

**EFFECT OF DIFFERENT SURFACE TREATMENTS
ON THE TENSILE AND WEAR BEHAVIOR OF
INCONEL 625 SUPERALLOY AND AISI 321
STAINLESS STEEL**

A Thesis

by

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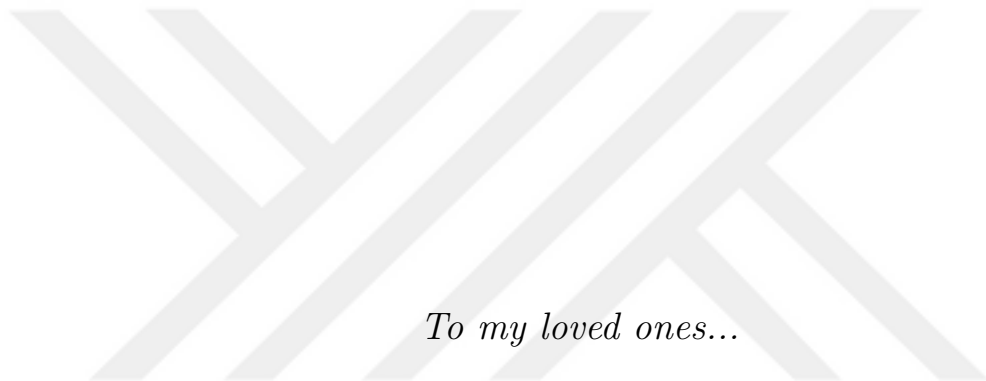
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To my loved ones...

ABSTRACT

AISI 321 stainless steel and Inconel 625 superalloy are utilized in an extensive range of applications such as aircraft engines, nuclear powerplants and industrial furnaces due to their excellent mechanical properties at high temperatures.

The surface treatment of these materials is necessary due to the extreme working environments. Boronizing and WC coating is the most important surface hardening processes. In the current work, the effect of boronizing and WC coating on the wear performance and tensile behavior of AISI 321 and Inconel 625 at room and high temperature were investigated. Furthermore, damage mechanisms and fracture morphologies were observed using Scanning Electron Microscopy (SEM) and optical profilometry.

According to room temperature tensile test results, yield strength (YS) and ultimate tensile strength (UTS) of the samples are reduced after the boronizing process. This strength reduction may be attributed to the softening of the material throughout the boronizing process at high temperature of 1100°C. On the other hand, ductility of the Inconel 625 samples has been decreased due to dynamic strain aging at 600°C. The yield strength of the boronized samples was significantly reduced because of the decomposition of boron particles throughout tensile test at elevated temperatures. Consequently, the fracture morphology observations revealed that the main fracture mechanism of both coated and uncoated conditions at high temperature was a ductile fracture represented by deep dimples. However, the boronized sample was drastically affected by grain growth during boronizing process and decomposition of dispersed particles.

In contrast, the strain to failure of the WC coated sample was decreased due to the fast fracture of the ceramic WC layer at both room and high temperature tensile tests. Furthermore, results of SEM revealed that particle decomposition

occured on the fracture surface of the boronized 321 stainless steel represented by dispersed boron particles on the edges of the dimples after failure at high temperature.

Due to the high hardness and heat resistance of WC coated layer, the friction coefficient of the sample was higher than that of boronized and uncoated conditions at room tempearature. It was seen that the wear rates of boronized and WC coated AISI 321 were extremely lower than that of uncoated sample. On the other hand, at 600°C, WC coated wear rate was still lower but uncoated and boronized samples' values were similar to the room temperature results.

Inspection of room temperature the wear test results revealed that the wear rates of Inconel 625 uncoated and coated samples were similar to the results of AISI 321. WC coated samples' wear rate was tremendously lower than the others. At high temperature, the boronized Inconel 625 had higher friction coefficient than the WC coated sample. A fluctuation was also noticed, which is linked to boron particle aggregation on the surface at high temperatures.

ÖZETÇE

AISI 321 paslanmaz çelik ve Inconel 625 süper alaşımı, yüksek sıcaklıklardaki mükemmel mekanik özelliklerinden dolayı uçak motorları, nükleer santraller ve endüstriyel fırınlar gibi geniş bir uygulama yelpazesinde kullanılmaktadır.

Zorlu çalışma koşulları nedeniyle, bu malzemeler üzerine yüzey işlemleri uygulamak bir gereksinim haline gelmiştir. Borlama ve Tungsten Karbür (WC) kaplama en önemli yüzey sertleştirme işlemlerindendir. Mevcut çalışmada, AISI 321 ve Inconel 625'in oda sıcaklığı ve yüksek sıcaklıktaki aşınma performansı ve çekme davranışı üzerine borlama ve Tungsten Karbür kaplamanın etkisi araştırılmıştır. Ayrıca, Taramalı Elektron Mikroskobu (SEM) ve optik profilometri kullanılarak hasar mekanizmaları ve kırılma morfolojileri gözlemlenmiştir.

Oda sıcaklığında çekme testi sonuçlarına göre borlama işleminden sonra numunelerin akma dayanımı (YS) ve nihai çekme dayanımı (UTS) azalmaktadır. Bu mukavemet azalması, 1100°C'lik yüksek sıcaklıkta borlama işlemi boyunca malzemenin yumuşamasına bağlanabilir. Öte yandan Inconel 625 numunelerinin sünekliği, 600°C'de dinamik gerinim yaşlanması nedeniyle azalmıştır. Borlanmış numunelerin akma mukavemeti, yüksek sıcaklıklarda çekme testi boyunca bor parçacıklarının ayrışması nedeniyle önemli ölçüde azalmıştır. Sonuç olarak, kırılma morfolojisi gözlemleri, yüksek sıcaklıkta hem kaplamalı hem de kaplamasız koşulların ana kırılma mekanizmasının, derin çukurlarla temsil edilen sünek bir kırılma olduğunu ortaya koymuştur. Bununla birlikte, borlanmış numune, borlama işlemi sırasında tane büyümesinden ve dağılmış parçacıkların ayrışmasından büyük ölçüde etkilenmiştir.

Buna karşılık, seramik WC tabakasının hem oda sıcaklığında hem de yüksek sıcaklıktaki çekme testlerinde ani kırılması nedeniyle WC kaplı numunenin sünekliği azalmıştır. Ayrıca, taramalı elektron mikroskobu sonuçları, yüksek sıcaklıkta

kırılmadan sonra çukurların kenarlarında dağılmış bor parçacıkları görülen borlanmış 321 paslanmaz çeliğin kırılma yüzeyinde parçacık ayrışmasının meydana geldiğini ortaya koymuştur.

WC tabakasının yüksek sertliği ve ısı direnci nedeniyle, aşınma testinde WC kaplı AISI 321'in sürtünme katsayısı borlanmış ve kaplamasız numunelere göre daha yüksek çıkmıştır. Borlanmış ve WC kaplı AISI 321'in aşınma oranı, oda sıcaklığında kaplanmamış numuneden çok daha düşüktür. Öte yandan, 600°C'de WC kaplı numunenin aşınma oranı hala düşükken kaplamasız ve borlanmış numunelerin değerleri, yüzeyin delaminasyonu nedeniyle hemen hemen aynıdır.

Inconel 625'in kaplanmamış ve kaplanmış numunelerin aşınma hızı davranışı AISI 321'in sonuçlarına benzerdir. WC kaplı numunelerin aşınma hızının diğerlerinden çok daha düşük olduğu görülmüştür. Yüksek sıcaklıkta, borlanmış Inconel 625, WC kaplı numuneden daha yüksek bir sürtünme katsayısına sahiptir. Yüksek sıcaklıktaki sürtünme katsayısı grafiğinde yüzeyde bor parçacıklarının birikimiyle bağlantılı bir dalgalanma gözlenmiştir.

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CHAPTER I

INTRODUCTION

1.1 Motivation

In mechanical systems, it is much more attractive to use unalloyed or low-alloyed materials that can be easily shaped, provide the necessary structural properties, by applying different surface techniques, instead of expensive materials with high wear resistance. Thus, the surface treatment gives the main material new features that it does not have. With the developments in recent years, surface coating and surface hardening techniques to increase wear resistance have led to revolutionary changes in the selection and design of engineering tools and elements. There are different types of surface treatment technique according to modification of the microstructural and chemical composition of the base materials.

There are three main surface treatment methods in the literature. These three methods are modification of the component surface without microstructural change, modification of the component surface with microstructural and chemical change and material implementation on surface.

Although, many research have been done to identify the effect of surface treatments on the surface mechanical behavior of different materials, there is a lack of information about the bulk properties of coated materials. The bulk mechanical behavior of the coated samples may be changed, adversely due to the effect of bonded coated layer on the fracture mechanism. Moreover, due to the wide range of the high temperature applications of coated materials, the impact of various coating methods on the elevated temperature tensile and related fracture mechanism have to be investigated.

1.2 Research Objectives

In the current thesis, two different materials of Inconel 625 superalloy and AISI 321 stainless steel were selected to investigate due to their wide applications in various industries such as aircraft engines, industrial furnaces, nuclear powerplants and automotive industry. The effect of boronizing and WC coating on the surface mechanical behavior and bulk mechanical properties of the mentioned materials were investigated at room and high temperature up to 600°C. To this end, ball-on disc wear tests and tensile tests were conducted on the samples before and after coating. The tested samples were inspected via scanning electron microscopy (SEM), energy dispersive spectrography (EDS), optical and confocal profilometer to identify the failure and fracture mechanisms.

CHAPTER II

BACKGROUND AND LITERATURE REVIEW

2.1 Background of the work

In the last two decades, nickel based superalloys and austenitic stainless steels are widely utilized in many significant engineering systems such as aircraft engines, nuclear powerplants, industrial furnaces and automotive industry due to their excellent mechanical properties at high temperatures. However, despite their excellent mechanical properties, those materials are commonly subjected to wear due to the mechanical effects in the system they are being used. Since wear leads to the formation and progression of fractures, many of those system fail and result in financial loss. Abrasion and corrosion cause significant financial losses every year. Wear and corrosion caused losses of countries varies between 3,5% to 5% of their national income. Consequently, the demand of the industry for durable and steady materials increased considerably. Materials resistance to external effects is related to its bulk and surface properties. The operation of the material at the service conditions without any plastic deformation depends on its bulk properties such as the modulus of elasticity, yield and tensile strength. On the other hand, possible surface defects such as wear and corrosion can be caused by the material's surface properties. To avoid that, anti-wear surface improvement processes are highly on demand in recent years.

Despite the significant improvement in surface mechanical properties after coating, the bulk mechanical behavior of the coated substrate material may be altered adversely due to the effect of bonded coated layer on the fracture mechanisms [1]. According to the literature, a few papers have been published to declare the bulk mechanical behavior of coated materials such as tensile and compressive response [2, 3]. Gopkalo et al. investigated the effect of the different types of PVD

coatings of on the mechanical behavior of the AISI 321 stainless steel and titanium alloys. The results show that yield strength of the samples were increased with the 4-10 μm thickness of PVD coating methods. The results are shown in Figure 1.

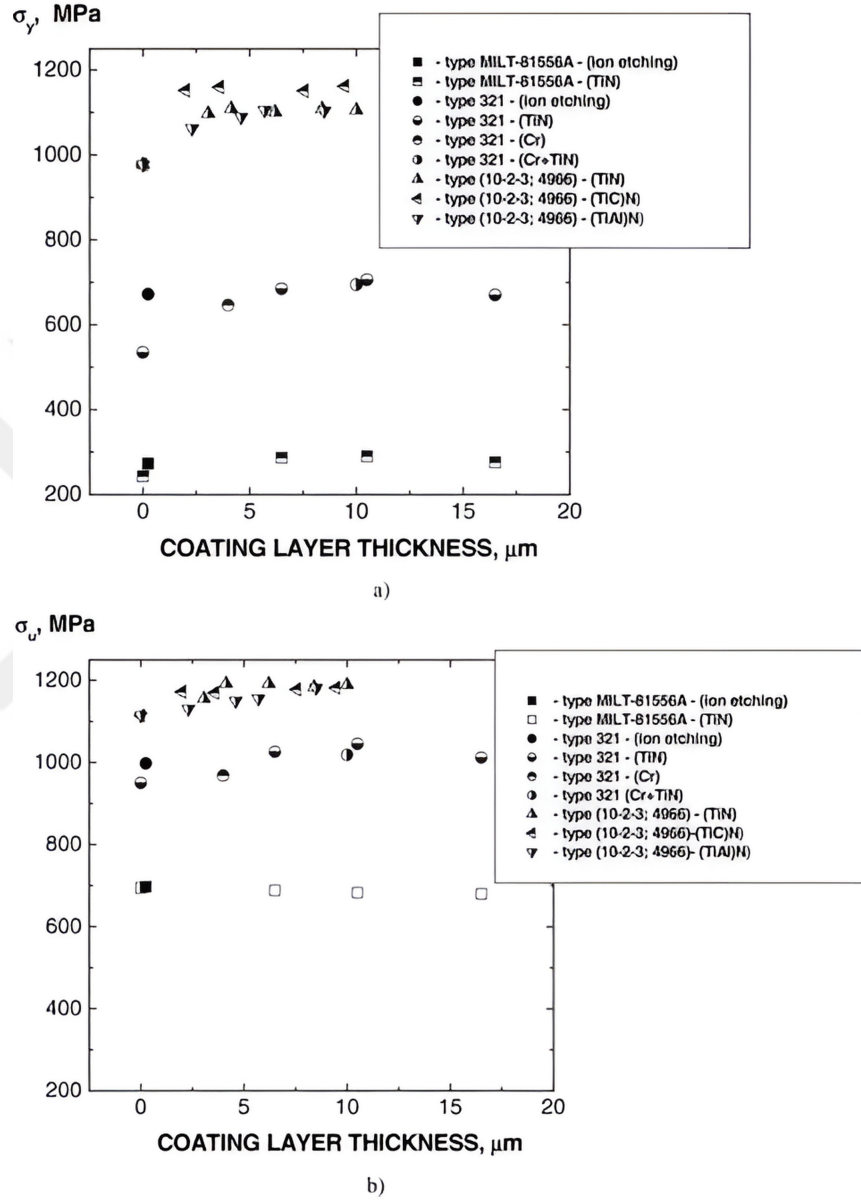


Figure 1: Yield and tensile strength of the PVD coated AISI 321 and titanium alloys [2]

Li et al. have been investigated the microstructure and tensile properties of the AISI 321 stainless steel with aluminizing coating and annealing treatment after

the coating process. The results show that the strength and the plasticity of the aluminized steel is lower than the uncoated samples. However after the annealing treatment, the ductility increased while the strength was still lower [4].

Tugay et al. investigated the effects of the burnishing method, which provides surface improvement with plastic deformation, on the wear behavior. It was observed that the surface hardness of the burnishing method increased by close to 45%, while the amount of wear was considerably reduced [5].

Tehrani and collaborators, made WC-Ni coating on AISI 321 material with laser cladding method and examined its mechanical properties. They determined the modulus of elasticity with the Nop Indentation Method and found the fracture toughness values with Evan's model. However, no study has been conducted on the tensile behavior of the coated sample [6].

Xu et al. investigated the tensile behavior by applying coatings with different ratios of tungsten carbide content on grade 45 steel. According to the results, the ratio of WC content changes the fracture behavior of the material as elastic or inelastic by affecting the elastic modulus of the material [7].

In this study, among the anti-wear coatings, Boronizing and Tungsten Carbide plasma spray coating methods are applied on Inconel 625 nickel superalloy and 321 grade stainless steel.

Although the mechanical properties of Inconel alloys at high temperature are investigated in detail, the fracture morphologies of coated and uncoated surface of structural parts need further investigation. Also, the number of studies related to the effect of material mechanical properties on the wear behavior is not adequately enough. In addition to fracture morphology, these coating methods are compared on coated and uncoated materials by being subjected to wear testing at room and high temperature.

2.2 *Materials*

With the development of the technology, materials used in industry have left their places to new ones. In the 1800's, Titanium has been discovered by the mineralogist and chemist William Gregor. Obtaining titanium from titanium oxide has taken place over a hundred years. Titanium alloys began to be used rapidly in the industry in the 1900s due to their mechanical properties [8]. However, the corrosion (oxidation) state of titanium and its alloys at high temperatures is 7-8 times higher than at room temperature, has led mankind to research new materials to be used at high temperatures. The first superalloys were developed between 1950 and 1970 as a modification of the Austenitic stainless steels [9].

Superalloys are materials that can operate at an average of 60% of their melting temperature, yet can withstand oxidation, high strains and stresses while maintaining their metallurgical stability. In general, they are examined in three groups as Ni, Fe and Co-based [10].

Superalloys maintain their mechanical and metallurgical stability up to 900-1050 °C [11]. Nickel-based alloys are known for their excellent resistance to corrosion and oxidation [12]. Superalloys are frequently preferred in especially high temperature applications such as combustion chamber, high- and low-pressure turbine modules of gas turbine engines, reaction molds in petrochemical industry, pipes and pumps, control rods, springs and flow valves in nuclear power systems [13].

Inconel 625 is a nickel-chromium-based superalloy. It is an alloy used due to its high strength, easy machinability and weldability and superior corrosion resistance. The operating temperature range of Inconel 625 is from freezing cold to 982°C [14]. The high strength of Inconel 625 comes from the reinforcing effect of molybdenum and niobium additives on its nickel-chromium base. Therefore, precipitation hardening processes are not required for Inconel 625 as in other nickel-based alloys [15]. Another task of these elements in Inconel 625 is to provide high resistance to corrosive environments in a wide operating range and high

temperature effects such as oxidation and carburization. The main features that make Inconel 625 a good choice for use in gas turbine engines are its good corrosion resistance even at high temperatures, its weldability, no need for post-weld heat treatment and high fatigue resistance [16].

There is different information about the first production of stainless steels in history. In one of them, after the First World War, a European scrap dealer sees a shining cannon among rusty scraps. Wondering why it doesn't rust like other steels, he has it analyzed. As a result, he learns that the steel contains a high amount of Cr. After the discovery, stainless steel production begins in Europe. In 1911, C. Dantsizen produces a Fe-Cr alloy containing 14-16% Cr for use as a filament in electric lamps at General Electric. In the same years, Harry Brearley in England developed an alloy containing 12.8 Cr to make cutlery, spoons and knives by taking advantage of the high corrosion resistance of Fe-Cr alloys. The same researcher called these alloys "stainless steel" because of their resistance to metallographic reagents [17].

After obtaining the patent of the alloy developed as a result of long studies, Krupp was put into the service of the industry under the name of V2A steel (20% Cr, 7% Ni, 0.25% C) and the first stainless steel production on an industrial scale was realized by Maurer. Since the 1920s, as a result of the development of production techniques and the increase in production quantities, ferritic with 17% Cr and austenitic stainless steels containing 18% Cr-8 Ni have been widely used in the chemical industry [18, 19].

The difficulties encountered in finding Ni for austenitic stainless steel due to war conditions in the 1950s accelerated the studies on the usability of Mn and N instead of this element and led to the development of austenitic stainless steels, which are defined as the AISI 300 series today. As a result of the studies on the development of stainless steel types to meet the very different requirements of the industry, new types of ferritic stainless steels and super-austenitic stainless steels containing very little carbon and nitrogen and very few impurities were produced,

as well as duplex (double-phase) and precipitation hardening steel types [20].

Austenitic stainless steels are stainless steel types that contain chromium and nickel to provide an austenitic structure against corrosion, are non-magnetic at room temperature, cannot be hardened by heat treatment and are suitable for cold forming. AISI 321 type austenitic stainless steels are used in areas such as aircraft exhaust manifolds, cabin heaters, heat treatment equipment, etc., up to temperatures up to 800°C, because they can be used in corrosive environments without losing their mechanical properties, and are easily shaped and weldable [21, 22].

2.3 Surface Treatments

In mechanical systems, it is much more attractive to use unalloyed or low-alloyed materials that can be easily shaped, provide the necessary structural properties, by applying different surface techniques, instead of expensive materials with high wear resistance. Thus, the surface treatment gives the main material new features that it does not have. With the developments in recent years, surface coating and surface hardening techniques to increase wear resistance have led to revolutionary changes in the selection and design of engineering tools and elements.

Today, surface treatments, which have a very rich range for designers and users, are examined under the roof of "surface engineering" and generally under the headings given in Figure 2.

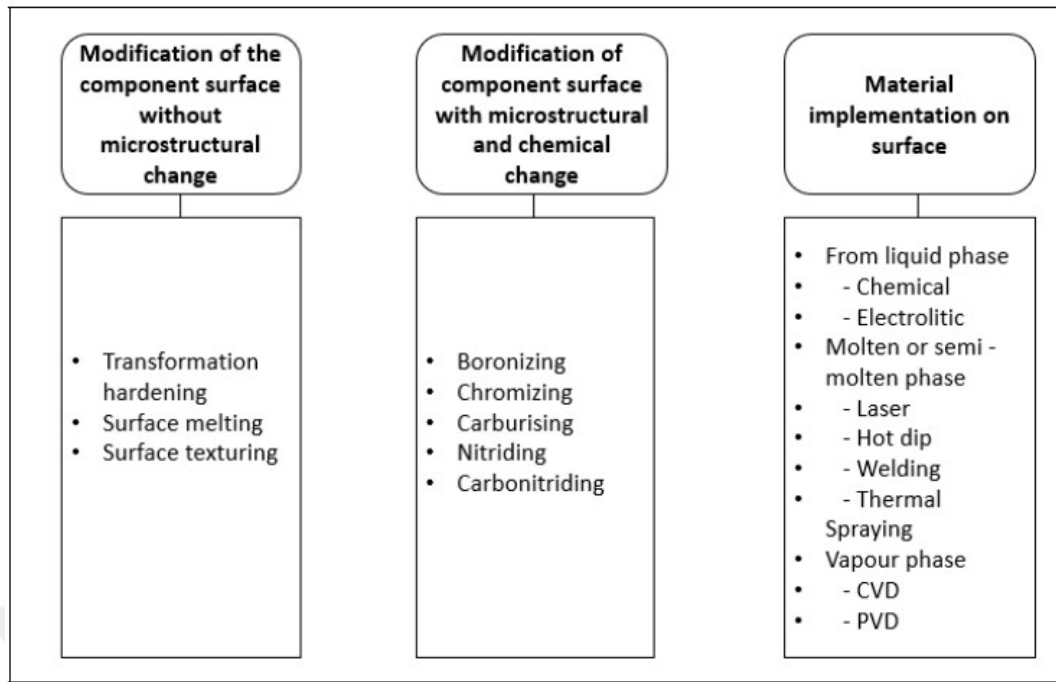


Figure 2: Surface Treatments [23]

Surface treatments performed by changing the surface of the base material are applied by changing the microstructure (quenching etc.), chemical structure (boronizing etc.) or both chemical and microstructure (cementation etc.) of the surface.

