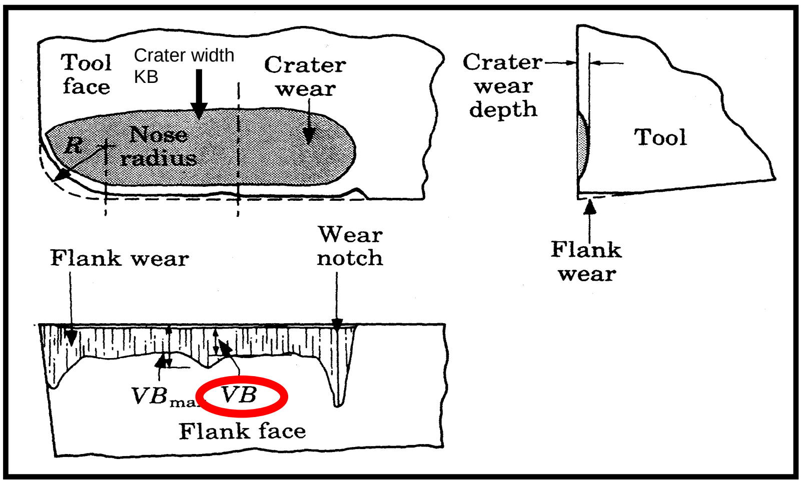


Figure 7.27 Locations of flank and crater wear on an insert

Flank and crater wears are observed for uncoated, TiAlN, CBN and CBN-TiAlN coated inserts. Results of the wear measurements are given in Figure 7.28. It should be noted that machining tests are done against titanium alloyed Ti6Al4V work piece material which is considered difficult-to-cut material with most cutting tools. It can be deduced from the graph that TiAlN coated WC insert exhibits the worst wear performance both in low and high speed machining. Interestingly, TiAlN coated insert exhibits worse performance than uncoated one. This is not a meaningful result but there may be some measurement errors. CBN-TiAlN coated WC insert has lowest crater wear in low cutting speeds. On the other hand, CBN coating results in lowest flank wear both in low and high cutting speeds. As a result, it is claimed that CBN coatings reduce flank and crater wear by a factor of two in dry machining of titanium alloyed Ti6Al4V material. From the results of wear measurements, tool life of CBN coated WC insert is expected to be at least twice longer in dry machining of titanium alloyed Ti6Al4V material. Wear on tool edges of the worn inserts after machining tests can be seen in Figure 7.29.

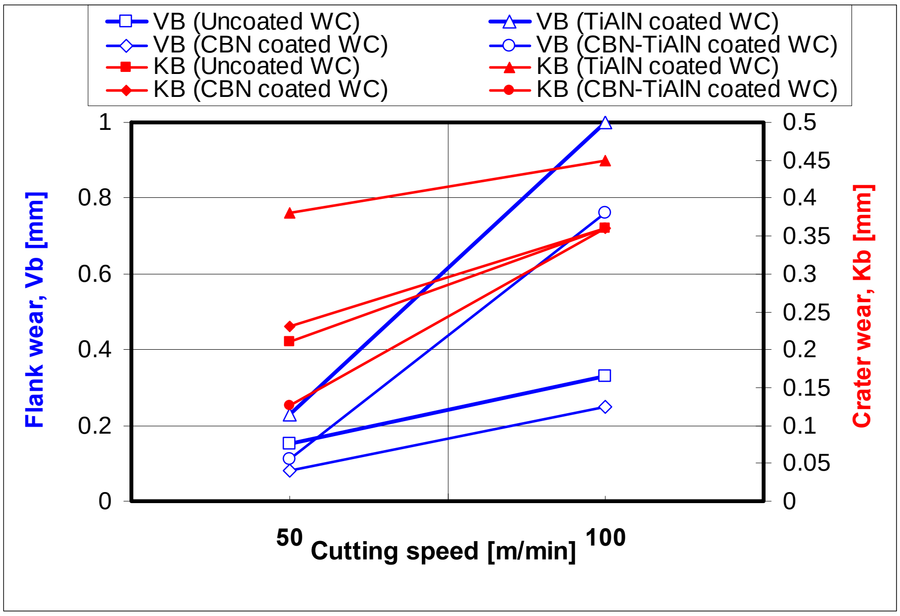
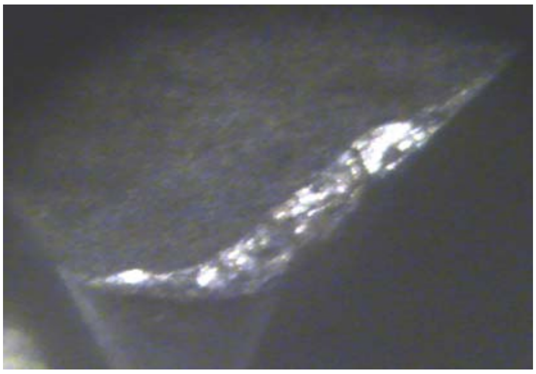
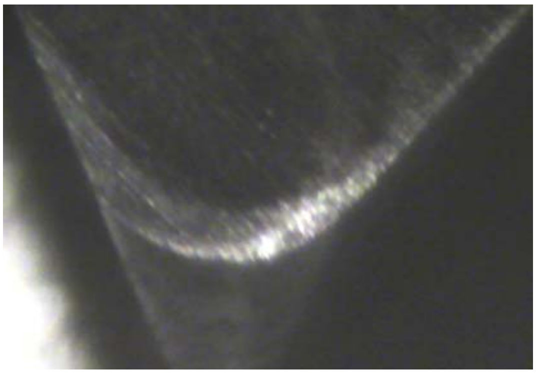