Heat treatment is defined as controlled heating or cooling operations applied to change the mechanical properties of materials in the desired direction. Heat treatments are carried out for the purposes of hardening, softening, creating a surface with low friction resistance, changing the grain structure, homogenization, stress relief and similar purposes. As a result of surface treatments performed without changing the surface of the base material (coating, etc.), the bond between the base material and the coated layer, that is, the surface, is generally weak. This is one of the important disadvantages of surface engineering, which has been studied intensively in recent years.

2.3.1 Modification of the Component Surface Without Microstructural Change

It is a process that is generally applied to medium and high carbon steels. The process is based on the principle of heating the material up to the austenitic transformation temperature and rapidly cooling it to obtain a martensitic structure. As a result of these processes, structures of different thickness and hardness can be obtained from the surface of the base material to the inside, depending on the applied process conditions. The hardness to be obtained depends on the carbon ratio in the material, the type and amount of alloying elements, the heating temperature, the holding time at this temperature, the cooling medium and its speed. The most important disadvantage of this process in terms of tribology is that the hardness created by temperature change decreases due to temperature increases that will occur during operation. The most important advantages are that the process is simple and the cost is low, it can be applied to the desired areas, large parts such as bench slides, and the hardness distribution from the surface to the inside is balanced. Thus, resistance to most of the wear mechanisms is increased in dynamic, impact, point or linear loading situations. It is widely applied to machine slides, tooth surfaces of gear wheels, camshafts and their counterparts, crankshafts, bearings and similar tribological system elements. As a result of these processes, since only the surface of the steel hardens, that is, the core remains soft, high wear resistance on the surface and high toughness on the core increase resistance against variable stresses. Here, there is an optimization between two opposite properties (hardness and toughness) in the same material.

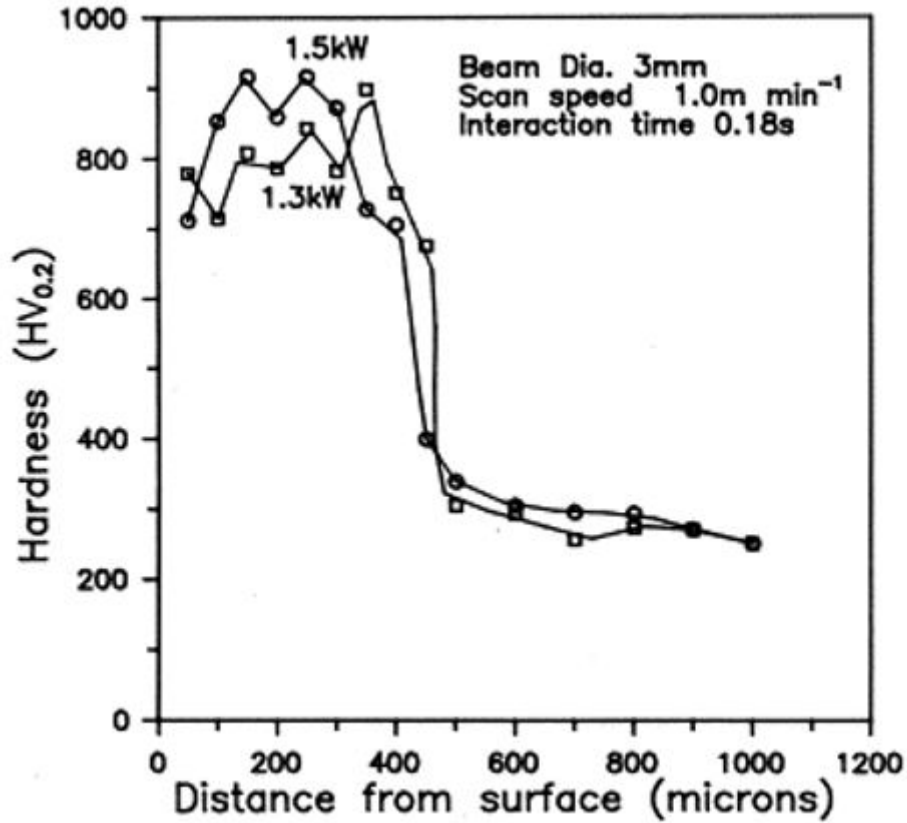


Figure 3: Hardness and distance from surface relationship after surface hardening of AISI 5135 Steel [24]

The shape and dimensions of the part directly affect the success of the method. While there is no problem in terms of size and shape in prismatic or cylindrical parts with homogeneous cross-sections, warping or cracking may occur as a result of overheating in changing cross-sections and especially in very thin sections. In order to prevent stresses in the parts and deformation and cracking due to these stresses, it is recommended to preheat before the process and a normalization annealing after the process. This method is applied using different techniques. The most common of these are induction, flame, dipping or melting hardening.

Recently, hardening can be done with laser and electron beams. In laser hardening, high-power infrared rays are sent directly to the steel surface and the surface is heated. In the second stage, the surface is cooled rapidly, and hardening is achieved. The method of sending the electron beam is also very similar to the

laser.

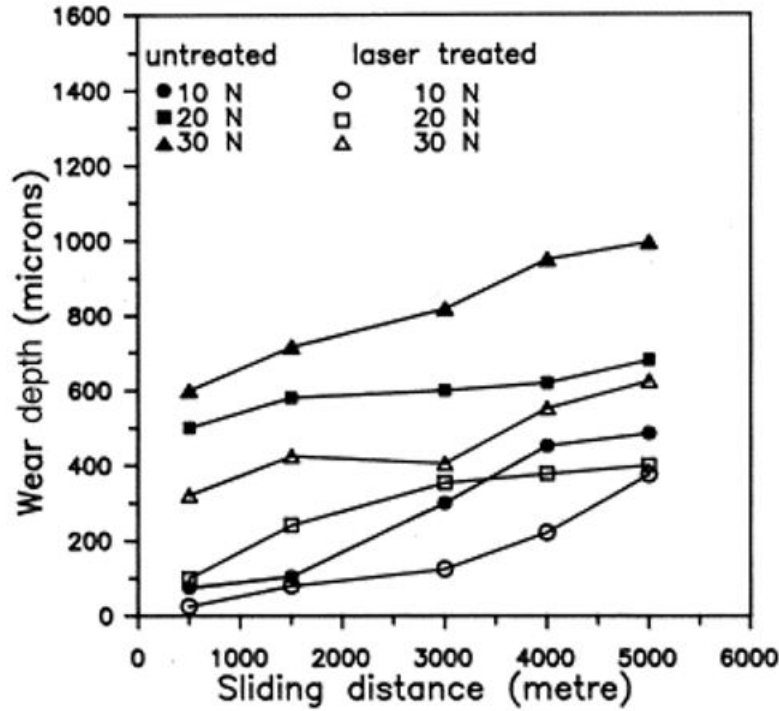


Figure 4: Wear behavior between untreated and laser surface hardened AISI 5135 steel [24]

2.3.2 Modification of Component Surface With Microstructural and Chemical Change

Boronizing is the most important hardening process applied by changing the chemical structure of the surface. Boronizing process is one of the most suitable methods in terms of both friction and wear among the surface hardening processes [25]. Since boron has a high affinity for oxygen, it acts as a natural solid lubricant by forming boroxide layers on the surface. Boronizing temperature varies between 800–1050 °C depending on the material to be boronized [26]. Boronizing time varies between 15 minutes and 30 hours [27, 28]. Boronizing process is carried out in environments prepared with boron sources such as amorphous boron (B), ferrous boron (FeB) and boroncarbide (B_4C), activators (KBF_4 , alkali metals, ammonium borofluorides, etc.) and deoxidants (SiC , Al_2O_3 , etc.). Activators help the layer to grow regularly, and deoxidants help to form a reducing environment

by keeping oxygen at boronizing temperature. The methods applied for the formation of boride layers are classified as chemical methods (boronizing in solid, liquid and gaseous media) and physical methods (boron ion grafting, physical vacuum deposition, scattering and ion plating).

Boronizing in solid environment is similar to box cementation. It has a wide application area due to factors such as ease of operation, easy changing of the powder composition, simplicity of the necessary equipments, and being cost effective. In Figure 5, the microstructures of the boride layers formed on the surface of the boronized steel material in solid environment are given. As can be seen from the figure, boride layers vary depending on the chemical structure of the material, but generally have a needle-like structure. This structure flattens with increase of the layer thickness. Boride layers with a thin and acicular structure are preferred for wear resistance, which have a very good connection with the base material and are less risky in terms of cracking and spilling and causing abrasive wear.

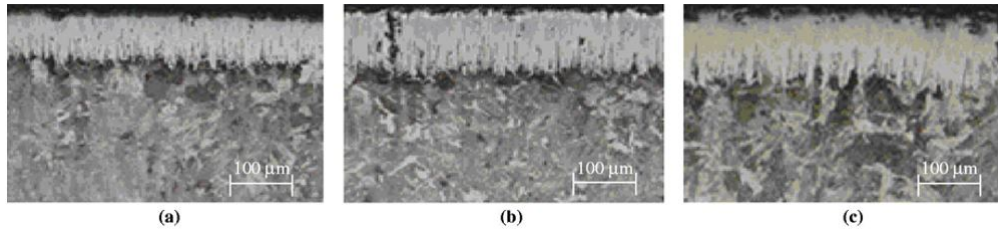


Figure 5: Microstructures of boronized layer on AISI 1035 steel at 920 °C a) 2h b) 4h c) 6 h [29]

Alloying elements affect the crystal structure of the boride layer and the transition zone, its composition (phase structure), the total layer and transition zone thickness cause significant changes in the mechanical properties. The effect of alloying elements also varies depending on the ability to penetrate the boride layers preferentially and interact with boron and form stable compounds. In Figure 6, the changes of the hardness on the coated surface according to the boronizing temperature are given.

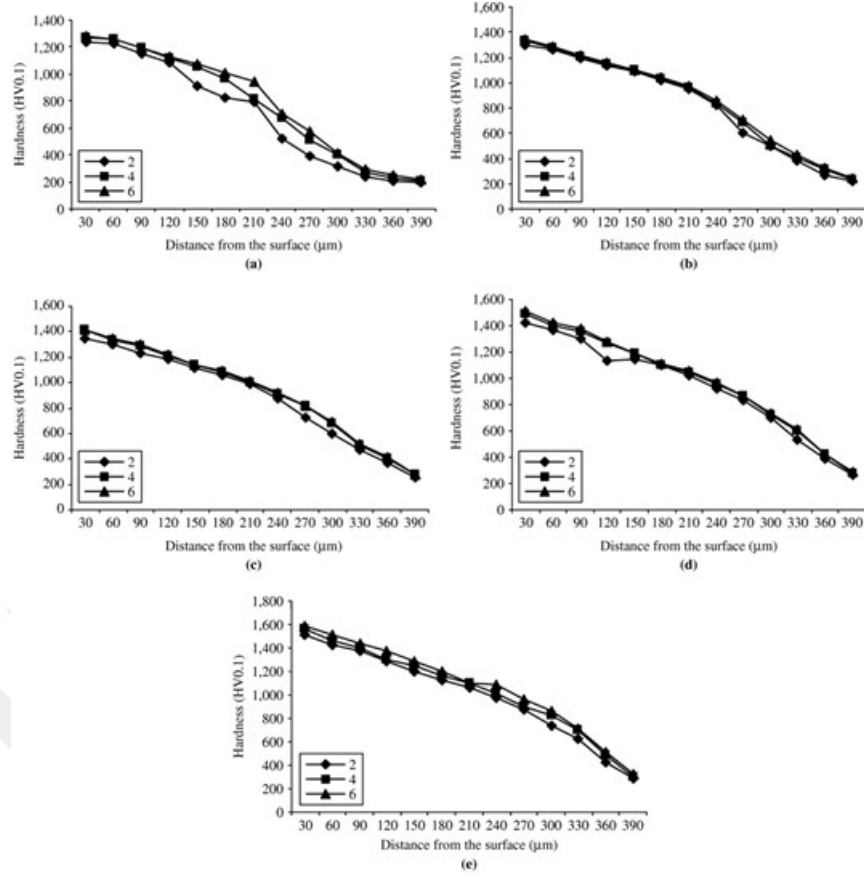


Figure 6: Hardness change at a) 840 °C; b) 880 °C; c) 920 °C; d) 960 °C; e) 1000 °C [29]

The increase in surface hardness in boronized materials depends on the FeB and Fe₂B phases formed on the surface. The FeB phase is much harder, but high hardness brings a brittle structure. Especially the FeB layer is a very brittle phase. Therefore, the less brittle single-phase Fe₂B layer is preferred.

For abrasive and erosive wear resistance, the material is required to be harder than the abrasive and the friction coefficient is low. These properties can be provided to a large extent by boronizing. The friction coefficients on the boronized steel surfaces are quite low.

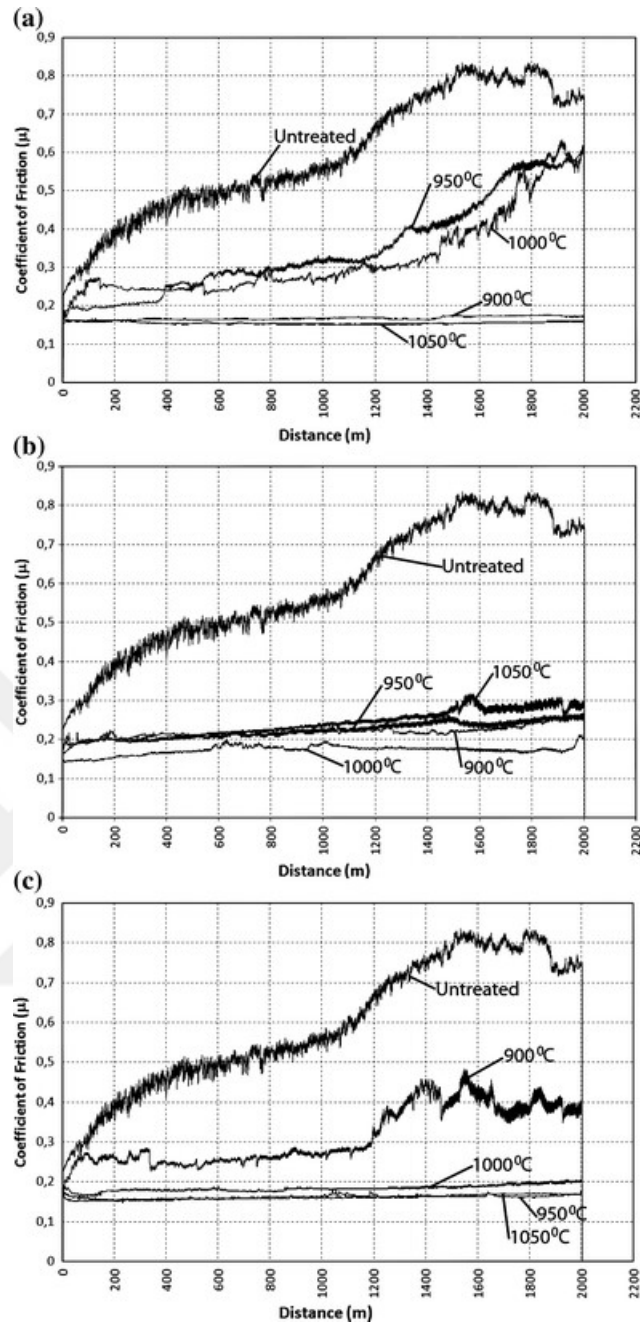


Figure 7: CoF of AISI 4140 samples at different boronizing time at 1050 °C a) 2h; b) 4h; c) 6h [30]

Changes to be achieved in processes applied by changing only the microstructure are limited to the chemical composition of the parent material. In the processes applied by changing the chemical structure of the surface, new properties can be obtained in a wide range and very different from the main material. In some processes, the microstructure can be changed first and then the chemical

structure can be changed (nitriding, etc.), while in others, the chemical structure is changed first and then the microstructure is changed (carburizing, etc.).

The carbonitriding process is a combination of carburizing and nitriding processes. Carbon and nitrogen are diffused into the metal simultaneously and in large quantities. Since nitrogen lowers the transformation temperature very much, cementation should be done at low temperature at the same time as nitrogen diffusion. Since the austenite field still exists in the temperature range of 650 – 700 °C, austenite carbon diffusion can also be performed at these temperatures where nitrogen diffusion can be rapid. If sudden cooling is made from this temperature, martensitic structure can also be reached. With this method, a better core structure is achieved compared to hardening with normal cementation. Carbonitrided parts below 590 °C have a remarkable increase in wear resistance, especially in the joint area. Therefore, the use of alloy steels becomes mandatory only when certain properties are required in the structural members and tooling. For example, additional improvement in wear resistance is achieved with the bond layer in hot work tool steels and speed steels whose tempering temperatures are approximately at the level of the carbonitriding temperature. In addition to the simultaneous diffusion of nitrogen with carbon or oxygen, it is also possible to obtain nitride-containing layers with additional elements, especially at low temperatures. Examples of these are sulfonitriding with sulfur, oxycarburizing with carbon and oxygen. Carbonitriding can also be applied to steel materials produced by powder metallurgy.

Process (and compound formed)	Hardness (HV)
Thermal hardening (0.5 wt.% C steel)	700
Carburizing and carbonitriding	850–900
Nitriding:	
Cr-Mo steel	650
Cr-Mo-V steel	900
Cr-Mo-Al steel	1100
Nitrocarburizing (forms $\text{Fe}_2(\text{C},\text{N})$)	500–650
Boronizing (forms FeB , Fe_2B)	1500
Chromizing (forms Fe_2Cr_3)	1500
Toyota diffusion (TD) process	3000–4000

Figure 8: Maximum allowable hardness limits by various thermochemical methods [23]

2.3.3 Material Implementation on Surface

The surface treatments to be examined under this title are called coating. The most important difference of coatings from those described in other titles is that the bond formed between the base material and the coated layer in coating processes is not chemical but mostly mechanical. Today, industrially suitable coatings can be made with the combination of plastic formable and tough metallic materials and ceramics with high temperature and wear resistance. Coatings made in order to increase abrasion resistance and reduce friction resistance are applied in liquid phase, gas phase or solid phase by plasma spraying and ion implementation methods. Liquid phase coatings are widely used metal coating techniques in aqueous or organic solutions using chemical or electrochemical methods.

In the coating by means of homogeneous chemical reactions in aqueous solutions, the coating solution and the reducer are sprayed onto the surface to be coated, so that the reduction reaction takes place on the surface. In electroless coatings, the ions to be coated and the reducer are in the same solution. As a result of the reaction that develops with the catalytic effect of the surface to be coated, a coating develops on the surface. Electroless coating is an important coating method with increasing industrial importance. The biggest advantages of

such coatings are that they can be coated with equal thickness everywhere, regardless of the surface shape, some types can be hardened by heat treatment and allow easy composite coating. These coatings have superior corrosion resistance and hardness.

Wear-resistant layers with very fine powder SiC, WC, CrC and Al₂O₃ added to the electroless coating bath; Teflon, graphite, calcium fluoride and even small polyethylene globules with oil in the coating can create surface layers with excellent friction properties. Conversion coatings are formed by the reaction of metals with liquid solutions that can react with the material and form protective films. Chroming, phosphating, and anodic oxidation are the most well-known and widely used conversion coatings. Phosphating is applied as a substrate for limited corrosion protection of steel and aluminum surfaces. Chroming, on the other hand, is a very common type of coating on steel, aluminum, magnesium and zinc surfaces that both increases corrosion resistance and allows oil films to adhere well to the surface. Anodic oxidation is commonly applied to aluminum and its alloys.

Metal and alloy coating with electrolytic method is one of the oldest known coating methods. Provided that the surface is conductive, any material can be coated with almost any metal desired. Recently, studies on composite and multi-layer coatings have been intensified to give different properties to surfaces. Electrolytic cadmium coatings are especially used in the protective coating of iron and steel products. Cadmium is a silver-white metal that is very durable, especially in salty and humid environments. The higher passivation tendency of iron compared to cadmium makes the potential of iron more positive than cadmium under normal conditions. Therefore, cadmium is always more active than iron in atmospheric conditions. Therefore, it protects the iron cathodically. Likewise, electrolytic chromium plating is applied on steel alloys to impart properties such as high wear resistance and low coefficient of friction. Glossy and very hard surfaces are formed in chrome plating. However, this hard surface can cause cracking and collapse under the impact loading [31].

In tribological processes, mostly hard chrome plating is applied. One of the reasons why hard chrome coatings gain importance in the industry is that the coating has a very low surface energy and therefore a low coefficient of friction. The wear resistance of hard chrome plated surfaces mainly depends on its hardness and low surface energy of the coated metal. The average hardness of hard chrome plating is around 800-1000 HV. Hard chrome plating is resistant to corrosion and thermal effects. At around 400 °C the hardness drops slightly, at 700 °C the hardness decreases considerably and above 1200 °C the hardness drops to that of pure chromium [32]. Hard chrome plating is applied in parts such as cutting tools, plastic molds, sheet metal rolling, extrusion screws, turbine shafts, chemical industry.

Coatings made in the vapor phase are the coating techniques that have been the most concentrated in recent years among all coating techniques, and very rapid technological and scientific developments have been achieved. One of the main reasons for this is that ceramic coatings can be made quite easily using these new technologies. Coatings made in the vapor phase are grouped under the following main groups:

- Chemical Vapor Deposition (CVD)
- Physical Vapor Deposition (PVD)

Chemical vapor deposition techniques are the oldest in industrial application among coatings made in the vapor phase and are used to reduce wear on hard metal cutting tools (TiN, TiC, alumina, etc.). The basic principle of CVD is that a volatile compound of the metal to be coated is sent by means of a chamber carrier (inert or reactive gas) under vacuum and coated as metallic or compound as a result of the chemical reaction occurring on the preheated base material. The biggest disadvantage of CVD is the necessity of heating the base material to high temperatures such as 800 – 1000 °C in order to create the desired coating and the difficulties it brings for the materials to be coated. The biggest advantages are that the coating adheres very well to the base material and a homogeneous

coating thickness is obtained everywhere, regardless of the shape of the material to be coated. All types of wear resistant coatings (borides, carbides, nitrides, oxides, carbonitrides and oxycarbides) can be coated with this method.