Figure 7.29 Flank and crater wear of turning inserts after machining 7 mins at 50 m/min and 3 mins at 100 m/min

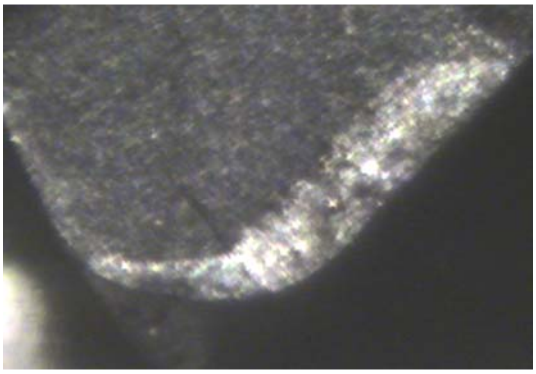
Uncoated WC
V=50 m/min, a_p= 2 mm, f=0.1 mm



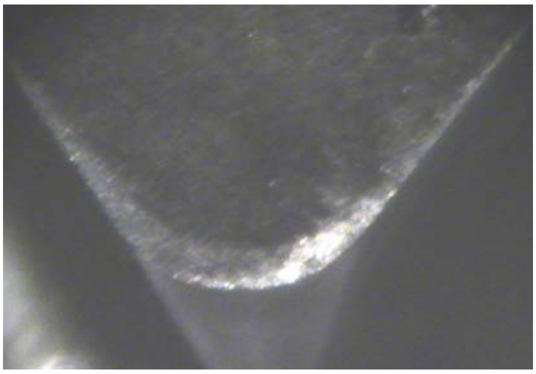
Uncoated WC
V=100 m/min, a_p= 2 mm, f=0.1 mm



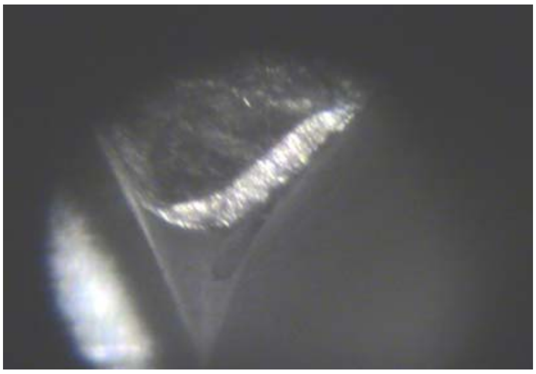
TiAlN coated WC
V=50 m/min, a_p= 2 mm, f=0.1 mm



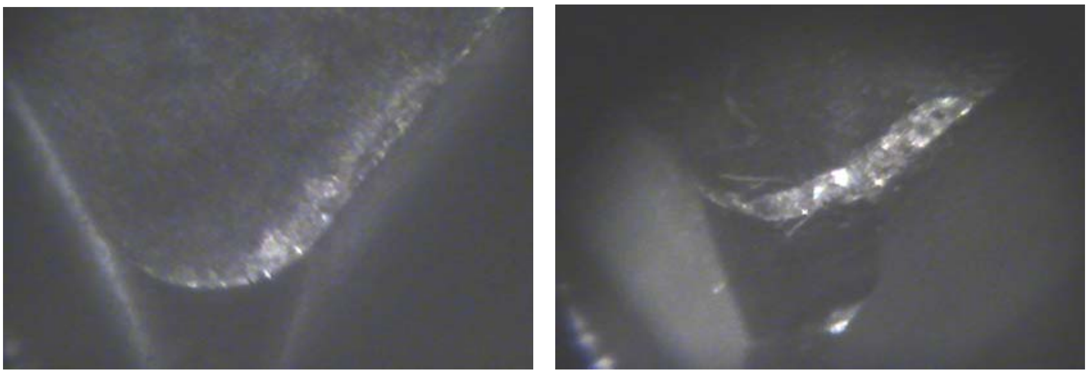
TiAlN coated WC
V=100 m/min, a_p= 2 mm, f=0.1 mm



CBN coated WC
V=50 m/min, ap= 2 mm, f=0.1 mm



CBN coated WC
V=100 m/min, ap= 2 mm, f=0.1 mm



CBN-TiAlN coated WC
V=50 m/min, a_p= 2 mm, f=0.1 mm

CBN-TiAlN coated WC
V=100 m/min, a_p= 2 mm, f=0.1 mm

Figure 7.28 Images of worn inserts

7.3.3 Tool Life Determination

In order to determine tool life of the cutting tools, a study together with industry is carried out. Collaboration with a local company called Turk Traktor is attempted. In the production plant of the company, chip removal processes are widely used and different types of cutting tools are utilized in machining operations. Tool engineers of the company suggested applying cBN coatings on carbide drills which have been used extensively in the machining of the engine block. Two carbide drills having diameters of 6.8 mm and 8.8 mm which are having grooves inside for coolants are used as substrates (Figure 7.30). Ø6.8 mm drill is used in machining of cylinder cover. On the other hand, Ø8.8 mm drill is used in machining of engine block which is made of cast iron.