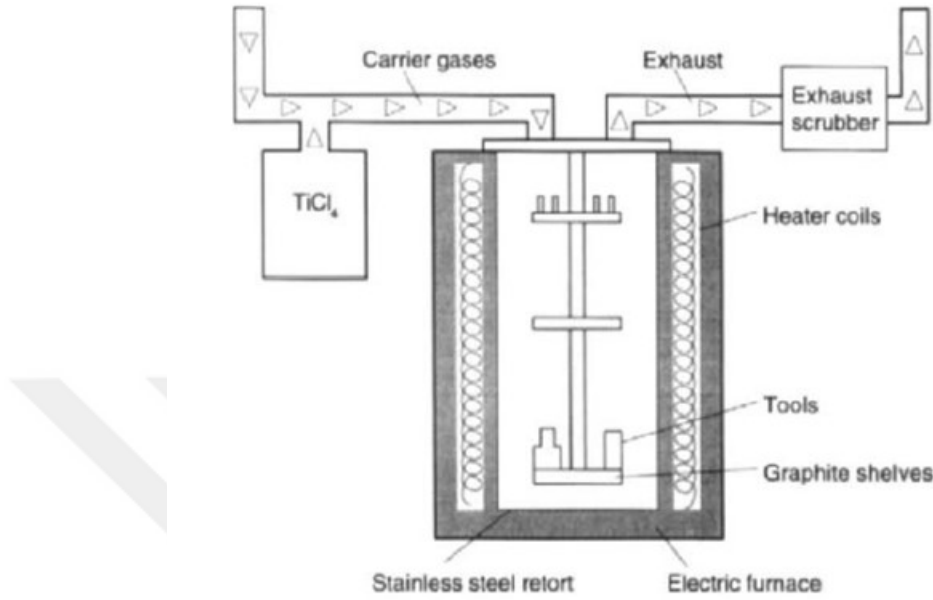


Figure 9: Typical cutting tool CVD process layout [33]

In physical vapor deposition, a solid source is evaporated under vacuum or converted to atomic form and deposited on the base material. PVD techniques have developed and become widespread in high power electricity and electronics in parallel with the development of vacuum technologies at a sufficient and economical level to be used in PVD systems. With the development of plasma-assisted PVD techniques, cleaning of the parts to be coated with a splashing mechanism during heating, diffusion of the materials to be coated on the surface to be coated at low temperatures, a more dense coating structure and mechanical properties developed accordingly have been achieved.

Due to the fact that the parts do not need to be heated with an additional heater, and high deposition rates can be provided, it has enabled the production of high hardness and density ceramic coatings, which are very suitable for wear and friction applications, adhere very well to the surface. Ceramic, hard, wear-resistant

nitrides (TiN, CrN, ZrN, TiAlN etc.), oxides, borides, carbides and diamond-like coatings can be applied using PVD techniques [34].

Plasma spray coating method is a very common method to protect the base material against wear, corrosion and thermal effects. Plasma is a condensed gas with equal numbers of electrons and positive ions, often referred to as the 4th state of matter. Therefore, the term plasma is used to describe the gaseous state that has reached a higher energy level than the known gaseous state. By keeping the base metal temperatures low during the plasma coating process, the risk of thermal deformation of sensitive parts can be reduced.

There are many parameters that affect the plasma coating process. Some of these parameters depend on the plasma spray gun and some on the coating powder. The function of the plasma gas is to accelerate the sprayed material, carry it to the coating surface and protect the hot surface from the chemical effects of the atmosphere by surrounding the particles. The gases used in the process are nitrogen, argon, hydrogen, and helium. Because of its high enthalpy, nitrogen gas allows to produce denser coatings. Since compounds such as TiC and MoS₂ are affected by nitrogen, argon is used in these applications. The high process temperature of the plasma spraying method allows working with metals and alloys with high melting points, enabling the production of functional surface coating layers in an almost unlimited spectrum. It is possible to coat with this technique for a wide range of materials such as metals, hard metals, composites, oxide and non-oxide-based ceramics, etc. Different materials and different sized powders used in the coating are adjusted according to their chemical composition, particle size and usage areas. Thanks to the developments in the powder feeding system, it is possible to mix more than one powder homogeneously and send it to the system. The plasma spray method is also used to repair surfaces damaged by wear, thermal effects or corrosion [35].

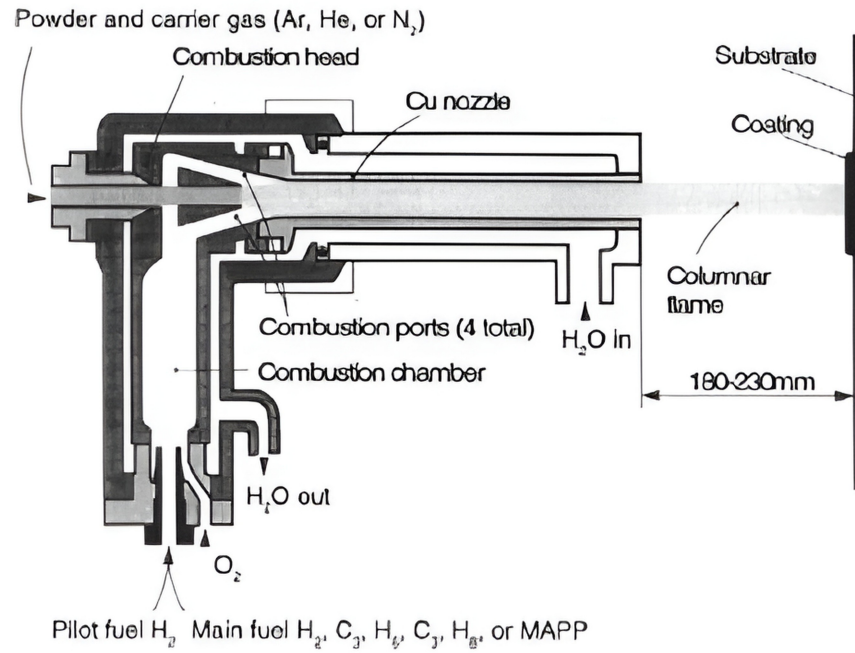


Figure 10: Typical high velocity oxy-fuel process layout [33]

Plasma spraying method is generally used as a thermal barrier coating on turbine blades, rocket body shell, combustion chamber, propeller platforms exposed to high temperature in aircraft and aerospace industry, against erosive wear in diesel engines and aircraft engines [35].

2.4 Wear

Friction and wear is a complex and microscopic interaction that occurs when one surface touches another surface, resulting from the mechanical action of the surfaces and their movement on each other. The geometric and topographic characteristics of the material surfaces formed as a result of this interaction vary depending on many factors such as the forces acting on both surfaces, temperature, shear rate, conditions of the surfaces (wet, dry) and atmospheric conditions [36]. Abrasion phenomenon is the name given to exfoliation that starts from the top layer of the material by a mechanical effect caused by friction. Friction is the mechanical interaction of two surfaces with each other [23]. Wear is a type of damage

that is usually known beforehand. Even if the material surfaces in contact with each other are protected by oxide films or lubricants, deterioration of the oxide layer or lubrication under mechanical loads can cause the two surfaces to come into direct contact with each other. The friction that occurs as a result of this contact causes wear that limits the life and performance of the material under operating conditions. This damage can be minimized by factors such as proper lubrication, filtration, appropriate material selection and appropriate design, but it cannot be prevented [36]. In order to better understand the concept of wear, the definition of friction and friction force should be emphasized. If the applied shear force is expressed as F and the applied load is W during the sliding that occurs when two material surfaces come into contact with each other, the direct proportional relationship between these two can be expressed as follows [37]:

$$F = \mu W \quad (1)$$

μ in the formula is expressed as the coefficient of friction. If the applied shear force is perpendicular to the surface and the applied load, the friction force is taken as one:

$$F = W \quad (2)$$

Friction can be defined as the energy distribution between sliding surfaces. The four fundamental experimental laws of friction can be explained by da Vinci and Amonton as [36]:

- The ratio between the maximum tangential force on the sliding surfaces and the normal force on the stationary surface changes as the tangent force increases.
- Friction force is proportional to shear force and normal force.
- The friction force is independent of the contact area.
- Friction force is independent of sliding speed.

The environmental issues like temperature and humidity is effected on the wear behavior of materials. Due to high temperature, wear types of the mechanism can

be changed. Wang et al. have investigated the wear behavior of Inconel 625 during the hot extrusion process with the help of the ball on disc tribometer to simulate the mold materials. In the study in which AISI 52100 and Si_3N_4 are used as counterparts, the results show that the friction coefficient generally increased with the temperature, but the specific wear rate increased at 250°C and then decreased at 500°C . The wear mechanisms of the Inconel 625 with the GCr15 is adhesive and fatigue wear at all temperatures. The tribocharacteristic of the Inconel 625 and the Si_3N_4 couple is adhesive and abrasive at room temperature but fatigue wear at 500°C [38]. Thirugnanasambantham et al. also investigated same behavior on the nickel superalloys. The results show that dominant wear mechanism of Hastelloy X and Inconel 718 are debris generation and delamination, respectively [39].

In another study, the effect of boronizing on the wear performance of Inconel 625 and austenitic stainless steel have been investigated. They reported that resistance of oxidation of boronized coating is limited to 800°C and the friction coefficient is 0.2-0.4 under 500g load at the room temperature according to ASTM G99 pin-on disc test [27].

2.4.1 Adhesive Wear

Adhesive wear, which is called adhesion wear, occurs as a result of the breaking of asperities on the fused (or adhered) surface during the relative movement of one metal surface on another metal surface.

In practice, adhesive wear occurs mainly due to sliding friction between metals, and wear parts are detached from the soft metal. If two metals are of the same hardness, wear occurs on both surfaces. Adhesive wear can be reduced by perfect lubrication between metals, preventing overheating of the resulting tribosystem, brightening the surfaces of the contacting materials, reducing the load acting on the surface and increasing the hardness of the material [36].

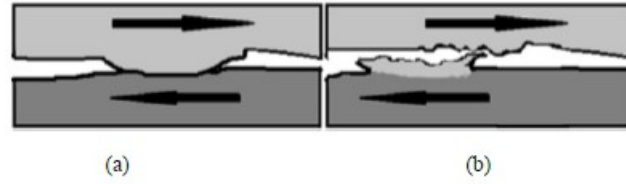


Figure 11: Adhesive wear; (a) surfaces sticking together before they begin to slide
(b) Material loss as a result of plastic deformation after adhesion on the surface after sliding [40]

Although adhesive wear is one of the most common types of wear, it usually does not accelerate damage [36].

2.4.2 Scuffing Wear

According to ASTM standards, scuffing is a type of wear that occurs in tribosystems with insufficient lubrication and causes macro-level changes in the structure of the material depending on the direction of movement. Any problem with lubrication increases scuffing wear on the cylinders. However, it has been observed that the use of a large number of cylinders also causes scuffing problems. Despite many studies on the scuffing mechanism, there is no definite consensus among scientists as to why this mechanism occurs. The factors leading to the scuffing mechanism can be listed as follows:

- Lack of lubrication
- Damage accumulation
- Plastic fatigue
- Adiabatic shear instability [41]

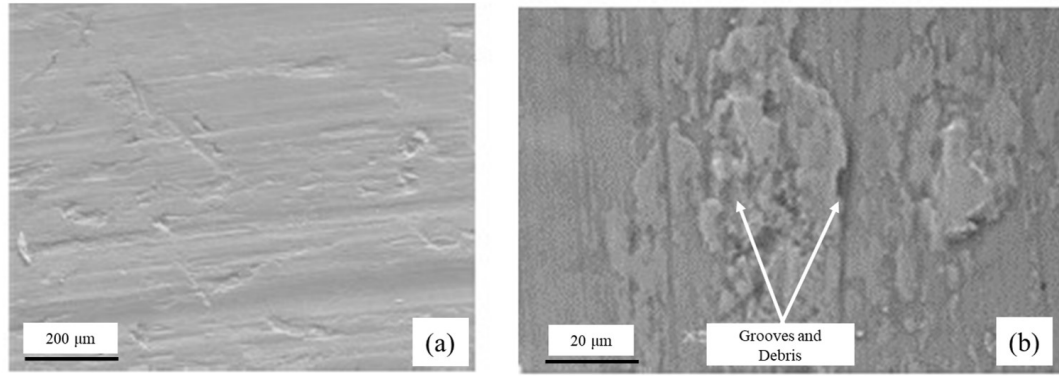


Figure 12: Scuffing wear examples (a) engine cylinder (b) engine piston ring [42, 43]

2.4.3 Abrasive Wear

It occurs when one surface is harder than the other surface with which it is in contact, or where there are hard particles in the contact area. When the surface roughness of the hard surface is pressed against the soft surface, plastic flow occurs in the soft material. If the hard surface is moved tangentially, plunging occurs and grooves and scratches occur on the surface of the soft material. It removes material from the surface depending on the geometry of the hard surface and the degree of penetration. There are two basic types of abrasive wear: two-body and three-body [44].

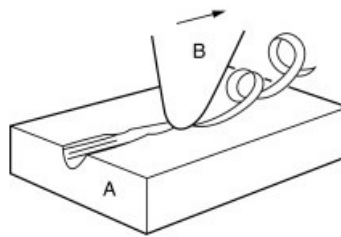


Figure 13: Abrasive Wear A: work piece, B: cutting tool [45]

Two-element abrasive wear occurs when one surface is much harder than the other. This wear occurs in cutting and grinding operations. Three-element abrasive wear occurs when one or both surfaces are eroded by a hard mass (abrasive

sand, dust, mineral grains, microchips, or fragmented oxide layers, etc.) that enters between two surfaces and is released from both surfaces. Hydrodynamic or elasto-hydrodynamic lubricants can be used to reduce friction on surfaces [46].

The abrasive wear rate can be reduced by reducing the load acting on the material surface. Thus, it is ensured that the particles sink less into the surface and leave less traces during deburring.

In terms of materials, recommendations for reducing abrasive wear are:

- Using harder alloy,
- Applying heat treatment to increase hardness,
- Covering the material surface with a hard layer [46].

2.4.4 Erosion Wear

Wear caused by the impact of solid or liquid particles against the surface of an object is called erosion wear. Erosion wear occurs in a wide range, such as damage to gas turbine blades, abrasion by dust cloud during aircraft flight, wear of pump compressor wheel [47].

Erosion wear can occur in several different ways:

- The particles in the fluid medium hit the solid surface at high speeds,
- Liquid droplets in motion hitting the solid surface at high speeds and destroying them with shock waves,
- The repetitive collision of the solid in high speed motion with the water drops slower than itself,
- Gas bubbles hitting the material surface with high pressure [13].



Figure 14: Gas turbine engine blade that failed by erosion fatigue [48]

2.4.5 Fatigue Wear

Fatigue occurs in machine parts over time due to repeated loads or vibration. In particular, the continuous and variable loads of surfaces cause fatigue cracks to form just below the surface. These microcracks move towards the surface over time, causing the pieces to break off from the surface of the material. The wear that occurs in this way is called “fatigue wear”. Fatigue wear occurs on rolling bearings, gear wheels, and surfaces such as cam mechanisms [49]. Unlike volumetric fatigue, friction fatigue is limited only to the surface and sub-surface regions. The loss of material from solid surfaces due to friction fatigue is referred to as fatigue wear. It is known that fatigue cracks start at points where tensile strain occurs or where maximum tangential stress is present. Theoretical and experimental studies show that the position of the maximum tangential stress under contact loading depends on the coefficient of friction. With a low coefficient of friction, the point of maximum shear stress is located below the surface ($\mu < 0.3$). When the friction coefficient increases, the point of maximum shear strength appears on the surface ($\mu > 0.3$). On the other hand, if a body is subjected to vertical and tangential loading, stress strain appears in the surface and sub-surface regions. Therefore, cracks nucleate on and/or above the surface [49].

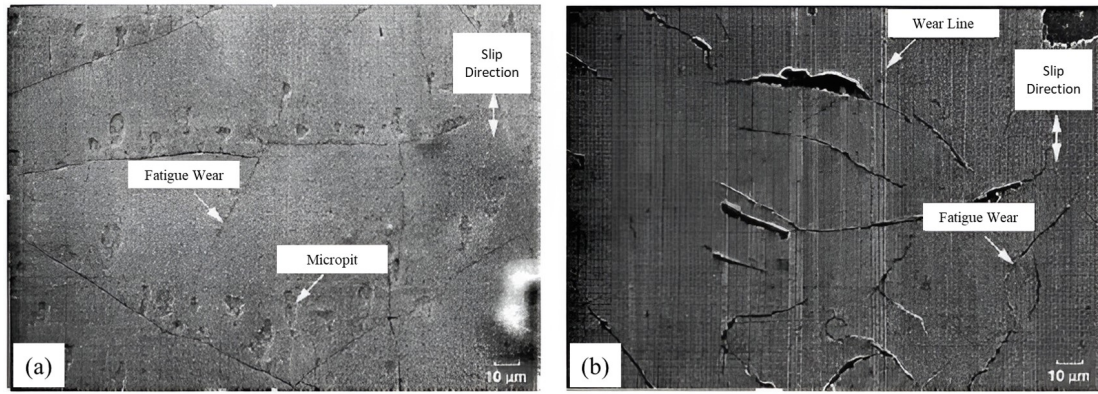


Figure 15: Fatigue Wear examples for internal combustion engines (a) piston side (b) cylinder side [50]

2.4.6 Corrosion Wear

On the wear damaged surfaces, in addition to this, the loss of material caused by chemical reactions on worn surfaces is called corrosive wear. Chemical wear can occur alone or in combination with other types of wear. As a result of damage to the films (layers) used to protect the surfaces, elements such as acids and oxygen, which have high chemical affinity with the material structure, react and form compounds on the surface. Corrosive wear often causes billions of dollars in damage to metal-using industries due to the damage it causes to the material structure [51]. Chemical corrosion occurs in a chemical and corrosive environment where slip occurs. The most dominant corrosive medium is oxygen. Therefore, chemical corrosion in air is usually oxidative. In the first place, corrosion creates a thin film on the surface and reduces material loss. However, after this, a lot of material is lost with shear stresses applied to the surface [52].

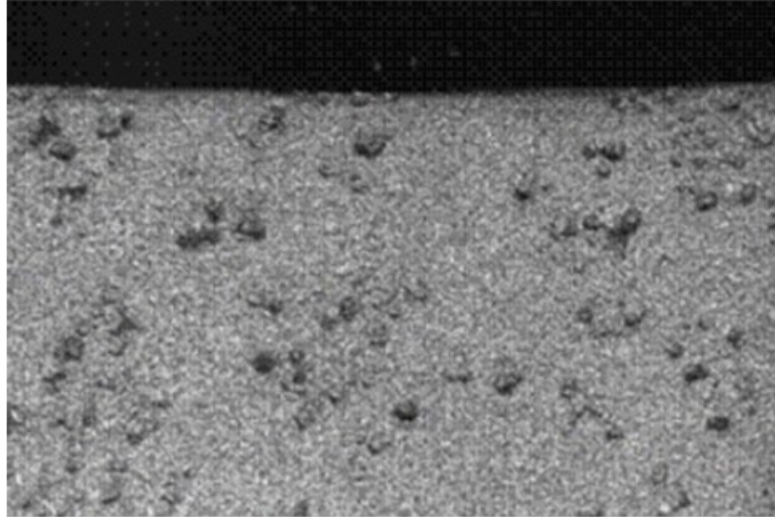


Figure 16: Pitting corrosion on gas turbine blade [52]

The main features of corrosive wear are there is no crack in the base material, fracture appears in the thin surface layers produced during friction. As a result of the dynamic balance of mechanical and chemical processes, oxide films are formed and then broken [52].

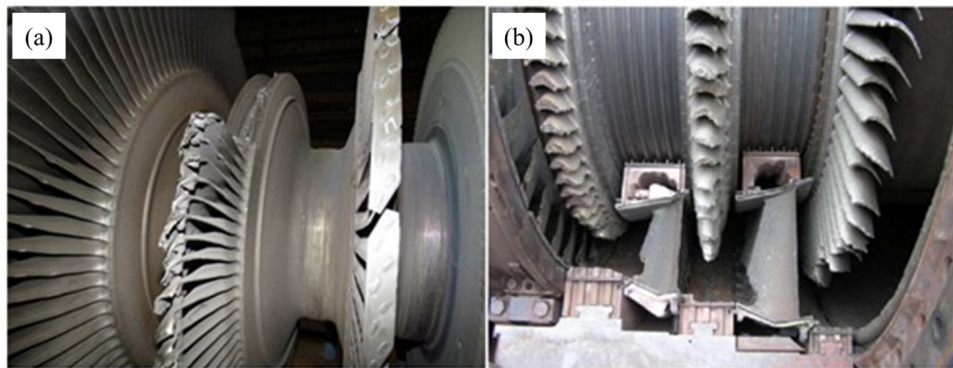


Figure 17: Examples of corrosion wear failures (a) deformation of turbine blades (b) deformation of shrouds [52]

2.4.7 Diffusion Wear

The temperature increase caused by friction between surfaces in contact with each other causes diffusion in the crystal lattice, moving from regions with high atomic density to regions with low atomic density. During friction, the contact

of the surfaces is ensured by the applied force, and with the stress concentrations on the material surfaces and the increase in temperature, yielding and creeping begin at the contact points. As a result, the contact points are subjected to plastic deformation, increasing the contact area. Thus, an exchange of atoms occurs between the surfaces and a surface layer with low strength is formed. This layer causes material loss by breaking off from the main material surface with the continuation of friction and relative movement. The wear that occurs in this way is called “diffusion wear”. This wear mechanism is seen in brake pads, spacecraft, and machine tools [53].

2.4.8 Fretting Wear

The term fretting is expressed as the small oscillating motion between two solid surfaces in contact. The amount of this oscillation is between 1 and 100 μm . The type of wear that occurs as a result of this movement is called fretting wear.

Fretting wear occurs between machine components. This wear usually occurs with the operation of the mechanism that is subjected to repeated stress or vibrating in fixed contact.

In order to reduce fretting wear, methods such as reducing the amount of micro-displacement, increasing the abrasion resistance of the contact pair, increasing the corrosion resistance against ambient conditions, using special anti-vibration devices can be used [54].

2.4.9 Bore Polishing Wear

In internal combustion engines, shines may occur in the upper part of the cylinder surface, which is under the effect of the fire zone. This is due to hard residues caused by uneven combustion, the use of poor quality oil, low combustion temperatures due to frequent idling or partial load operation of the engine. The oil carbon layer causes wear on the cylinder wall, damage to the oil film, friction, high ring wear and as a result, high oil consumption. For prevention;

- Operation of the engine as described in the instructions

- Use of the prescribed oil quality
- Use of quality fuel
- The injection system must be maintained, checked and adjusted in accordance with the instructions [55].

2.4.10 Delamination Wear

The stresses created by the load exerted by an object on another object under the surface rise above the tensile strength of that object and form cracks under the surface. When these cracks progress under the surface and reach the surface, the particles on the surface are released in the appearance of shells. This wear type is known as "delamination" (layer separation) [56].

2.5 Hertz Contact Theory

The Hertz stress theory is used to calculate the stress distribution for two curved solid bodies in contact. Heinrich Hertz was the first to notice it in 1882 [57]. The localized tension caused by two curved bodies touching one other is known as Hertzian contact stress. Hertz was the first to contribute to the solution of elastic deformation for contacting bodies, and many other researchers followed.

According to Hertzian Contact Theory, the bodies are in contact, should be isotropic and homogeneous. The friction between two bodies should be negligible and deformations could be calculated linear elastic theory. Nevertheless, in a lot of examples, at least one of the contact couples subject to plastic deformation. Because of the difficulty of the theoretical calculations of elastoplastic stress due to the plastic deformation, engineers utilize the Hertzian mathematical model for theoretical and numerical finite element models for stress calculations [58, 59].

The contact is between spherical and flat bodies for ball-on disc wear tribometer configuration. To determine the stress values on the surfaces for ball-on disc configuration, a group of equations are used given below [60].

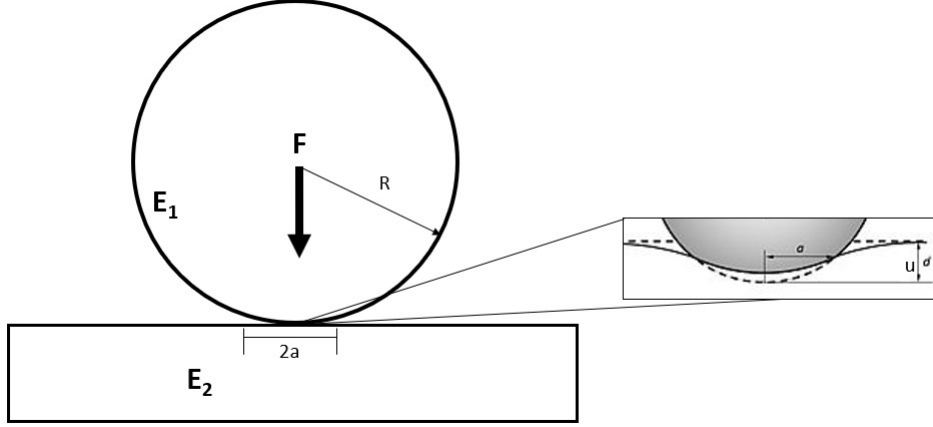


Figure 18: Sphere on disk hertz contact

As showed in Figure 18, a contact area of radius "a" is created when an elastic sphere of radius R indents an elastic half-space to depth u.

$$a = \left(\frac{3RF}{2E_*} \right)^{\frac{1}{3}} \quad (3)$$

The indentation depth is given by :

$$u = \left(\frac{2F^2}{E_*^2 R} \right)^{\frac{1}{3}} \quad (4)$$

where,

$$\frac{1}{E_*} = \frac{1}{2} \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) \quad (5)$$

In equation 5, E_1 and E_2 are the Elastic modulus of the contact couples, ν_1 and ν_2 are the Poisson ratios.

According to Hertz Contact Theory, for elastic materials, Stiffness is;

$$k = \frac{dF}{du} = \left(E_*^2 R F \right)^{\frac{1}{3}} \quad (6)$$

The compressing and shear stresses are also important for the design engineers to select the proper materials. Crush (σ_c) and shear (τ) stresses can be calculated

from equation 7 using Hertzian Contact Theory to give an idea at the conceptual design stage of the structural parts.

$$\sigma_c = \frac{3}{2} \frac{F}{\pi a^2} = 0.4 \left(\frac{E_*^2 F}{R^2} \right)^{\frac{1}{3}} = 0.4 \frac{k}{R} \quad (7)$$

$$\tau_{\max} = \frac{\sigma_c}{3} \quad (8)$$



CHAPTER III

MATERIALS AND EXPERIMENTAL METHODS

In the current thesis, two different materials including AISI 321 and Inconel 625 have been used. After preparation process the samples were coated with two different techniques of boronizing (via diffusion method) and tungsten carbide coating (via HVOF method). The box boronizing was conducted at the temperature of 1100°C for 8 hours. Tungsten carbide powder material was deposited using HVOF gun on to AISI 321 and Inconel 625 substrates. The raw materials for both samples were rod shape with diameter of 16 mm, and all the test samples were cut by using electrical discharge machine (EDM). The chemical compositions of the as-received materials are given in Table 1 and 2, respectively.

Table 1: Chemical composition of AISI 321

Element	C	Si	Mn	P	S	Cr	Mo	Ni
Mass%	< 0.12	< 1.0	< 2.0	0.045	0.03	17.0	–	9.0

Table 2: Chemical composition of Inconel 625

Element	Al	Cr	Fe	Mo	Nb	Si	Ti	Ni
Mass%	0.21	22.27	4.43	8.90	3.50	0.16	0.27	60.0

Figure 19 demonstrates the diffusion depth of boron layer through the samples. The Marble's and Kalling's etching solutions were employed for AISI 321 and Inconel 625, respectively. The diffusion depth were determined using Future Tech FM 300e optical microscope. Results revealed that the boron particles diffused 45-60 μm in to the AISI 321 sample while the thickness of the boron layer was about 60-75 μm for the Inconel 625 sample. The core microhardness of AISI 321 sample was 235 HV with an standard deviation of 15 HV while the microhardness of the

surface was increased up to about 2250 HV with 40 HV standard deviation. The core and surface microhardness of Inconel 625 sample after boronizing was about 345 HV and 2150 HV with standard deviation of 10 HV and 40 HV, respectively.

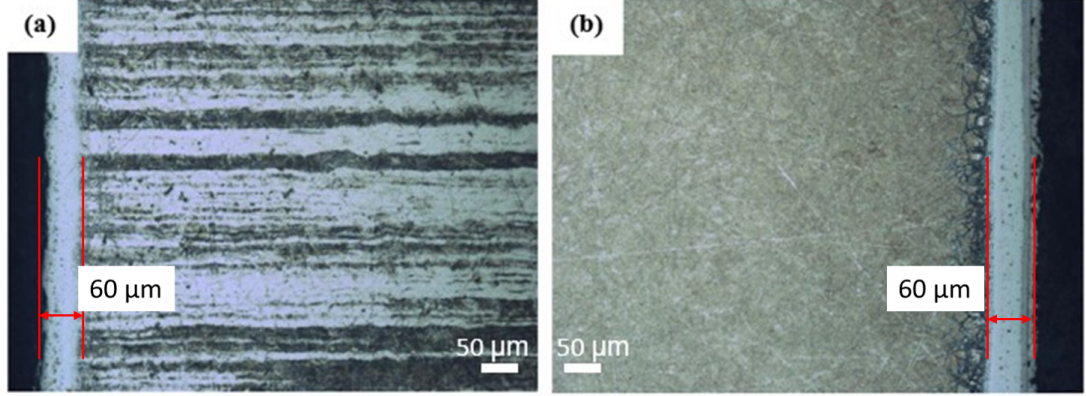


Figure 19: Thickness of the coated surface for (a) AISI 321 and (b) Inconel 625.

3.1 Sample Preparation

As-received bars were sliced to 2 mm sheets using wire cut electrical discharge machine (EDM). Dog bone shape tensile samples were cut from the prepared sheets and subsequently ground and polished to eliminate the residual stresses. ASTM-E8 standard was used for the tensile samples and the gage length of the samples for room and high temperature tests were 15 mm and 36 mm, respectively.

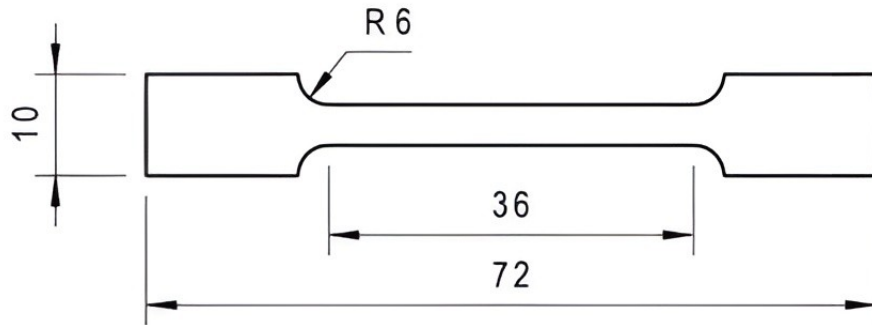


Figure 20: Technical design of high temperature tensile samples (all dimensions are in mm)

3.2 Tensile and Hardness Tests

The tensile tests were performed in a quasi-static situation with a strain rate of 10^{-3} s^{-1} on an Instron mechanical test platform. For each condition, at least three samples were examined, and the standard deviations of the results were less than 3%. The samples were heated to 600°C using an induction coil system wrapped around the gage length. Figure 20 shows the sample geometry for room and high temperature tensile testing. Vickers microhardness of the samples after coating was also examined under a load of 300 gf with a dwell duration of 15 s from both the core and surface of the samples.

3.3 Wear Tests

ASTM G99 Ball on disc wear testing method were used for all samples at room temperature and high temperature of 600°C. 16 mm disk shape samples with thickness of 4 mm of different uncoated and coated conditions were utilized to examine the wear performance. 3 test have been done at each condition and distance. The applied load on the 6 mm diameter alumina ball was 4 kg and sliding speed was set to 320 RPM. Also, the wear track diameter was selected as 8.5 mm for all testing conditions. Moreover, electrical resistance furnace was utilized to heat up the setup for the high temperature tests. It is worth noting that for all high temperature tests, the setup heated up with heating rate of 100°C/min, and 10 minutes dwell time conducted to stabilize the samples and setup before starting the tests.

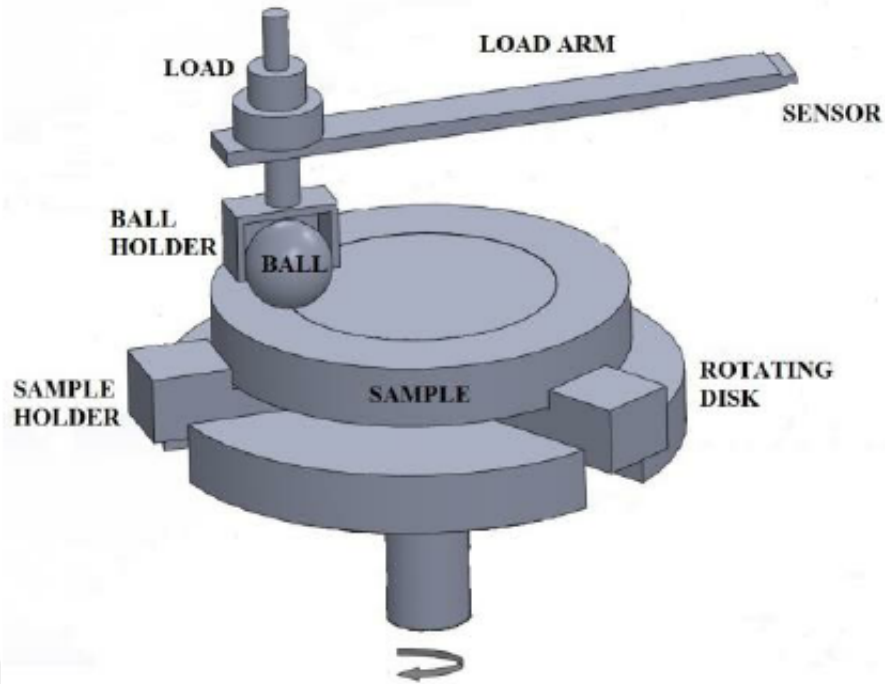


Figure 21: Schematic of ball on disc wear test setup [61]

3.4 Surface and Fracture Analysis

Wear amount measurements given below is done under:

- Weight losses of samples,
- Measuring the weight of wear products,
- Measuring the dimensional change of the samples.

Measurements made on the basis of weight loss cannot give detailed information about the changes that occurred during the test. A large number of experiments are required to see the time-dependent course of the event in detail [62].

Measurements based on length reduction often do not give clear information about which element, to what extent, whether the wear is straight or curved. Therefore, volume changes are mostly used in measurements based on dimensional change.

As a result of all these evaluations; The measurement of wear, which is generally defined as material loss, can be made on the basis of the volume or weight

loss in one or both of the contacting parts. Wear can be given by direct or indirect measurements. Depending on the test system used in the wear test, linear, planar or volumetric wear is in question [36].

Wear is most commonly evaluated by the wear rate (dw/dt).

$$W = \frac{dW}{dt} = \frac{\Delta m}{sA\rho} \quad (9)$$

where;

Δm = mass loss (gr)

A = contact area (mm^2)

s = sliding distance (mm)

ρ = density of the worn material (gr/mm^3)

On the other hand, the amount of volume (ΔV) loss through wear in a material:

$$\Delta V = kW_s(\text{mm}^3) \quad (10)$$

Where;

ΔV = Volume Loss (mm^3)

k = Wear coefficient

W = Mass (kg)

s = sliding distance (mm)

In wear tests, the measurement of wear is made by weight difference, thickness difference, trace change and radioisotope method [63].

Volume loss method, is the most used method because it is economical and the measurand is within the instrument sensitivity capability. Measuring the weight loss is done with the aid of a highly sensitive balance with an accuracy of 10^{-4} g.

When the weight loss is wanted to be expressed as the volumetric wear amount, it can also be found by going from the volume loss corresponding to the unit path and unit loading weight, taking into account the density of the material used and the loading weight acting on the test sample, again based on the weight loss.

$$W_a = \frac{\Delta m}{Ms\rho} \quad (11)$$

where;

W_a is wear rate (mm^3/Nm), Δm is mass loss (gr), M is loaded force (N), s is sliding distance (m), and ρ is density of the material (gr/cm^3).

The inverse of the Wear rate (W_a) is represented as the wear resistance (W_r).

$$W_r = \frac{1}{W_a} (\text{Nm}/\text{mm}^3) \quad (12)$$

A Zeiss Smartproof 5 3D scanning confocal microscope was used to inspect the volume loss of the wear track on the surface of each sample. Zeiss Smartzoom 5 is also used to investigate the wear track and surface profile of the uncoated and coated samples. Fracture surface observations and energy dispersive spectroscopy (EDS) were done by scanning electron microscopy (SEM) inside Zeiss Ultra Plus and ThermoFisher Scientific Quattro S devices, operating at an accelerating voltage of 5 kV under ultra vacuum.

Wear resistance of materials are compared by wear rates. Wear rate is volume or mass loss per unit distance. That means, the wear resistance of the material with a low wear rate is higher than others. Mass loss generally measured by scientific precision balance. In most of example, it can be correlated to volume loss and convertible directly. But some of the materials or surface properties is not available for mass volume loss because of their crushable structure. In this case, volume loss measurement gives more effective results for the wear rate calculations.

The most traditional method to measure the volume loss of the worn surfaces is using profilometer. After pin-on disc or ball-on disc wear test, surface roughness profilometer measures the one part of the wear scar, and whole volume loss can be derived with the known radius formulation as shown in Figure 22.

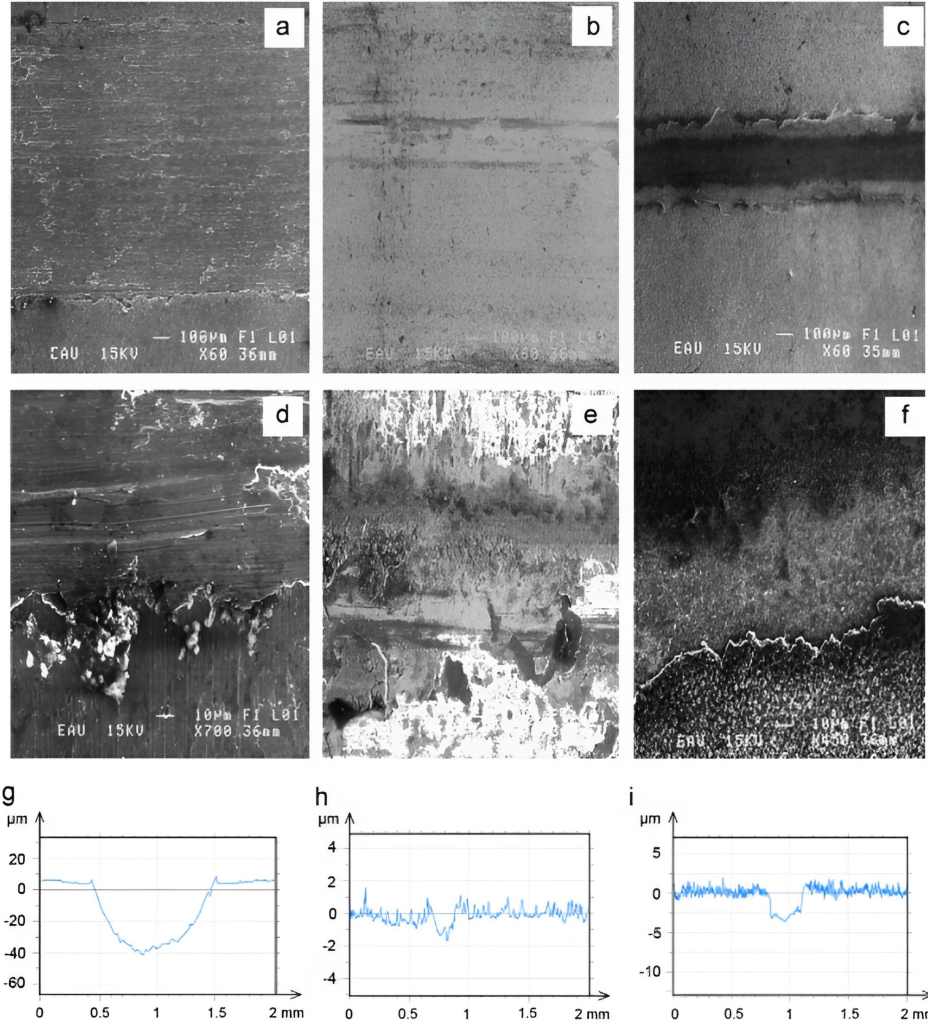


Figure 22: SEM and section wear track of the untreated (a,d), nitrided (b,e), duplex treated (c,f) samples and their section profiles of wear track (g,h,i) [64]

In the last few years, optical profilometry is substitute for stylus profilometry with the giant developments on the optic and image processing technology. The precision of the optical profilometer results are very sensitive, even better than stylus profilometry, anymore. With the help of the high resolution cameras and developed image processing applications, tribologists can observe the whole or a part of worn surfaces, and taken volume loss directly.

In this work, uncoated, boronized and WC coated AISI 321 and Inconel 625 samples were tested at ball-on disc tribometer according to ASTM G99 standard. All of the samples' weight measured with scientific precision scale before and

after test intended for mass loss calculation. For uncoated samples, mass loss measurement is suitable for calculation of wear rate, because it is known that rupture materials' density is same with base material. However, the density of the wear debris of coated samples are variable because it is include different materials in their surface. Therefore, for wear rate calculation of coated samples, volume loss method is more suitable.

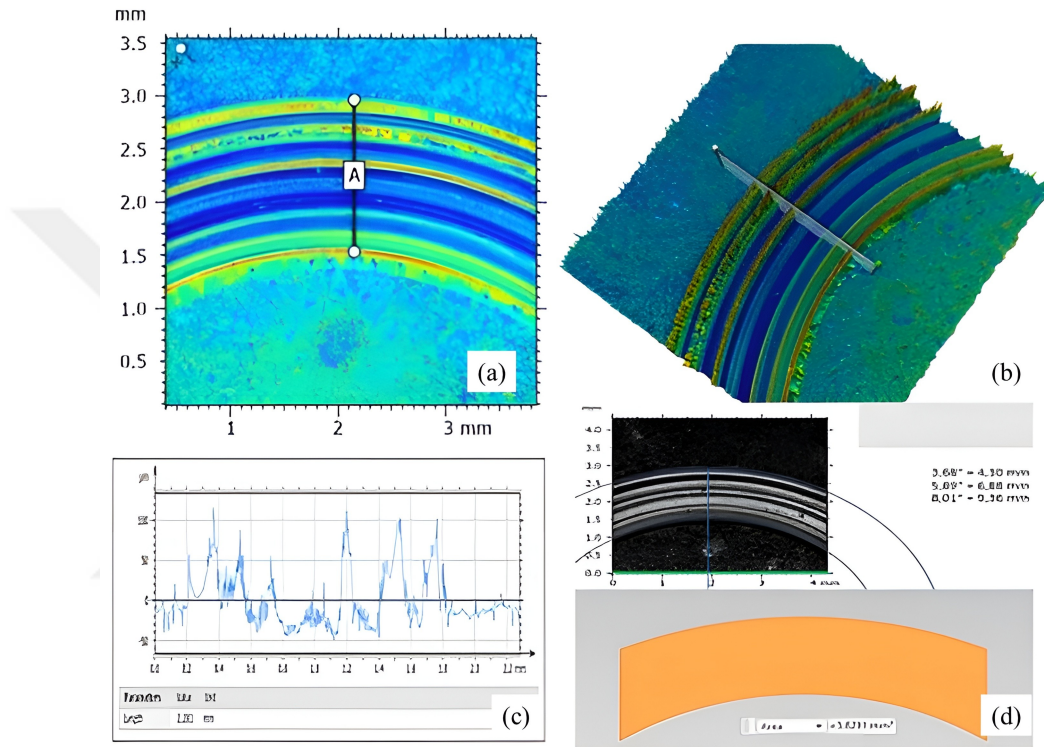


Figure 23: Volume loss calculation methodology (a) Wear track measurement (b) Wear scar volume measurement (c) Wear scar optical profilometry (d) Whole area calculation with image processing application and CAD model validation

For this reason, wear tracks are investigated by Zeiss Smartproof 5 3D confocal microscope from the 3,5x3,5 mm area of wear samples. The volume loss was measured by Digisurf Mountains® analysis software and it is expanded to the whole sample by Fiji image processing application from the calibrated view. As shown in Figure 23, to crosscheck the area calculation, the area of interest was modelled with the Siemens NX and calculated the whole area again. The volume

loss results were multiplied by the density of the materials to obtain the mass loss, compared with the measured weight losses from precision scale for uncoated samples, and it is calculated that the difference between two mass loss calculation was 7%. It was shown that, the volume measuring method is suitable for the wear rate calculations.