Figure 7.30 TiN and cBN-TiN coated carbide drills

Composite cBN-TiN coating is applied to Ø6.8 mm drill. Titanium nitride is formed as the initial layer to provide sufficient adhesion with the substrate material. After that, boron nitride is deposited together with titanium nitride and a cBN-TiN matrix is formed. Finally, cubic boron nitride layer becomes top surface of the drill to exhibit resistance to wear. Drills are mounted on the tool holder and rotated axially so that homogenous coating all over the drill surface can be obtained. Boron nitride coatings have been deposited for three hours at 150 °C and 100 V bias voltage. For Ø8.8 mm drill, only TiN coating is introduced for comparison.

After applying coatings on carbide tools, machining performance of the tools are investigated in the machine tools of the production plant. Both of the drills are tested at cutting speed of 80 rpm and feed of 0.15 mm/rev. According to the results, cBN-TiN coated carbide drill exhibits tool life of 135 minutes while uncoated carbide drill has tool life of 35 minutes. This result showed that cBN-TiN coatings can improve tool life almost four times. However, for TiN coated drill, no significant improvement is observed.

Similarly, for a different cutter type which is used for generating gear teeth, cBN coating is applied (Figure 7.31). Both bottom surface and side surface of the cutter is coated with only cBN. Experiment parameters are same with the drill experiments. Machining results indicate that tool life of the cutter improved form 35 pieces to 100 pieces.

Figure 7.31 Gear Tooth Generating Cutter

In addition to above ones, many studies have been implemented together with the companies from industry. Different types of cutters such as milling cutters, thread cutters, gears and die parts have been coated with cubic boron nitride. Machining tests are continuing in these days.

The feedback from the Coşkunöz Company indicated that the thread cutters coated by cBN are still being used after six months, whereas the uncoated tools only lasted two weeks.

CHAPTER 8

CONCLUSION

At the beginning of this study, main objectives are stated as; to establish a new laboratory for research and development, to form cubic boron nitride coatings on sample substrate surfaces and cutting tool surfaces and to implement that process to industrial applications. At the end of this study, it can be clearly said that these objectives are successfully achieved.

A new physical vapor deposition laboratory is established with the supports of BOREN (National Research Institute of Boron) and TUBITAK (Scientific and Technological Council of Turkey). Experimental setup is completely designed and produced by a local company in Turkey. Laboratory is equipped with necessary devices to execute the system properly.

Experimental setup is a physical vapor deposition system which works on magnetron sputtering principle and used to obtain mono or multi layer coatings by employing different target plates. As one of the primary objectives of this study, boron nitride coatings are formed onto substrates surfaces by using the PVD system. In order to obtain cubic boron nitride coatings (cBN), hexagonal boron nitride (hBN) plates are utilized. Most common two phases of the boron nitride are hexagonal and cubic phases and show quite different properties with respect to each other. Due to outstanding properties of cBN, it is used as a wear resistant coating on cutting tools, a protective coating, an optical coating and an electrical insulating layer. In this study, it is applied on cutting tool surfaces to improve wear resistance as well as tool life.

Hexagonal boron nitride target plates are produced and provided within this study. Al-B-Ti-Mg-hBN alloyed target plate is produced in METU Material & Metallurgical Engineering department facilities. Moreover, sintered hBN target plates are produced in Eskişehir Anadolu University. Finally, one hBN target plate is purchased from abroad and experiments are conducted by utilizing that plate. Comparison of the target plates will be done as a future work.

Initially, steel samples and silicon wafers are used as substrates for characterization. Samples and cutting tools are prepared to experiments by following stages such as heat treatment, polishing and ultrasonic cleaning.

Different types of coatings are formed on substrate surfaces such as TiN, TiAlN, cBN and cBN-TiN coatings. For cBN deposition, experiments are performed under $3\text{x}10^{-3}$ Torr pressure at 900 W magnetron power for 1-4 hours. Temperature is varied between 100-250 °C and substrate bias voltage is changed from 50 to 200 V. Distance between target plate and substrates is kept as 50 mm.

Several tools are utilized for structural, compositional and mechanical characterization. Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Photoelectron Spectroscopy (XPS), X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) are used for structural and compositional characterization. FTIR results demonstrate that 75% cubic phase is formed at 200 °C and 200 V which are the optimum parameters for cBN film formation. XPS results indicate that boron and nitrogen binding energies are seen at 190.3 and 397.5 eV, respectively. Furthermore, it can be said that nearly stoichiometric boron nitride films are deposited. In the XRD analysis, peaks belong to cubic and hexagonal phases are observed at 45.5, 49.4 and 73.2 ° theta angles. SEM analysis is carried out to investigate the surface morphology of the films. Delamination, micro cracks, voids and other defects are observed and EDX analysis is performed to quantify the elemental proportion of the coatings. EDX analysis results also verify the XPS results. As a result, it can be claimed that, according to structural and compositional characterization, cubic boron nitride formation is observed on substrate surfaces.

For mechanical characterization thickness, hardness, friction coefficient and adhesion of the coatings are measured. A calotest device is utilized for thickness measurements and it is found that 3 hour deposition leads to 0.8 μm of cBN film thickness. Due to high residual stresses during depositon, maximum cBN film thickness is limited to 1 μm. On the other hand, 2 hour deposition of TiN leads to approximately 4 μm in thichness.