CHAPTER IV

RESULTS AND DISCUSSION

This chapter of the thesis is divided into four main sections. In the first and second sections the tensile behavior and wear performance of AISI 321 stainless steel are discussed in detail. The third and fourth sections include the tensile and wear behavior of Inconel 625 superalloy at different coating conditions and testing temperatures. Besides, all the tested samples were inspected by SEM and 3D optical microscope to investigate the fracture and wear mechanisms, respectively.

4.1 Tensile Behavior of AISI 321

4.1.1 Mechanical Properties

The Vickers microhardness test was applied on the different conditions of as-received AISI 321, boronized, and WC coated samples and the results is demonstrated in Figure 24. The hardness of as-received sample was about 182 HV with a standard deviation of 4 HV, while the mean microhardness of WC coated sample was 2423 HV with a standard deviation lower than 5%. Also, the core microhardness of boronized sample was 235 HV with a standard deviation of 15 HV while the microhardness of the surface was increased up to about 2250 HV with 40 HV standard deviation.

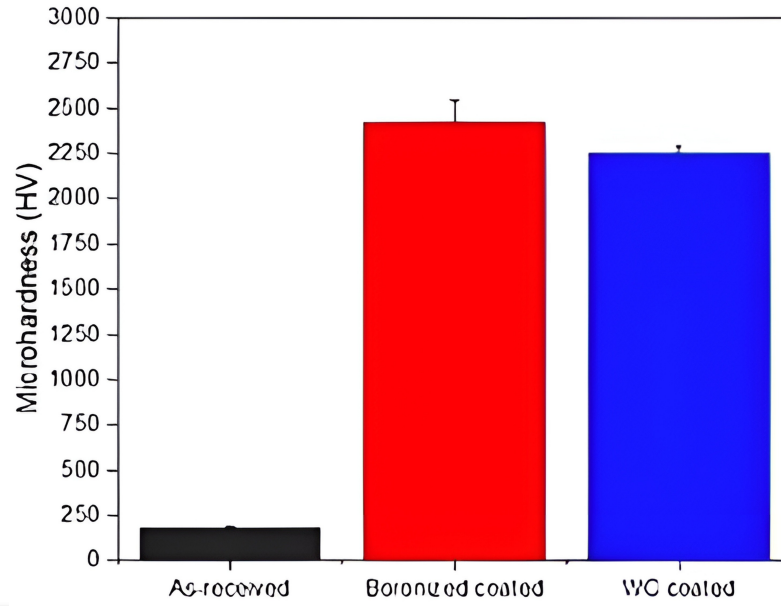


Figure 24: Hardness test results of AISI 321 stainless steel samples

Figure 25 illustrates the room and high temperature tensile stress-strain curves of the samples for different conditions. As shown in Figure 25a, the yield strength (YS) and ultimate tensile strength (UTS) of the samples after coating are reduced. Accordingly, this reduction in YS is more significant in boronized sample which is attributed to the softening of the material throughout the boronizing process at high temperature of 1100°C. Furthermore, strain at failure of the WC coated sample was lower than that of the uncoated and boronized condition, which can be related to the hardness mismatch between the WC layer and underneath AISI321 layer that can be the high potential region for crack initiation and fast fracture (see fracture morphology section). Also, high ductility of the boronized sample is related to the coating with diffusion method. Besides, the dispersed boron particles instigated the Orowan strengthening and consequently enhanced the work hardening of the sample during the plastic region of the tensile test [65, 66, 67].

As demonstrated in Figure 25b, the ductility of the AISI321 samples for all conditions has been diminished due to dynamic strain aging at 600°C [2]. It can be observed that WC coating has increased the high temperature yield strength of the AISI321, significantly (up to 150 MPa). This may be attributed to the

direct effect of high strength ceramic WC layer at 600°C. Although, the strain to failure of the WC sample was decreased due to the fast fracture of coated layer after 6% elongation at high temperature. Furthermore, the yield strength of the boronized sample was reduced to 130 MPa due to decomposition of boron particles at elevated temperature [68].

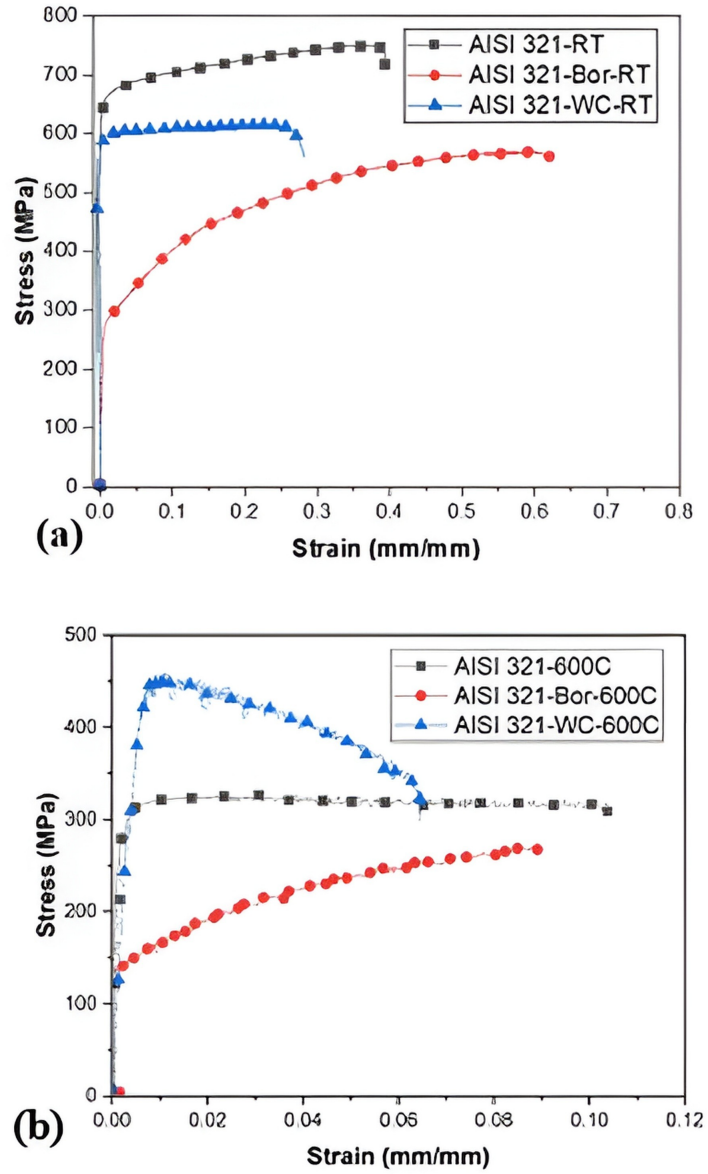


Figure 25: Tensile stress- strain curves of AISI 321 for different conditions tested at (a) room and (b) 600°C

$$E_c = E_f V_f + E_m V_m \quad (13)$$

The modulus of elasticity obtained by the tests was compared with the theoretical modulus of elasticity formula of composite materials (Equation 13). The volume ratio between materials was found by measuring the 36 mm gage length of the tensile specimens, can be seen in Figure 26. According to theoretical formula, AISI 321 - WC composite's elastic modulus was 238 GPa, when AISI 321 and WC's elastic modulus are 176 and 600 GPa, respectively. Nevertheless, it is shown that WC coated samples' elastic modulus was the test results that 206 GPa. The high temperature of the process and the bond coating's sensitivity to temperature may have led to a drop in the modulus of elasticity.

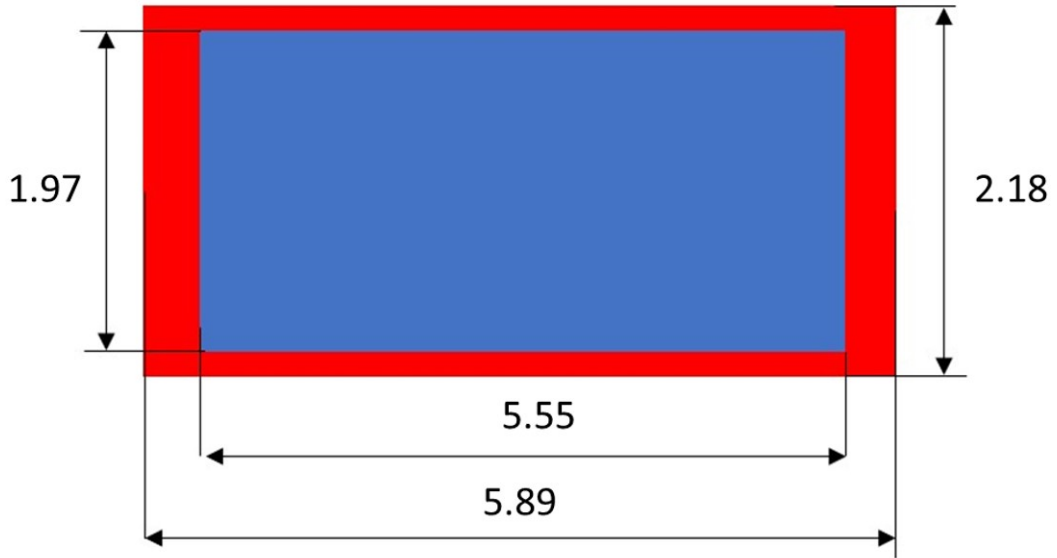


Figure 26: Gage dimensions of WC coated AISI 321 tensile samples

4.1.2 Fracture Morphologies

Figure 27 shows fracture surface of AISI 321 samples after tensile test at room temperature. As it can be seen in low and high magnification SEM images, a uniform ductile-fracture mechanism represented by fine dimples were observed on fracture surface of AISI 321 sample (Figure 27a-b.) A similar fracture mechanism was detected on the core region of boronized sample while the decomposition of

boron particles and subsequently their allocated positions represented by coarse deep dimples were observed on the outer side of the failed boronized samples (Figure 27c-d). In contrary, completely brittle fracture manner was seen in the outer surface of WC coated sample indicated by cleavage and facets while the core region of the sample was similar to the other conditions (see Figure 27e-f).

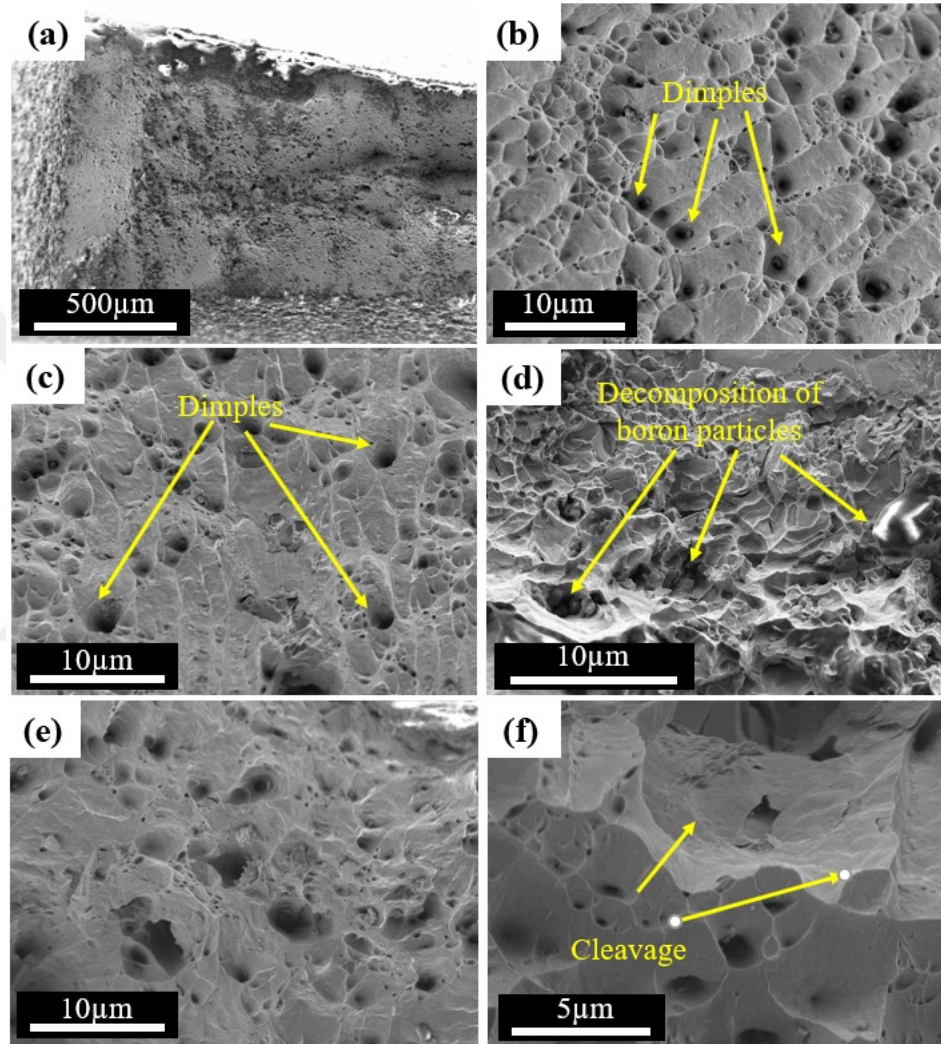


Figure 27: Room temperature fracture morphology of AISI321 for different conditions of (a, b) uncoated, (c) boronized (core region), (d) boronized (outer surface), (e) WC Coated (core region), (f) WC Coated (outer surface)

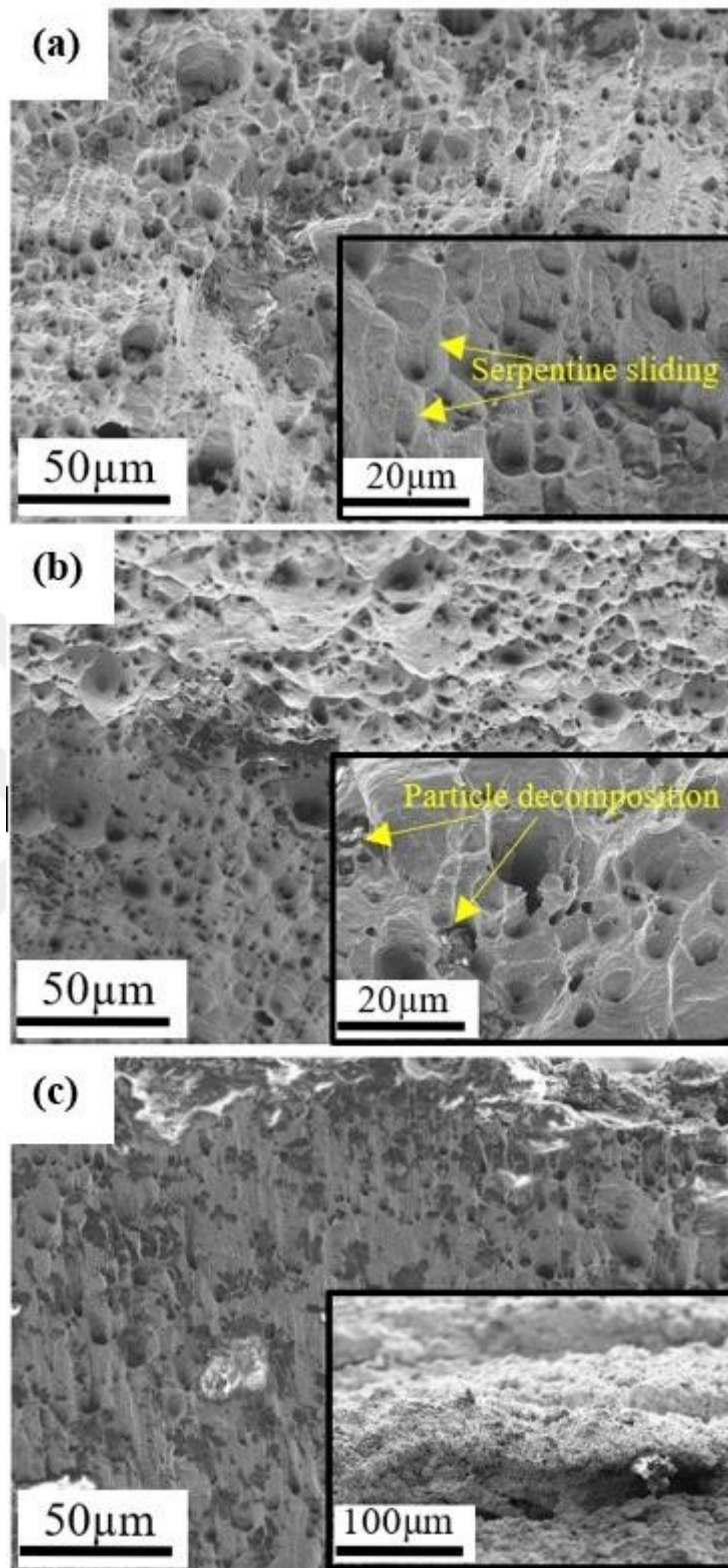


Figure 28: High temperature fracture morphology of AISI321 for different conditions of (a) uncoated, (b) boronized (c)WC coated

Prior to the fracture analysis of high temperature samples, EDX analysis obtained on the tested boronized AISI 321 sample. Accordingly, boron particle with diameter about 10 μm is identified in the sample (see Figure 29).

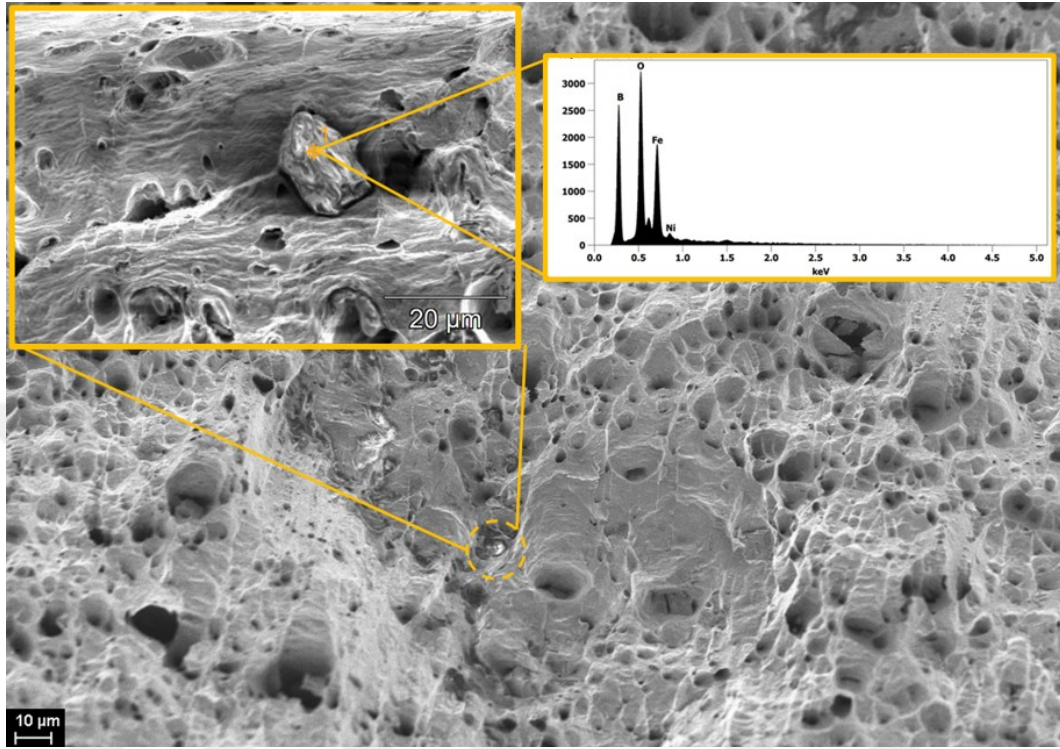


Figure 29: EDX analysis of boronized AISI 321 sample for spectrum of decomposed boron particles

Figure 28 demonstrates the fracture surface of the samples after tensile test at 600°C for all uncoated and coated conditions in different magnifications. As it can be seen in Figure 28a, relatively deep dimples with high concentration of serpentine sliding on the inner faces of the dimples were detected on the fracture surface of uncoated AISI 321. On the other hand, the high magnification SEM of the boronized sample revealed an approximately similar behavior with a noticeable boron decomposition on the fracture surface after the high temperature tensile test. This can be the main reason of the lower ductility and strength of the boronized sample at high temperature associated with the softening at high

temperature diffusion process (See Figure 28b) [69]. Furthermore, Figure 28c illustrates a typical brittle fracture behavior on the central region of WC coated sample represented by facets and cleavage features. Also, the low magnification figure, captured from the side of the sample shows the fast fracture happened on the ceramic WC layer which is the key reason of the low strain at failure of the WC coated AISI 321 [70].

4.2 Wear Behavior of AISI 321

The tangential force sensor of the tribometer was used to record the coefficient of friction (COF) during each pin-on-disk test. A frictional force is formed when two contacting surfaces move against each other in the opposite direction of sliding. The friction force is thought to be exerted in a direction perpendicular to the usual load when sliding. The coefficient of friction is equal to the ratio of these forces. The primary data for evaluating the material's friction behavior is the fluctuation in coefficient of friction with sliding distance.

The friction coefficient of the AISI 321 specimens for all various conditions weights losses with increasing sliding distance until a peak value was attained, and then it progressively approached steady state, as shown in Figure 30. This is known as the "running-in" stage. The contact of the asperities between the rubbing surfaces corresponds to the first running-in. The asperities of the surface limit the sliding speed in the beginning. During the running-in stage, friction causes some of the asperities to be removed [39].