Hardness of the substrates is measured by Rockwell C hardness device. After heat treatment of samples, hardness is evaluated approximately 55 HRc. In order to determine coating hardness, a nano indentation device is utilized. CBN and cBN-TiN coatings are tested and hardness values are found as 13 GPa and 15 GPa, respectively. Moreover, modulus of elasticity is measured as 280 GPa for cBN coating and 250 GPa for cBN-TiN multi-layered coating.

In order to determine friction coefficient, tribometer is employed and friction coefficient is evaluated as 0.5 against alumina material. Adhesion measurements are firstly carried out in the facilities of Erzurum Atatürk University by using a micro scratch device. Test results showed that under 80N loading, cBN coatings exhibit very good adhesion such that no side crack, fracture or failure is observed. Then, nano scratch device is used to test the adhesion of cBN-TiN multi-layered coating. According to the test results, critical loads which indicate the failure of the coatings are measured at 108 and 173 mN, respectively.

After obtaining satisfactory results, experiments are performed on cutting tools. Several types of cutting tools are coated with cBN and tested in the facilities of universities and private companies. A study is performed with Asst. Prof. Dr. Tuğrul Özel from Rutgers University to investigate cutting forces and wear of the coatings. Turning inserts are coated with cBN and tested in turning of Ti-6Al-4V material. Depth of cut is adjusted to 2 mm and feed is set to 0.1 mm/rev. Two different cutting speeds 50 and 100 m/min are used to make comparison. Measurements showed that CBN coated WC inserts has better performance at low cutting speeds. When the wear resistances of the coatings are compared, cBN coated cutting tools has the lowest flank wear both in low and high cutting speeds. As a result, it is can be claimed that CBN coatings reduce flank and crater wear by a factor of two in dry machining of titanium alloyed Ti6Al4V material.

Next, a collaborative project is made with Turk Traktor factory to see the machining performance of the cBN coated tools in terms of tool life. Tungsten carbide drills having diameters of Ø6.8 and Ø8.8 mm which are used to drill engine block made of cast iron are coated by cBN-TiN and TiN. Drills are tested at cutting speed of 80 rpm and feed of 0.15 mm/rev. According to the results, tool life is increased from 35 minutes to 135 minutes when cBN-TiN coating is employed on the tool. Similarly, for a different cutter which is used to generate gear teeth, cBN coating is applied and tool life of the cutter is improved from 35 pieces to 100 pieces.

This study has been supported by the Scientific and Technological Council of Turkey under research project no: 105M370 and National Research Institute of Boron under research project no: 2006.B0031. Results of this study are also presented in several international conferences and published in proceedings books and national journal. List of publications can be seen in *Appendix I*.

As a future work, experiment parameters may be optimized to increase the ratio of the cubic phase to achieve more hardness and more wear resistance. Furthermore, different target plates and reactive gases may be used to form multi-layered or composite coating systems. Besides improvement of tool life, cBN coated tools will be tested at different cutting conditions to reduce machining time.

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APPENDIX I

PUBLICATIONS

List of Publications (in chronological order)

1. Cesur H., Kaftanoglu B., Kalkanli A., Oral B., *Deposition of boron nitride coatings on steel substrates by rf magnetron sputtering,* 7th International Conference on Coatings, September 2008, Thessaloniki, Greece

2. Cesur H., Kaftanoglu B., Kalkanli A., Oral B., *Bor nitrür kaplamaların çelik alttaşlar üzerinde RF magnetron saçtırma yöntemiyle büyütülmesi*, Mechanical Design and Production Society Journal, (in press)

3. Cesur H., Kaftanoglu B., *An Experimental Study about CBN Formation for Cutting Tools,* 5th International Conference and Exhibition on Design and Production of Machines and Dies/Molds, June 2009, Kuşadası, Turkey

4. Cesur H., Kaftanoglu B. Özel T., *Machining Performance of CBN Coated Cutting Tools for Die/Mold Applications,* 5th International Conference and Exhibition on Design and Production of Machines and Dies/Molds, June 2009, Kuşadası, Turkey

5. Cesur H., Kaftanoglu B., *Applications of Cubic Boron Nitride Coatings on Cutting Tools*, 4th International Boron Symposium, October 2009, Eskişehir, Turkey (Abstract accepted)

APPENDIX II

Table II. Experiment parameters for cBN deposition

Target plate	Sintered hBN
Substrate	Si (100), steel disc samples
Base pressure (Torr)	$5x10^{-6}$
Deposition pressure (Torr)	$3x10^{-3}$
Ar/N_2 flow ratio	5/1
Target Power (W)	900
Substrate Bias voltage (V)	50-200
Heater Temperature (°C)	100-250
Target-Substrate Distance (mm)	50
Deposition duration (hr)	1-4

APPENDIX III

HARDNESS VALUES OF SUBSTRATES

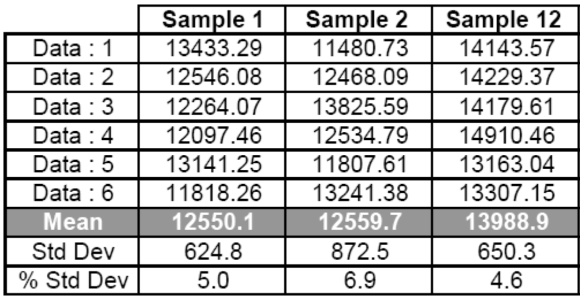
Table III. Hardness values of steel samples in terms of HRC

Sample No	**Hardness Values (HRC)**			
	1.Measurement	2. Measurement	3. Measurement	Average
1	52.6	52.7	53.3	52.9
2	52.5	52.3	52.5	52.4
3	52.4	52.7	53.0	52.7
4	53.0	53.4	53.6	53.3
5	56.2	54.7	55.8	55.6
6	53.0	53.0	53.5	53.2
7	58.7	58.5	56.6	57.9
8	55.5	55.7	54.6	55.3
9	53.4	53.5	53.5	53.5
10	54.8	54.3	54.9	54.7
11	53.2	52.8	53.6	53.2
12	54.3	54.3	54.1	54.2
13	53.3	53.0	53.6	53.3
14	52.4	52.4	53.8	52.9

APPENDIX IV

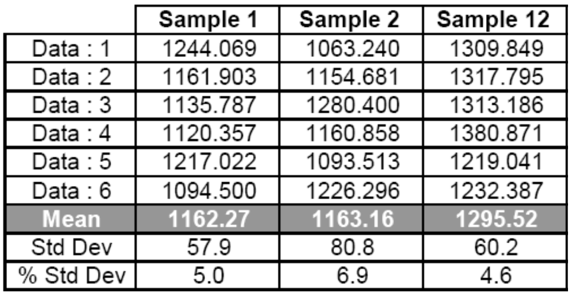
HARDNESS AND ELASTIC MODULUS MEASUREMENTS

Hardness (MPa):



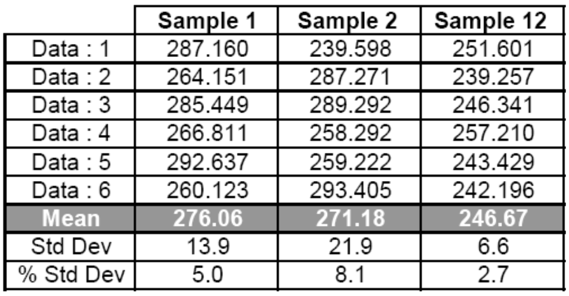
	Sample 1	Sample 2	Sample 12
Data : 1	13433.29	11480.73	14143.57
Data : 2	12546.08	12468.09	14229.37
Data : 3	12264.07	13825.59	14179.61
Data : 4	12097.46	12534.79	14910.46
Data : 5	13141.25	11807.61	13163.04
Data : 6	11818.26	13241.38	13307.15
Mean	**12550.1**	**12559.7**	**13988.9**
Std Dev	624.8	872.5	650.3
% Std Dev	5.0	6.9	4.6

Vickers Hardness (HV):



	Sample 1	Sample 2	Sample 12
Data : 1	1244.069	1063.240	1309.849
Data : 2	1161.903	1154.681	1317.795
Data : 3	1135.787	1280.400	1313.186
Data : 4	1120.357	1160.858	1380.871
Data : 5	1217.022	1093.513	1219.041
Data : 6	1094.500	1226.296	1232.387
Mean	**1162.27**	**1163.16**	**1295.52**
Std Dev	57.9	80.8	60.2
% Std Dev	5.0	6.9	4.6

Elastic Modulus (GPa):



	Sample 1	Sample 2	Sample 12
Data : 1	287.160	239.598	251.601
Data : 2	264.151	287.271	239.257
Data : 3	285.449	289.292	246.341
Data : 4	266.811	258.292	257.210
Data : 5	292.637	259.222	243.429
Data : 6	260.123	293.405	242.196
Mean	**276.06**	**271.18**	**246.67**
Std Dev	13.9	21.9	6.6
% Std Dev	5.0	8.1	2.7

APPENDIX V

X-RD OF H-BN TARGET PLATE

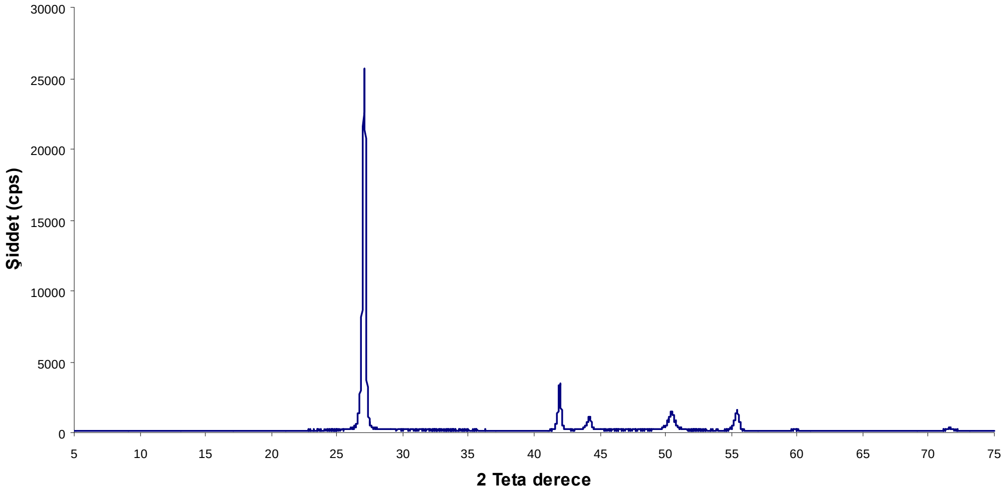


Figure IV. XRD analysis of h-BN target plate produced in Eskisehir Anadolu University