Figure 30 represents the friction coefficient versus sliding distance of uncoated and coated AISI 321 samples at room temperatures. As it shown in figure, the highest friction coefficient peak and steady state belongs to WC coated sample at room temperature wear tests. Also, it can be seen that after 100 m sliding distance, WC coated sample has reached to steady state and friction coefficient is approximately 1, until 600 m sliding distance. In contrary, there is a significant fluctuation in friction coefficient of boronized sample until 300 m sliding distance.

This can be attributed to decomposition of boron particles from the surface during wear test. Moreover, a high peak and then steady-state friction coefficient was observed for the uncoated sample. Which may be related to the contact of asperities of the surface at the beginning of run-in stage follow-up with removing the asperities after 100 m sliding distance [39].

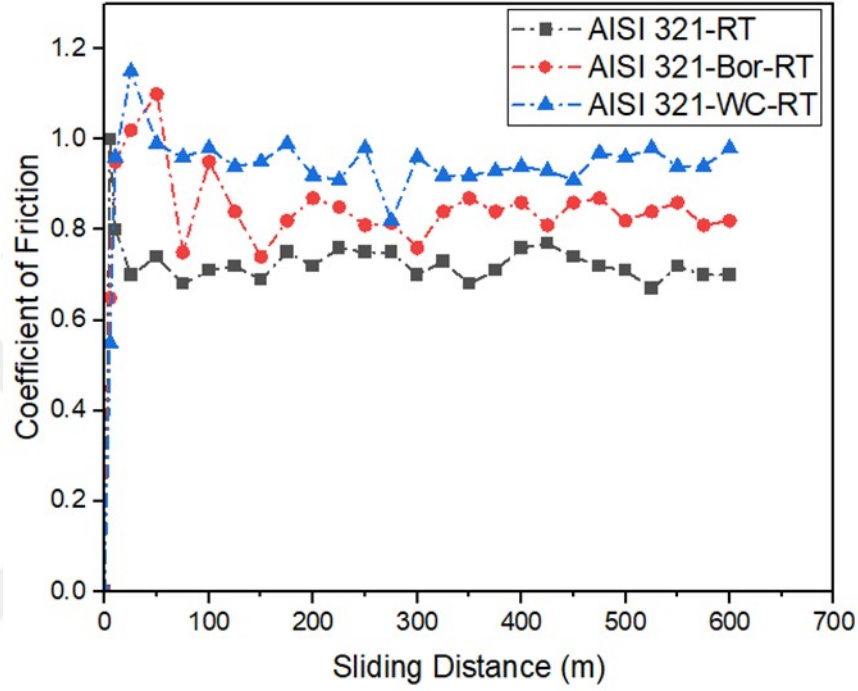


Figure 30: Friction coefficient versus sliding distance of AISI 321 at room temperature for different uncoated and coated conditions.

Figure 31 shows the wear rates of AISI 321 versus sliding distance of all conditions at room temperature. It is obvious that the wear rates of boronized and WC coated AISI 321 were extremely lower than that of uncoated sample. Furthermore, the wear rate of uncoated sample has reached to steady-state after 500 m. It is worth noting that the slightly higher wear rate of boronized sample in comparison with WC coated condition, at the beginning of the sliding is related to removing the boron particles from the surface while the steady low wear rate of WC coated sample is due to high wear and heat resistance of WC layer.

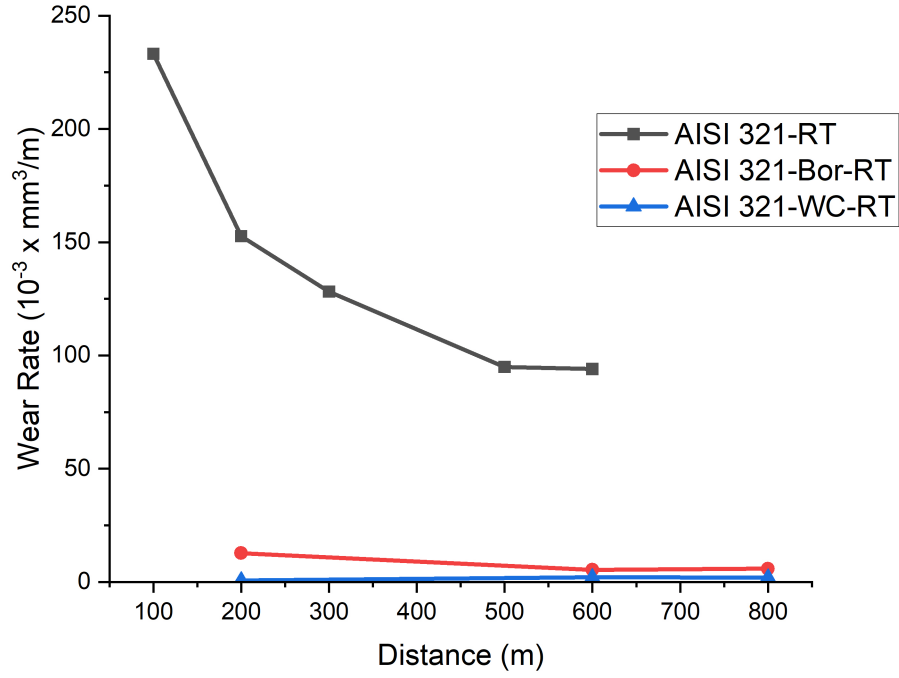


Figure 31: Wear rates of AISI 321 stainless steel samples at room temperature

Figure 32 illustrates Wear track of AISI 321 samples at different sliding distances. Also, color coded depth images on the right-hand side shows the different depth distribution on the wear track for different samples. It can be seen that the highest depth of the wear track is significantly increased from the sample tested for 100 m sliding distance to 300 m. However, the difference between highest depth of the sample tested for 600 m and 300 m is not notable. This can be attributed to steady-state wear after 300 m sliding distance. Moreover, the wear scar width is increased until 300 m, and it is also stabilized after on (Figure 32 d-f).

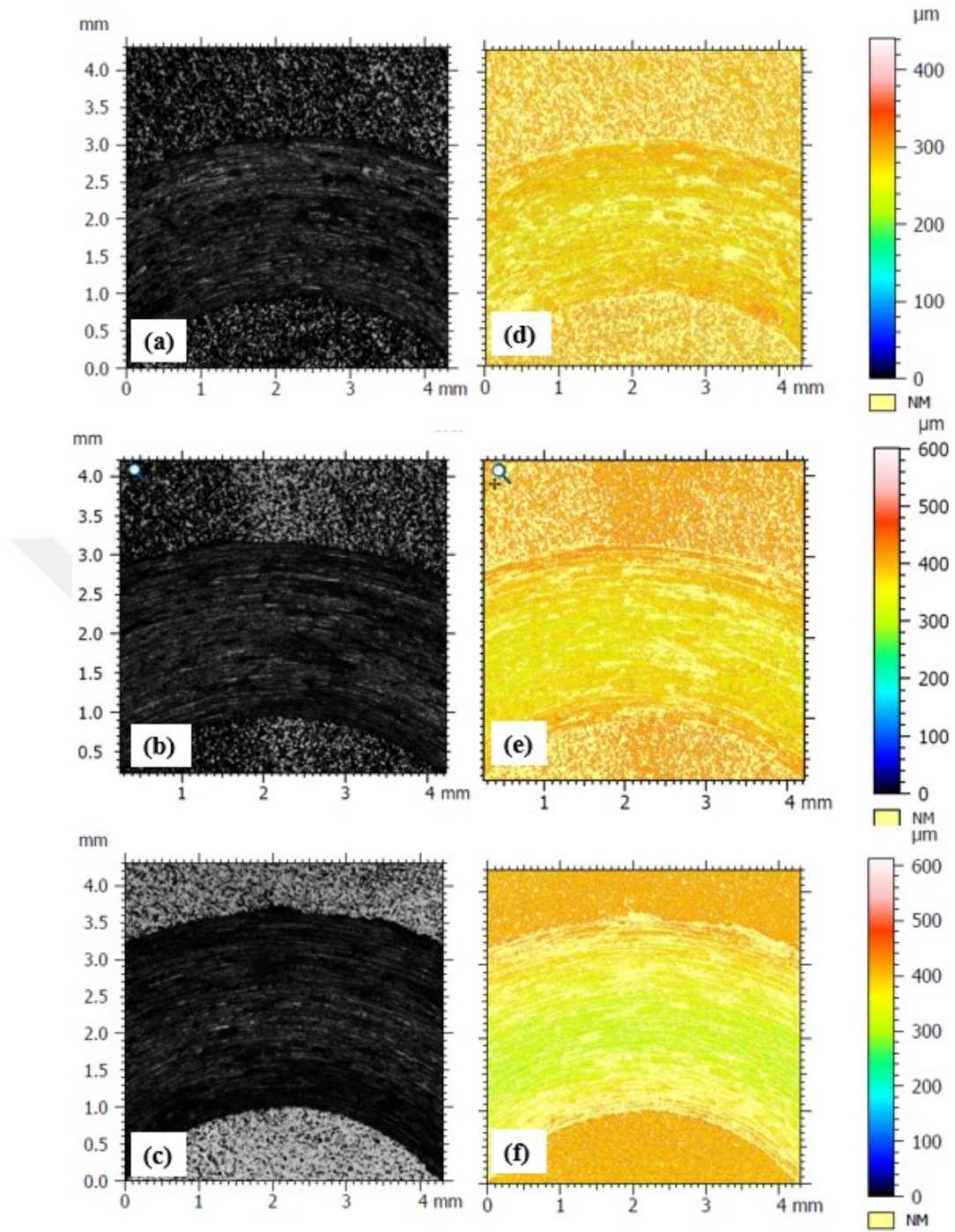


Figure 32: Wear track of AISI 321 samples at different sliding distances of (a) 100 m, (b) 300 m, and (c) 600 m with color coded depth image of (d) 100 m, (e) 300 m, and (f) 600 m tested at room temperature.

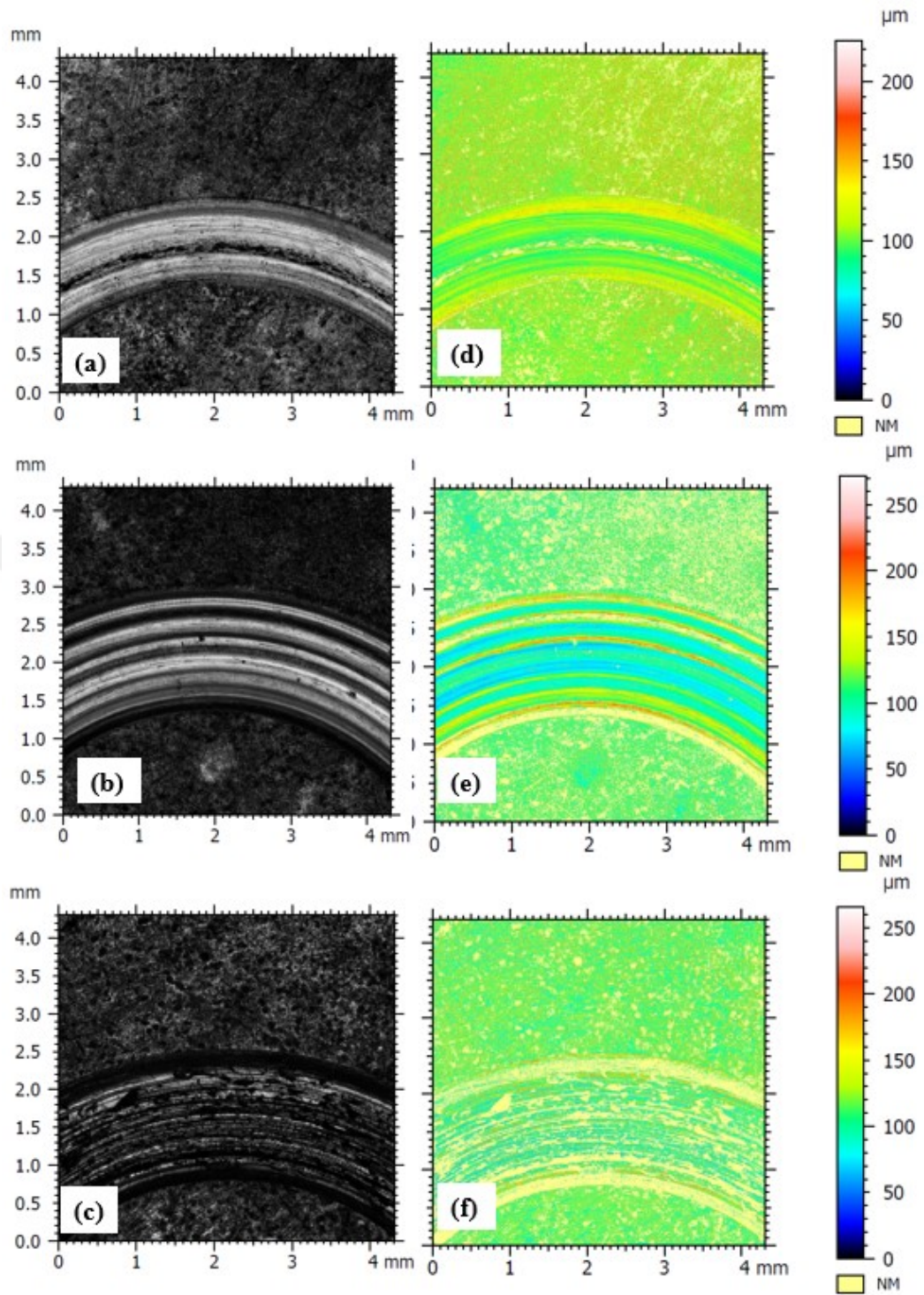


Figure 33: Wear track of boronized AISI 321 samples at different sliding distances of (a) 200 m, (b) 600 m, and (c) 800 m with color coded depth image of (d) 200 m, (e) 600 m, and (f) 800 m tested at room temperature.

In a similar way, Figure 33 represents Wear track of boronized AISI 321 samples

at different sliding distances with color coded depth images and contour bands. From the sample tested for 100 m sliding distance to 300 m, the highest depth of the wear track is greatly increased. The difference between the highest depth of the sample tested for 600 m and 300 m, on the other hand, is not significant. This is due to steady-state wear after 300 meters of sliding distance. Furthermore, the wear scar width is extended up to 300 m and then stabilized (Figure 33 d-f). Besides, the depth fluctuation on different diameter of wear track can be observed for the boronized sample due to the formation of grooves and entrapment of boron particles through them. This phenomena will be discussed using SEM images in detail (See Figure 49 c and d).

Figure 34 shows the friction coefficient for different sliding distances of AISI 321 samples at 600°C. It can be seen that the friction coefficient of all the samples at high temperature is much lower than that in the room temperature. In general, all samples' friction coefficient curves may be separated into two stages: running-in and steady wearing. At room temperature, the running-in stage of both boronized and WC coated AISI 321 samples lasts only a short time, with a very high instable friction coefficient at the start of the test, whereas it lasts much longer at elevated temperatures, with the duration decreasing as the temperature rises. The interfacial coating will be abraded and the cold welding action will occur in the contact site at the start of sliding between friction pairs. As a result, a large shear force is required to cut the "welded" spot, and the friction coefficient is significantly larger than in the stable wearing stage. Because the cold welding effect is exacerbated at higher temperatures, the running-in stage lasts longer. A descending trend can be seen for all conditions. Different wear mechanisms during wear testing might explain the discrepancies in friction coefficient behavior. Boronized and WC coated samples experience abrasive wear at room temperature, in which the particles wear off from friction pairs and function as abrasive, resulting in a higher friction coefficient. When particles wear off from friction pairs at a high temperature, they are simpler to cold weld and move between friction pairs.

As a result, at high temperatures, the adhesive wear mechanism takes precedence [71].

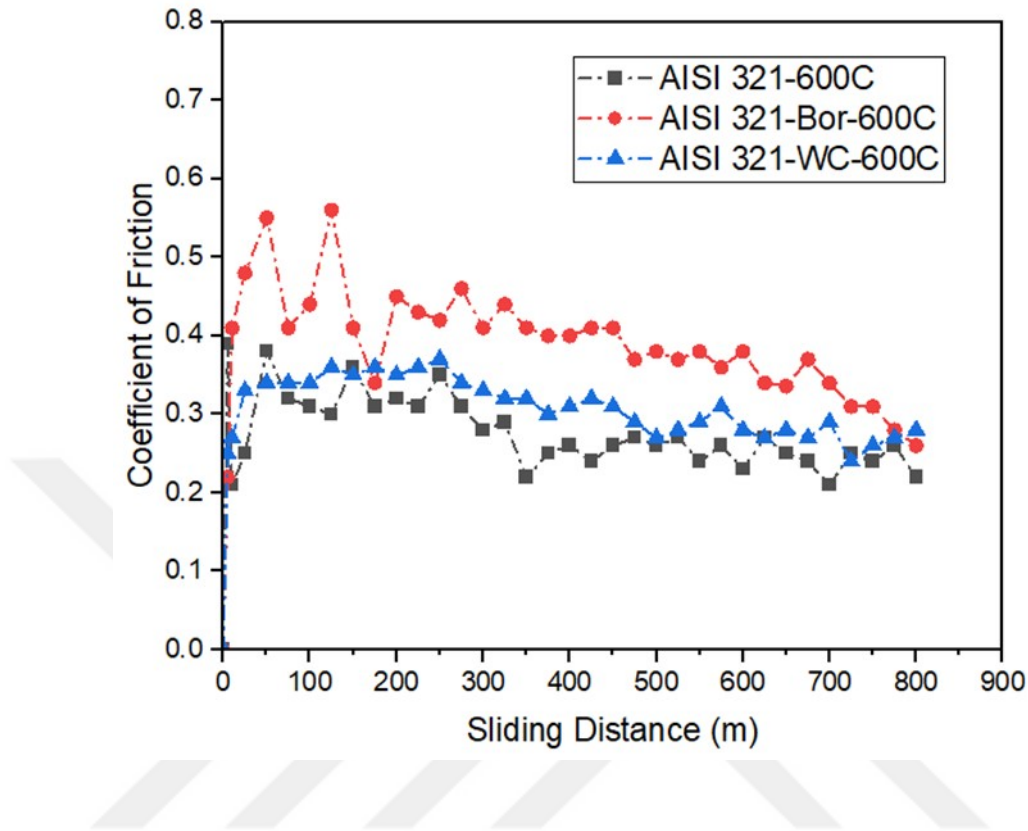


Figure 34: Friction coefficient versus sliding distance of AISI 321 at 600°C for different uncoated and coated conditions.

Figure 35 demonstrates the wear rates of AISI 321 samples at high temperature. In contrary to the room temperature, the wear rate of boronized AISI 321 at 600°C is showing a similar trend as the uncoated condition. This can be related to decomposition of boron particles after matrix softening at high temperature. Unlikely, WC coated wear rate was still low with some fluctuations due to delamination of WC coated layer from the soft matrix.

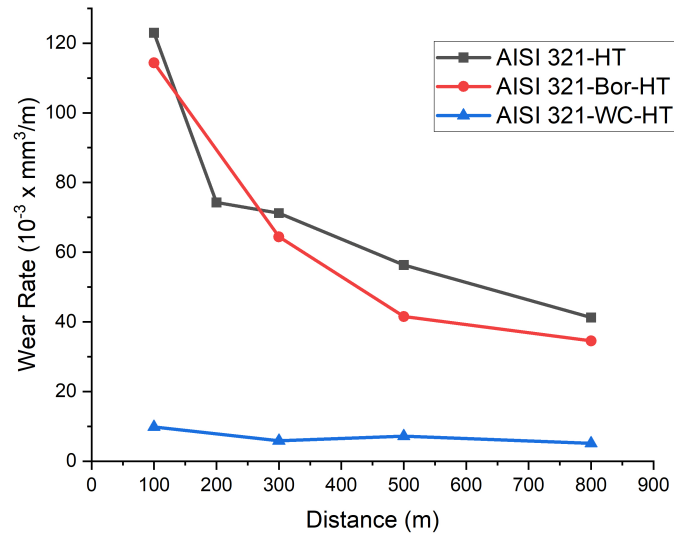


Figure 35: Wear rates of AISI 321 stainless steel samples at 600°C

Figure 36 shows the wear track of the uncoated AISI 321 sample tested at 600°C. The reason of the irregular shape of the wear track could be adhesive wear mechanism at high temperature. At this temperature, the yield strength of the material decreases. The yield strength of metals is an important concern in sliding wear, that is the major reason of the adhesive wear mechanism [39].

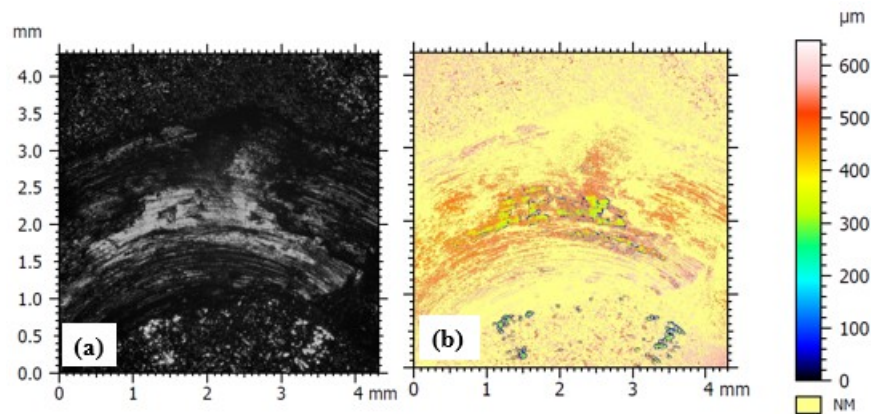


Figure 36: Wear track of uncoated AISI 321 sample after 100 m sliding distance tested at 600°C.

Figure 37 demonstrates the wear track of the boronized AISI 321 sample tested

at 600°C after different sliding distances. Non-uniform wear tracks were detected on the samples which can be related to decomposition of boron particles from the surface and formation of steps on the edges of delaminated layers. Also, the adhesive wear mechanism at high temperatures could be the cause of the irregular form of the wear track [39]. Moreover, the agglomeration of boron particles due to the composition of hard particles from soft matrix at high temperature can be seen in Figure 37c.