APPENDIX VI

IMAGES OF DIES FOR TARGET PLATE PRODUCTION

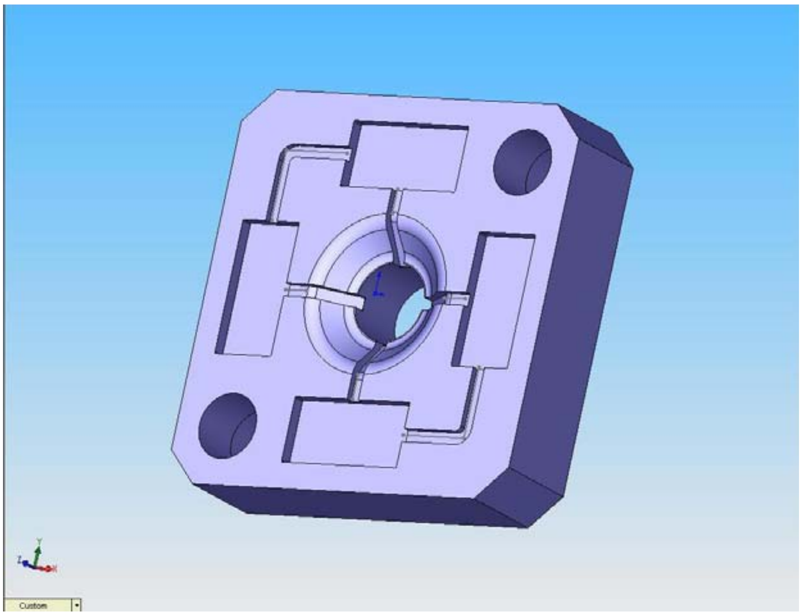


Figure VI.1. Die-1 Upper Part (for four plate production)