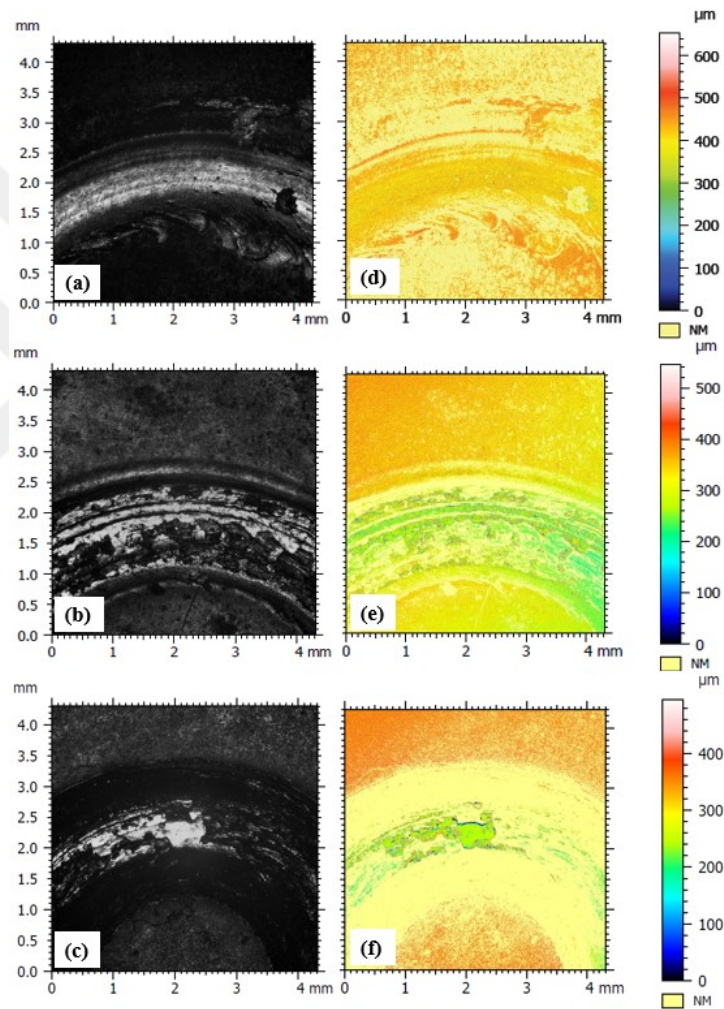


Figure 37: Wear track of boronized AISI 321 samples at different sliding distances of (a) 100 m, (b) 300 m, and (c) 800 m with color coded depth image of (d) 100 m, (e) 300 m, and (f) 800 m tested at 600°C.

In Figure 40, the SEM images of uncoated and coated AISI 321 steel are shown

after room temperature wear tests. The uncoated sample's wear track shows the existence of typical wear scars in the sliding direction of a worn surface, as well as ploughing grooves in some areas (Figure 40a). Multiple cracks and delamination fractures occurred along the ploughing grooves, as shown in a higher magnification in Figure 40b. Wear tests usually result in significant strain levels at the sample's surface that is in contact with the counter ball [72]. The formation of grooves was observed on the boronized samples and subsequently the entrapped boron particles were detected as shown in Figure 40c and d. Moreover, agglomeration of WC particles is shown in Figure 40d and f. The EDX analysis obtained on the room temperature tested WC Coated AISI 321 sample. It can be seen that in Figure 38 abrasive counter part's Alumina (Al_2O_3) particles flaked off onto agglomerated WC layers.

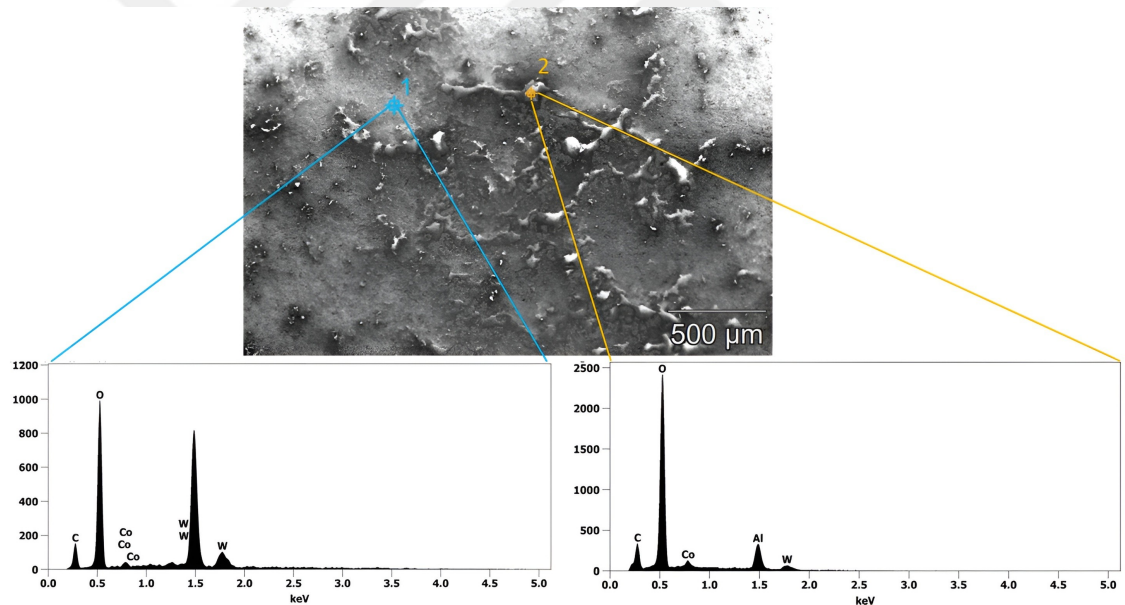


Figure 38: EDX analysis of WC Coated AISI 321 sample for spectrum of debris of Alumina particles

The wear tracks of uncoated and coated samples after high temperature wear test observed by SEM are illustrated in Figure 41. The damaged wear track and delaminated sheets due to the high temperature of 600°C were detected on the uncoated samples (Figure 41a and b). On the EDX analysis of worn surface of

the sample, there were boron particle with diameter about 10 μm is identified in the sample after wear test can be seen in Figure 39. Moreover, it can be seen that boron density is decreased on the worn surfaces. On the boronized samples, delamination layers and steps occurred after high temperature test (Figure 41c and d). The corner of these steps are suitable sites to entrap the boron particles and subsequently decrease friction coefficient (See Figure 34). Similar to the room temperature wear tests, the agglomerated WC particles were detected on the wear track of the sample.

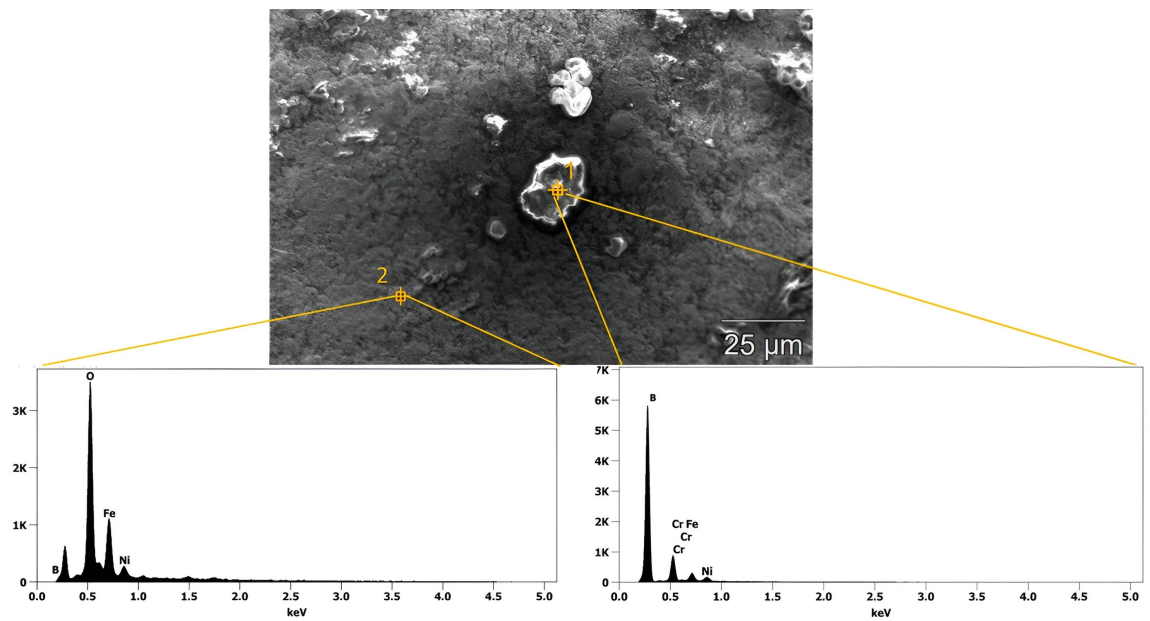


Figure 39: EDX analysis of boronized AISI 321 sample for spectrum of decomposed boron particles.

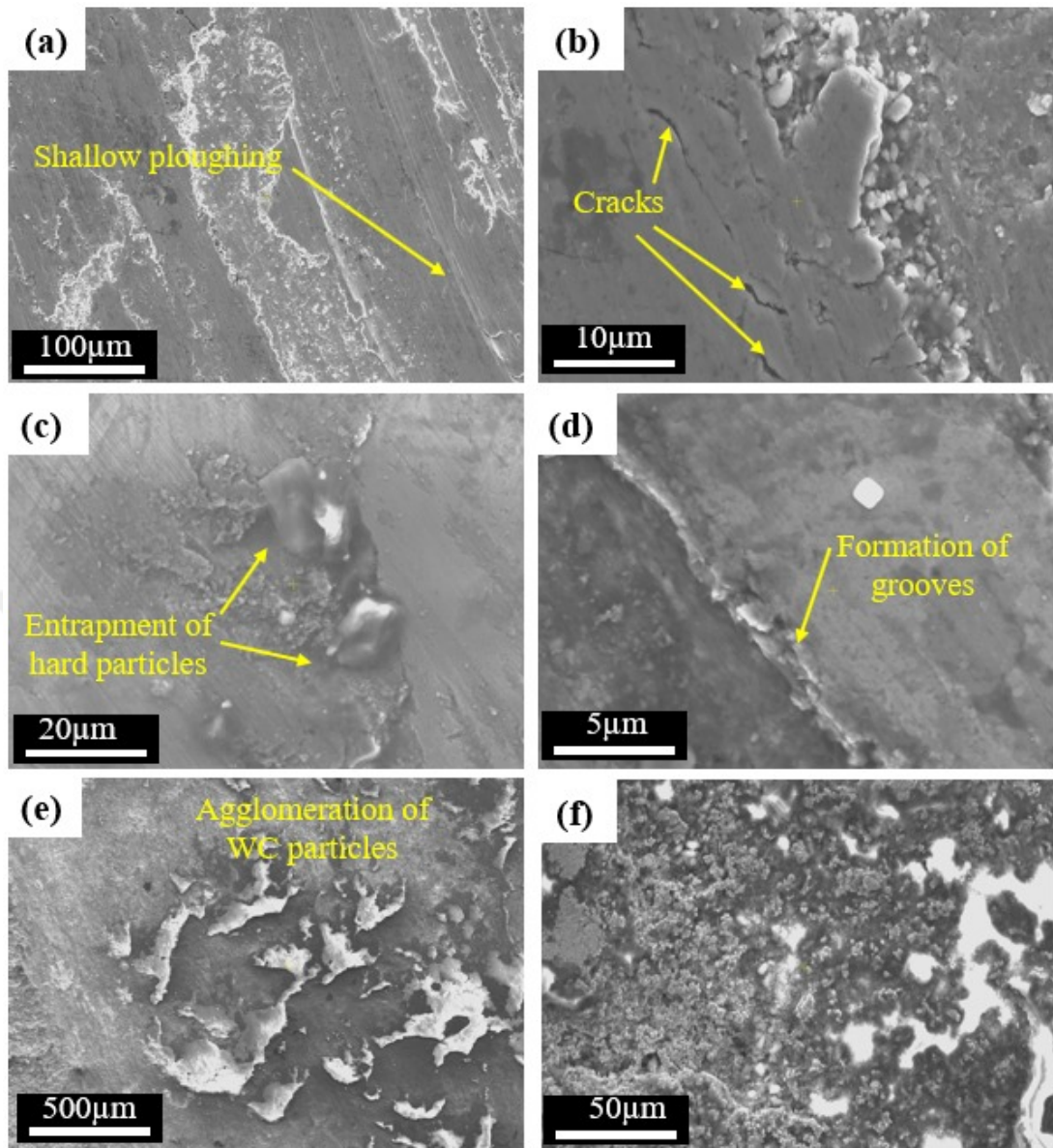


Figure 40: SEM images of AISI 321 samples at room temperature a-b uncoated; c-d boronized; e-f WC coated

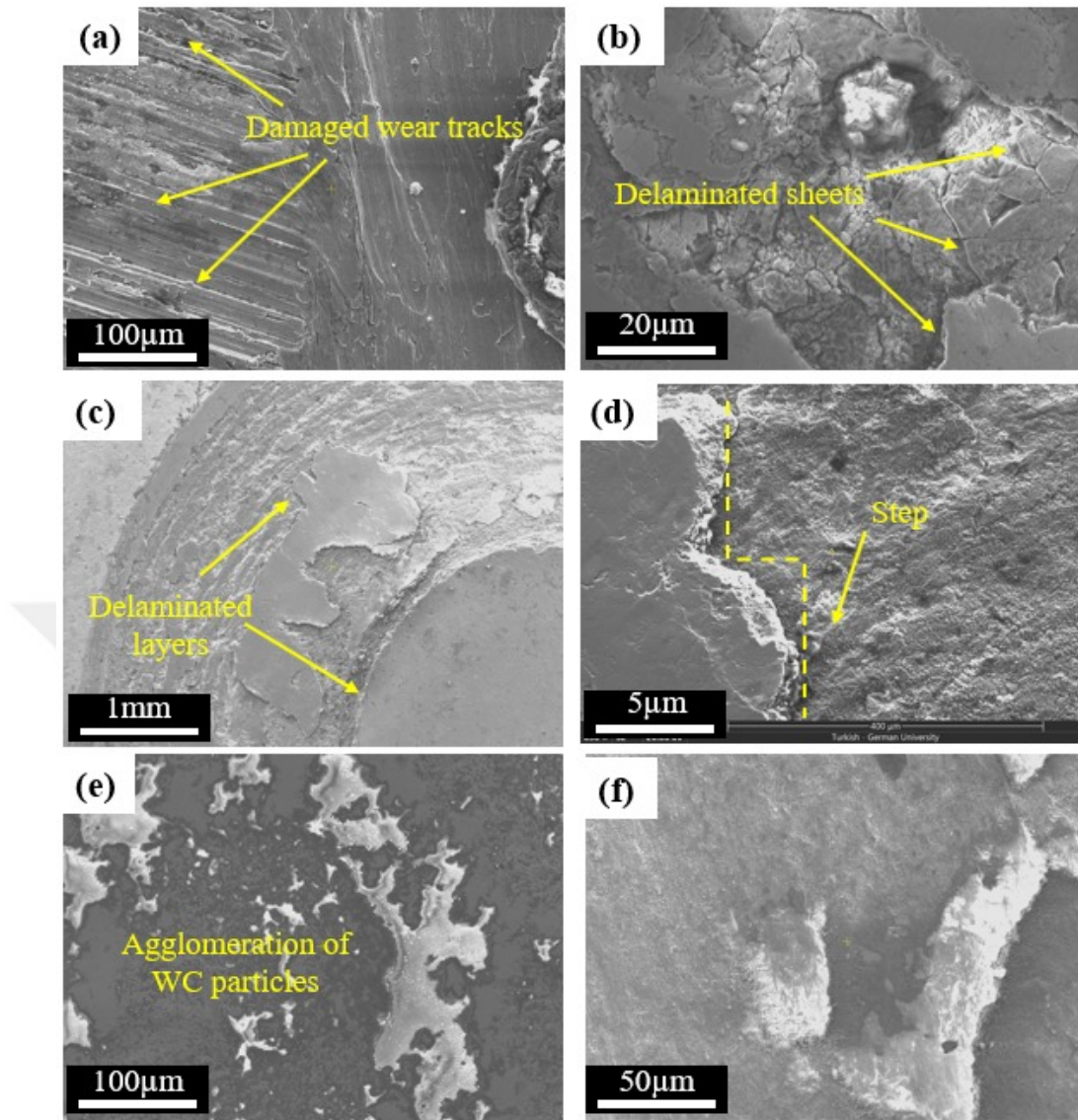


Figure 41: SEM images of AISI 321 samples at 600°C a-b uncoated; c-d boronized; e-f WC coated

4.3 Tensile Behavior of Inconel 625 Superalloy

4.3.1 Mechanical Properties

In this section, microhardness and tensile test results of the Inconel 625 superalloy at different uncoated and coated conditions are shown at room and high temperatures.

As shown in Figure 42 the hardness of as-received sample was around 221 HV with a standard deviation of 3 HV. Also, the core microhardness of boronized

sample was 345 HV with a standard deviation of 12 HV while the microhardness of the surface was increased up to about 2150 HV with 30 HV standard deviation. WC coated sample was 2423 HV with a standard deviation lower than 1%. This enhancement in microhardness of boronized sample, is related to diffusion of boron particles into the substrate and formation of hard borides within the surface of material.

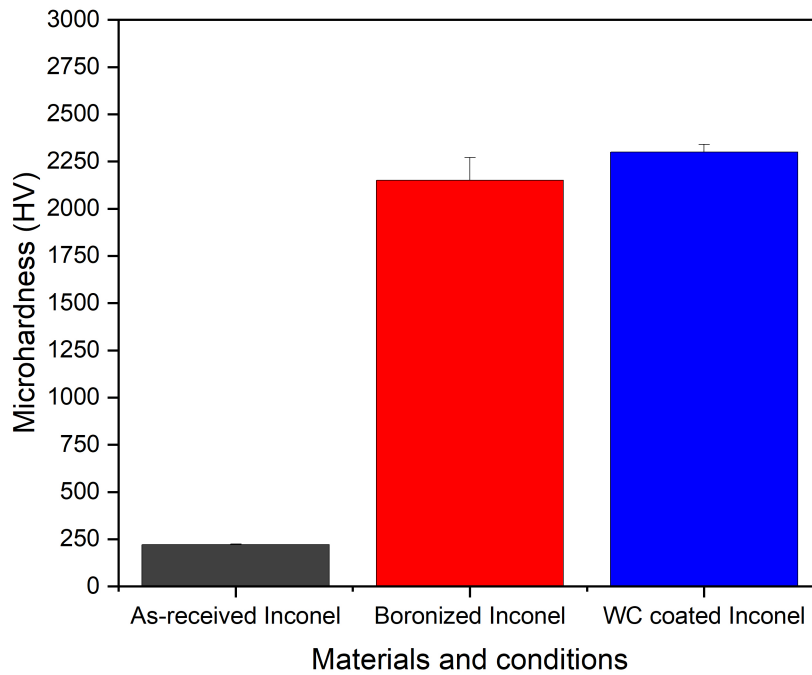


Figure 42: Hardness test results of Inconel 625 samples

Figure 43 illustrates the room temperature tensile stress-strain curves of the samples for different conditions. It is clearly seen that, the yield strength (YS) and ultimate tensile strength (UTS) of the samples after boronizing reduced noticeably. Accordingly, this reduction in YS is more significant in boronized sample which is attributed to the softening of the material throughout the boronizing process at high temperature of 1100°C for 8 hours which negatively affects the strength of the samples. Nevertheless, the addition of boron particles moderates

the negative effect of the softening by instigating Orowan strengthening to the material specifically near the surface of the samples where the concentration of boron particles is higher than the beneath layers [73, 74]. WC coated Inconel 625 sample was shown similar tensile behavior with 321 stainless steel. Its yield strength was higher than others because of the ceramic effect. Slope of the elastic section of the WC coated specimen is higher than the uncoated and boronized specimens. It can be caused by higher Young Modulus of the coating on the specimen [75].

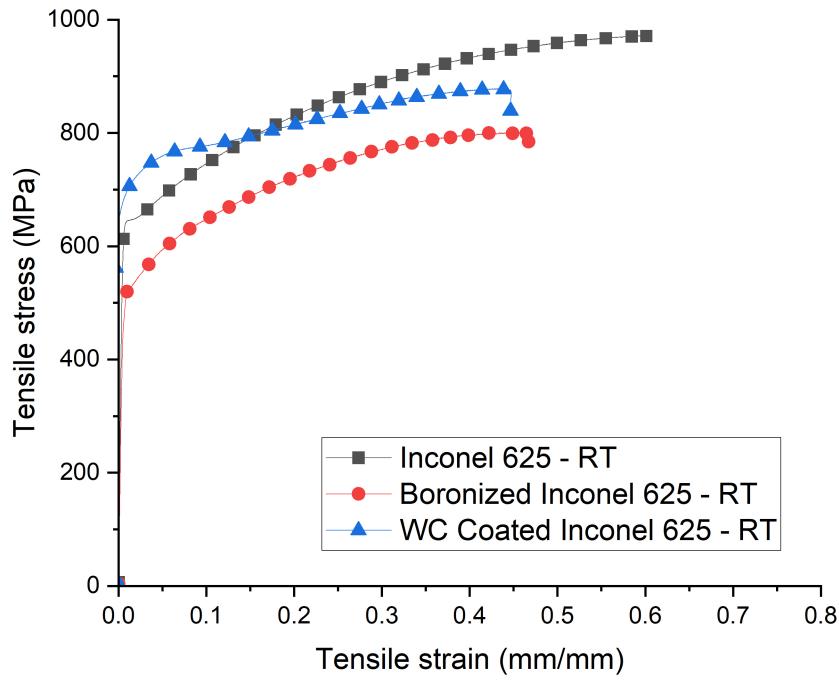


Figure 43: Tensile strain stress curves of Inconel 625 for as-received and coated samples at room temperature

When the elastic modulus of the WC coated sample with 36 mm gage length is performed with the composite material formulation from Equation 13 and dimensions that seems on Figure 44, the theoretical modulus of elasticity was found to be 174 GPa. However, according to the test data, the elastic modulus of the sample is 153 GPa. A decrease in the modulus of elasticity may have occurred due to the high temperature of the process and the softening of the bond coating

with temperature.