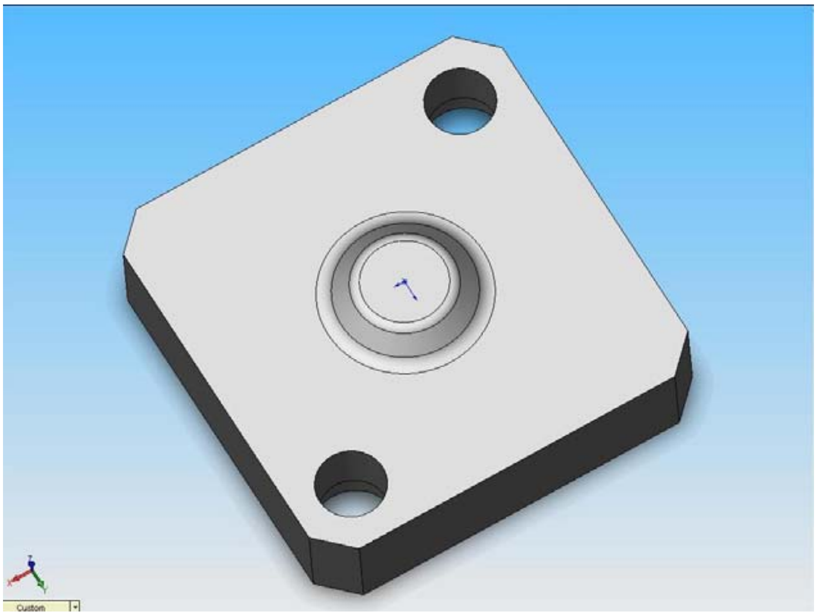


Figure VI.2. Die-1 Lower Part

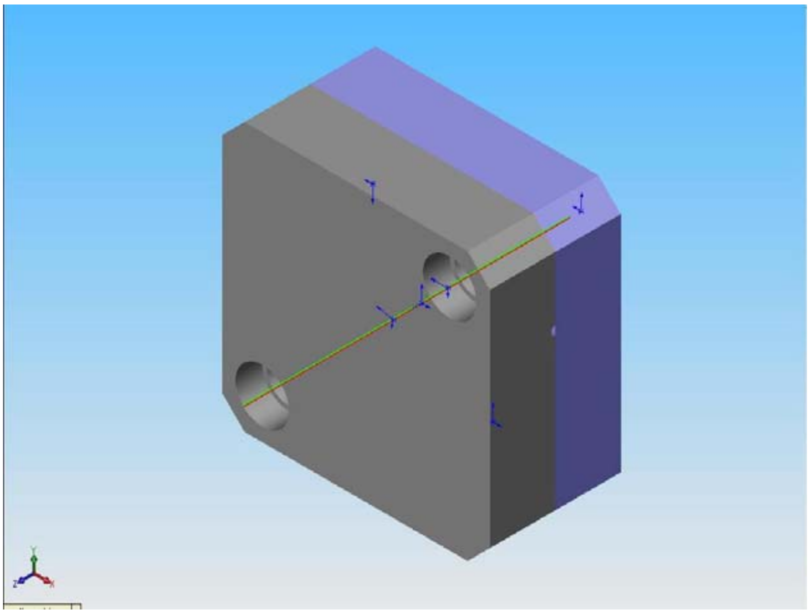


Figure VI.3. Die-1 Assembly View

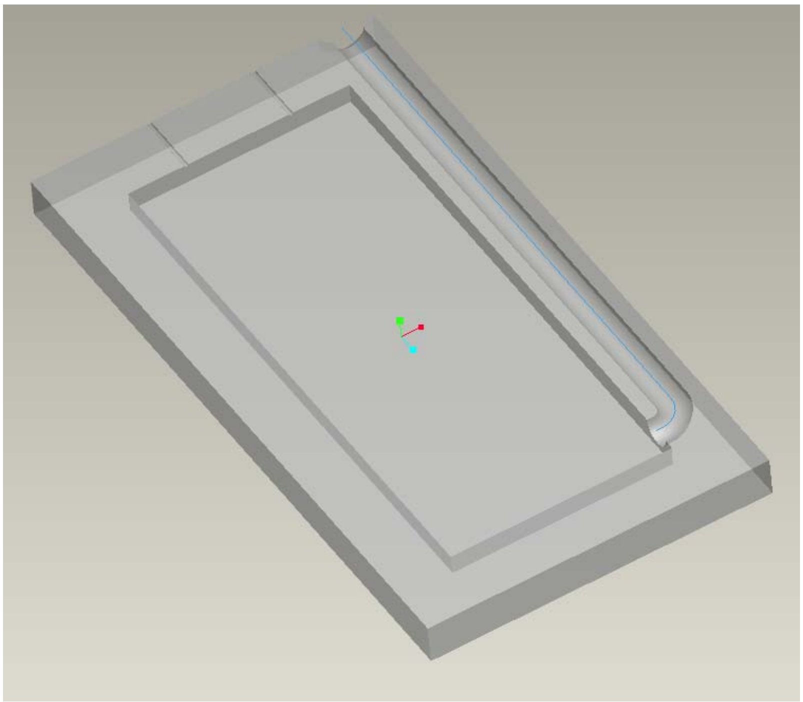
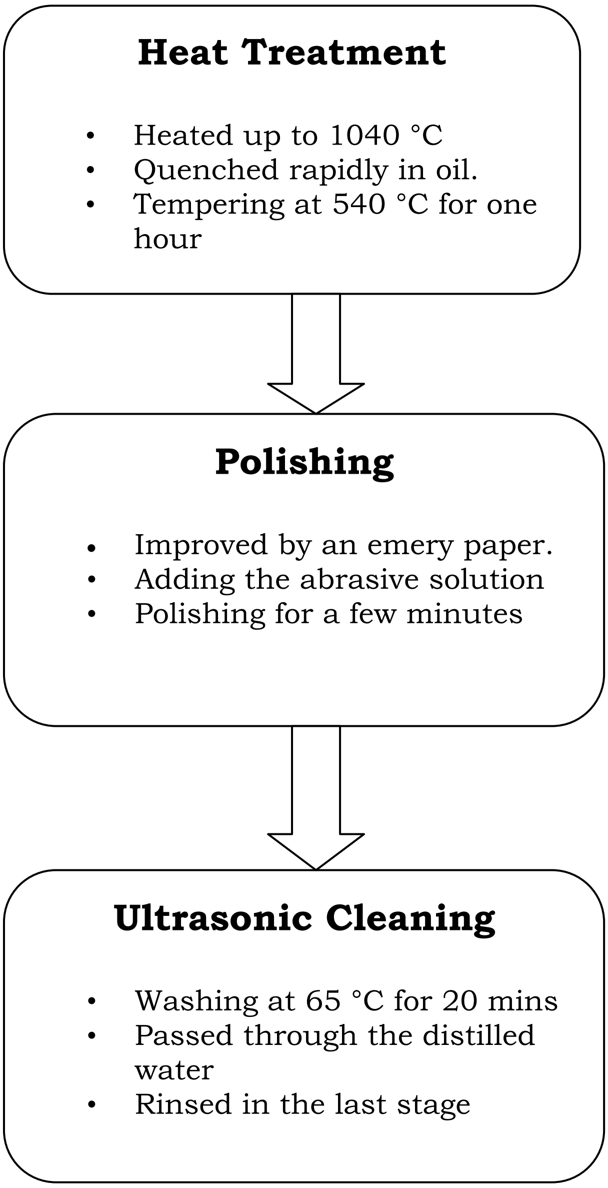


Figure VI.4. Die 2- Lower Part (for single plate production)

APPENDIX VII

FLOW CHART OF SAMPLE PREPARATION



Heat Treatment

- Heated up to 1040 °C
- Quenched rapidly in oil.
- Tempering at 540 °C for one hour

Polishing

- Improved by an emery paper.
- Adding the abrasive solution
- Polishing for a few minutes

Ultrasonic Cleaning

- Washing at 65 °C for 20 mins
- Passed through the distilled water
- Rinsed in the last stage

APPENDIX VIII

PROPERTIES OF TURNING INSERTS

Tool #1:

Insert Type:	TNMN-323T
Insert Material:	Solid Top PcBN (Pollycrystalline Cubic Boron Nitride-HTC2000 grade low PcBN content)
Micro-geometry design:	Uniform chamfer (20° chamfer angle, 0,1 mm chamfer height)
Major Applications:	Roughing and semi-finish turning of hardened steels (40 HRc or greater in hardness)

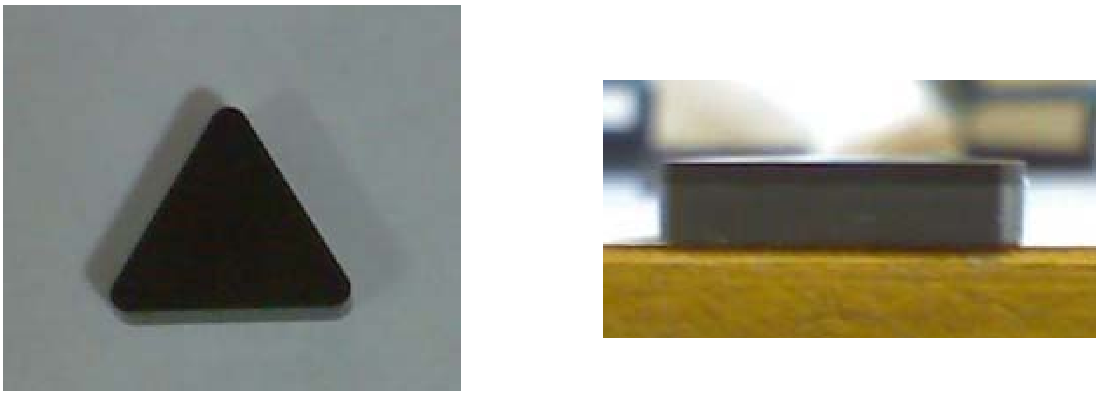


Figure VIII.1. TNMN-323T, PcBN turning insert- top and side views

Tool #2:

Insert Type:	TNMN-323H
Insert Material:	Solid top PcBN (Pollycrystalline Cubic Boron Nitride-HTC2000 grade low PcBN content)
Micro-geometry design:	Uniform Honed (0,020 mm edge radius)
Major Applications:	Finishing turning of hardened steels (40 HRc or greater in hardness)

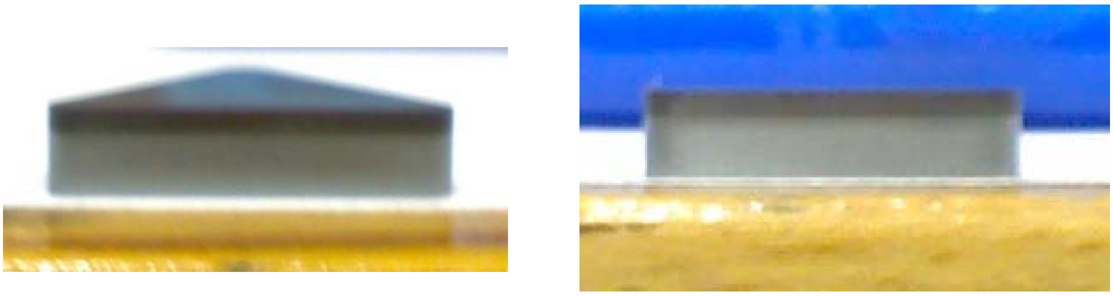


Figure VIII.2. TNMN-323H, PcBN turning insert- top and side views

Tool#3:

Insert Type:	TPG432
Insert Material:	Fine grain Tungsten Carbide (Uncoated) (Kennametal K313 grade)
Micro-geometry design:	Up-sharp (~0,003 mm edge radius)
Major Applications:	Finish turning of high temperature alloys (Titanium and Nickel alloys)

Figure VIII.3. TPG432, uncoated Tungsten Carbide turning insert- top view

Tool#4:

Insert Type:	TPG432
Insert Material:	Fine grain Tungsten Carbide (TiAlN coated) (Kennametal KC5010 grade)
Micro-geometry design:	Up-sharp (~0,005 mm edge radius)
Major Applications:	Finish turning of high temperature alloys (Titanium and Nickel alloys)

Figure VIII.4. TiAlN coated Tungsten Carbide turning insert-top view

APPENDIX IX

PVD LABORATORY

Figure IX.1. A view of the laboratory before system installation

Figure IX.2.A view of the laboratory after system installation

CLEANING & SAMPLE PREPARATION ROOM

Figure IX.3. A view of the cleaning & sample preparation room-1

Figure IX.4. A view of the cleaning & sample preparation room-2

GENERATOR ROOM & STORE ROOM

Figure IX.5. A view of the generator room

Figure IX.6. A view of the store room

APPENDIX X

SUPPORTERS & COLLABORATORS

SUPPORTERS

1. TUBITAK (Scientific and Technological Council of Turkey)
Research No: 105M370
2. BOREN (National Research Institute of Boron)
Project No: 2006.B0031

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3. Eskişehir Anadolu University, Prof. Dr. Nuran Ay
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6. Bilkent University, Asist. Prof. Dr. Erman Bengü
7. İstanbul Technical University, Prof. Dr. Hüseyin Çimenoğlu

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2. METU- MEMS (Micro-Electro Mechanical Systems)
3. METU- BILTIR Center

Industry:

1. CSM Instruments, Switzerland
2. Türk Traktor, Turkey
3. Coskunoz A.Ş. , Turkey
4. Vaksis R&D Engineering, Turkey
5. Van der Graaf Motors, Turkey
6. Element 6, South Africa
7. Som A.Ş., Turkey