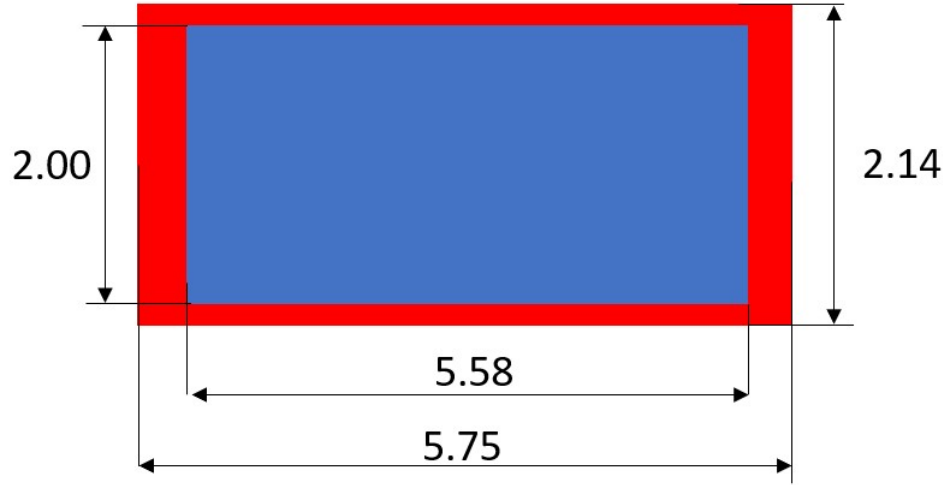


Figure 44: Gage dimensions of WC coated Inconel 625 tensile samples

The ductility of the Inconel 625 samples has been reduced for boronized condition due to dynamic strain aging at 600°C [2], as shown in Figure 45. The yield strength of the uncoated Inconel sample reduced to about 370 MPa at 600°C while the UTS decreased to about 515 MPa. Also, the strain at failure dropped to 38% at high temperature tensile testing. On the other hand, the YS and UTS of the boronized sample decreased to about 300 MPa and 320 MPa. At high temperatures, Inconel 625 shows an unstable plastic flow characterized by serrated yielding, known as the Portevin-Le Chatelier (PLC) phenomenon [76, 77, 78]. The reason of this phenomenon is typically dynamic strain ageing (DSA) [68]. The serrated plastic flow in the magnified section of the plastic region in Figure 45 proves PLC effect on the coated Inconel 625 samples [79]. Similar to AISI 321 results, it can be seen that the yield strength of the WC coated Inconel 625, higher than the others. This may be direct effect of the high strength ceramic effect at 600°C. However, high temperature tensile behavior of the WC coated Inconel 625 is more ductile than AISI 321.

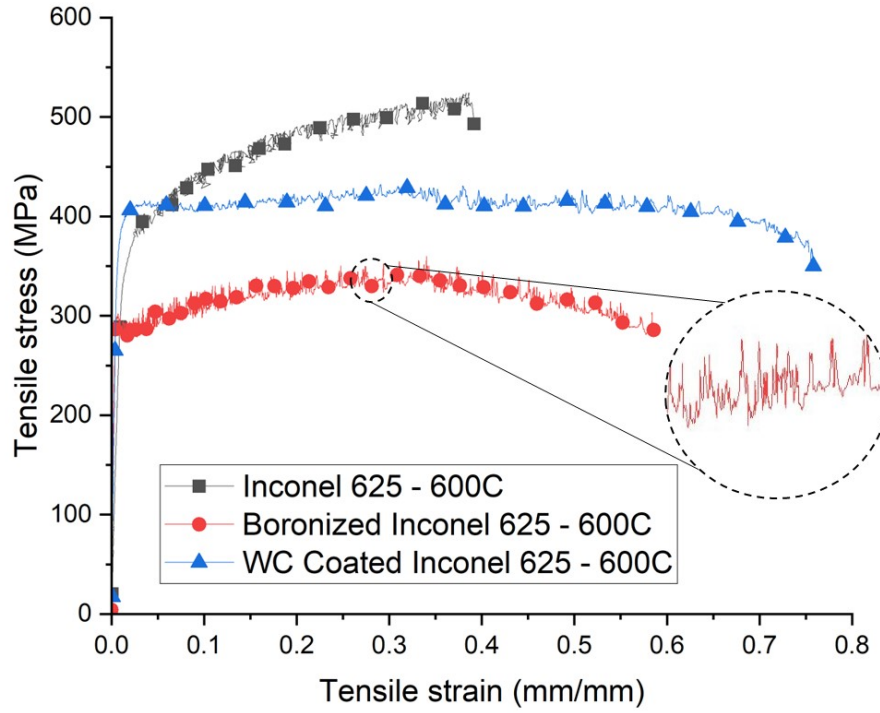


Figure 45: Tensile strain stress curves of Inconel 625 for as-received and coated samples at 600°C

4.3.2 Fracture Morphologies

Fracture morphology of uncoated and boronized Inconel 625 samples after failing at room and high temperature tensile tests are shown in Figure 46 and Figure 47, respectively. As shown in Figure 46a-c, the SEM results of the fractured uncoated Inconel 625 revealed a typical ductile fracture behavior represented by fine equiaxed dimples uniformly distributed on the fracture surface of the sample (see Figure 46c). On the contrary, a binary fracture mechanism was detected on the fracture surface of the boronized Inconel 625 samples (Figure 46e). A relatively brittle fracture surface manifested by cleavages was observed on the layer near to the surface of the sample while the fracture surface of the core section was covered by transgranular cracking with tear dimples showing the ductile fracture mechanism [79, 80]. The transition of fracture surface from the surface to the

core section is pointed in Figure 46f. The low strain to failure of the boronized sample detected in Figure 43 may be attributed to the brittle fracture manner of the underneath layer.

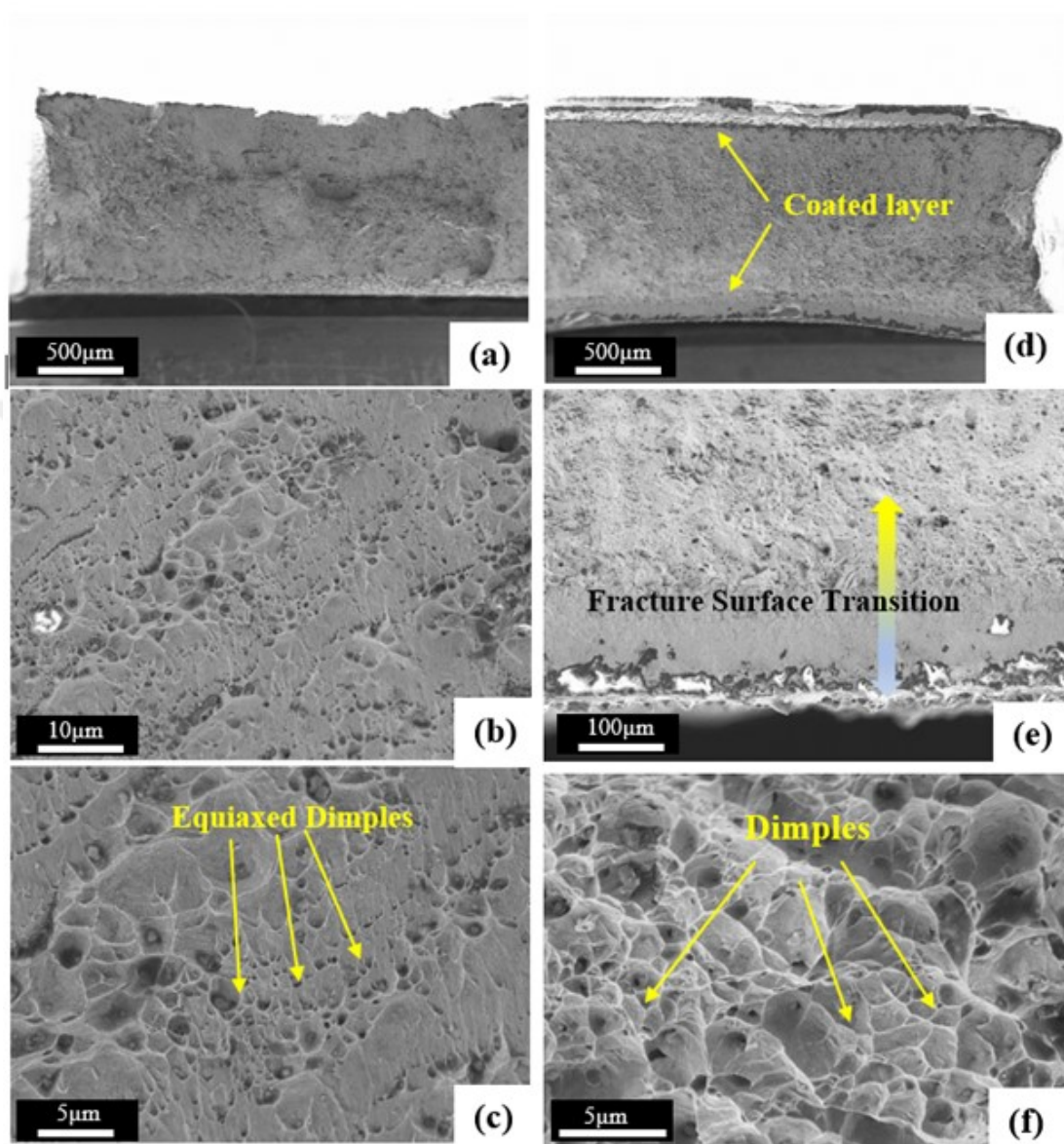


Figure 46: Fracture Surface of tested samples at room temperature for (a-c) Inconel 625 and (d-f) boronized Inconel 625 in different magnifications.

Figure 47 illustrates the fracture surface of the tested samples at high temperature of 600°C. The comparison of the SEM images in low magnification revealed that the necking of the uncoated sample is more homogenized due to the uniform fracture manner of the sample, while the boronized sample has facets on the sides

and ductile necking on the core of the fracture surface (see Figure 47a and d). Moreover, the uniform deep dimples shown in Figure 47c represented the ductile fracture mechanism on the fracture surface of the uncoated sample. Besides, high magnification SEM images of the boronized fracture surface revealed that decomposition of dispersed boron particles manifested by coarse dimples were dominant fracture mechanisms of the sample at the outer layer while the ductile fracture represented by dimples was the main fracture mode at the core section of the sample (Figure 47e) [81].

In Figure 48 and 49 the fracture mechanisms of WC coated Inconel 625 samples failed at room and high temperature are illustrated, respectively. As shown in Figure 48b, a completely different fracture surface can be observed between the outer surface and inner part of the WC coated sample. Inner surface may be identified by equiaxed dimples which reveal a ductile fracture manner while the outer surface is covered by cracks and cleavages represented the brittle fracture mechanism. Furthermore, the necking can be seen on the fracture surface of the WC coated Inconel 625 sample due to the ductile fracture behavior at high temperature (Figure 49a). The agglomeration of WC particles can be seen on the outer surface of the sample due to fracture after high temperature tensile test. Similar to the surface of samples tested at room temperature decomposition of WC particles was detected on the surface of failed samples at high temperature, and it was proved with EDX analysis(Figure 49c-d and Figure 50).

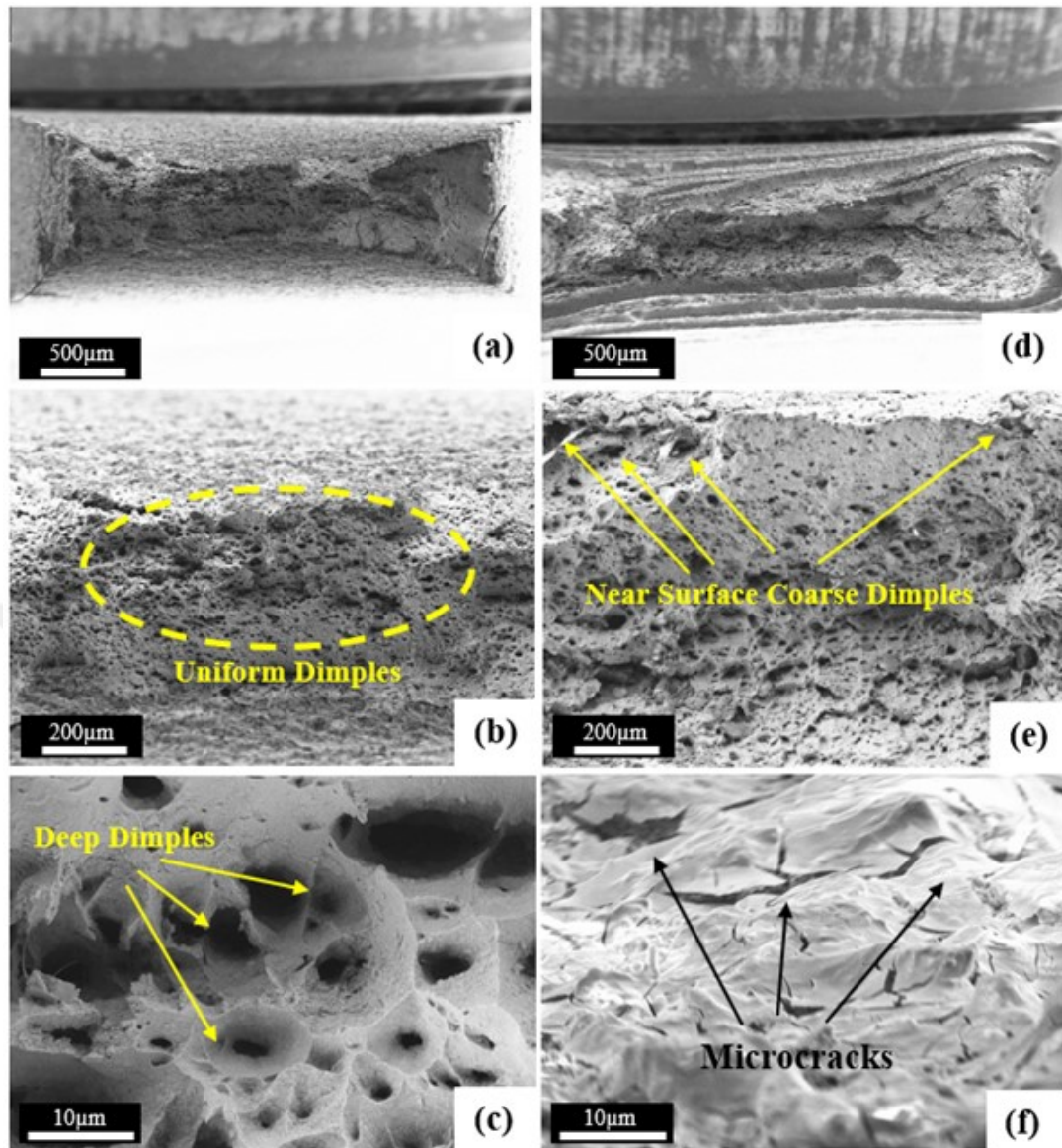


Figure 47: Fracture Surface of tested samples at 600°C for (a-c) Inconel 625 and (d-f) boronized Inconel 625 in different magnifications.

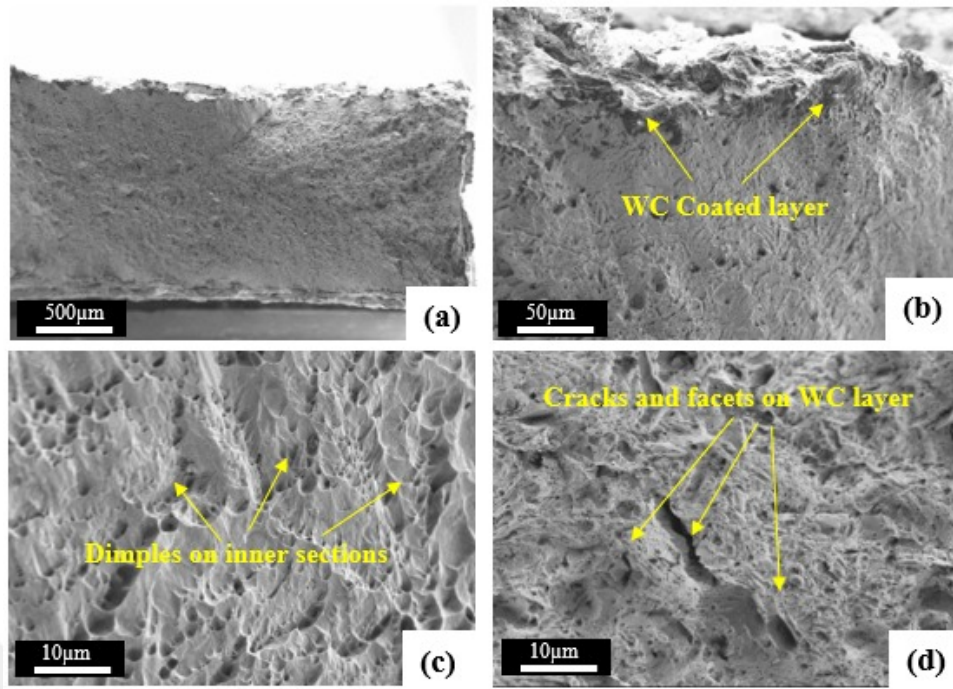


Figure 48: Fracture Surface of WC coated Inconel 625 sample at room temperature in different magnifications

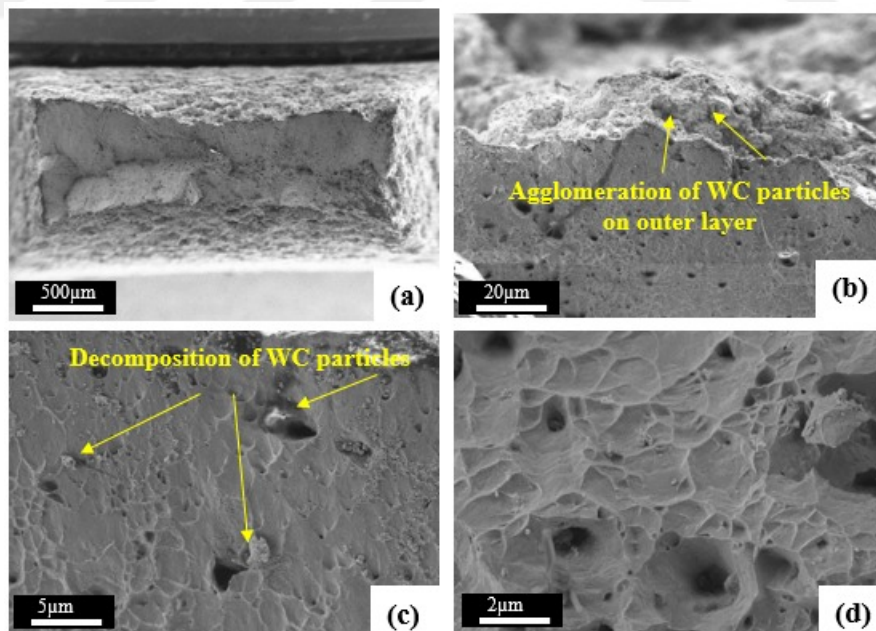


Figure 49: Fracture Surface of WC coated Inconel 625 sample at 600°C in different magnifications

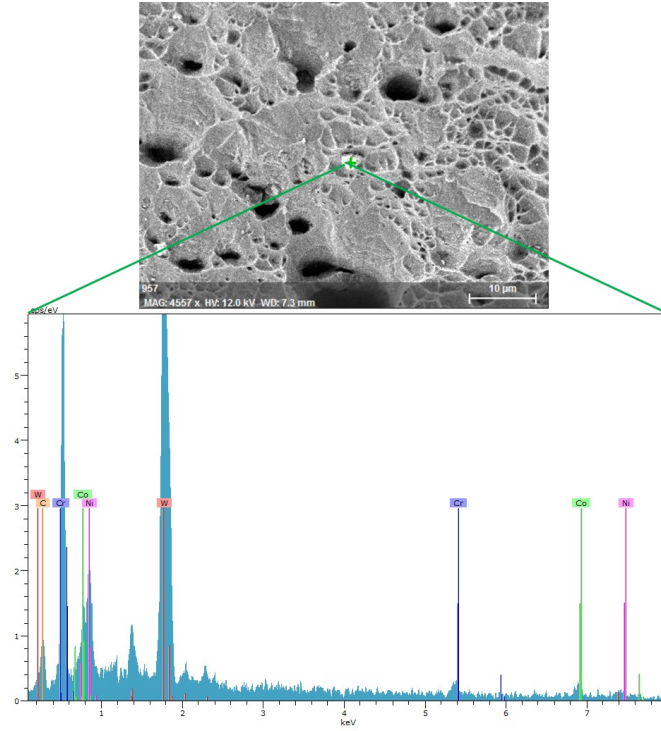


Figure 50: EDX analysis of WC coated Inconel 625 high temperature tensile test sample

4.4 *Wear Behavior of Inconel 625 Superalloy*

In this section, room and high temperature wear behavior of Inconel 625 is investigated at different coating conditions. Friction coefficient graphs not only give the information about the tribosystem but also give some clues about the wear behavior of the materials [82]. On the other hand, the materials which had same friction coefficient, might be not exhibit same wear behavior because of the energy dispersed different effects for example deformation of twins, work hardening etc [83]. For this reason, in this thesis, the wear behavior of the materials has been made by considering the friction coefficient, volume loss, wear trace observations and SEM examinations.

Friction coefficient results of room temperature tribometer tests according to ASTM G99 is given in Figure 51. As same with 321 steel COF results, all of the samples of Inconel 625 exhibit a short running-in stage because of the contact

asperities. The reason of friction coefficient exhibit peak values at the beginning, the asperities of the surface is resisted to sliding [84]. It can be seen that the highest friction coefficient value is belong to WC coated sample because of its hard self surface asperities. After 100 m distance, it has reached steady state and the coefficient friction average is 0.9 until 600 m. For the boronized samples, coefficient friction of Inconel 625 was about 0.67 at the runninn-in stage, at the end of the distance it was about 0.8. The reason of the boronized sample's graph shows upward trend can be self lubricating borided layers at the beginning, then the wear trend shifting to adhesive side due to the stic-slipping to the counterpart with the heated up the surface [27, 85].

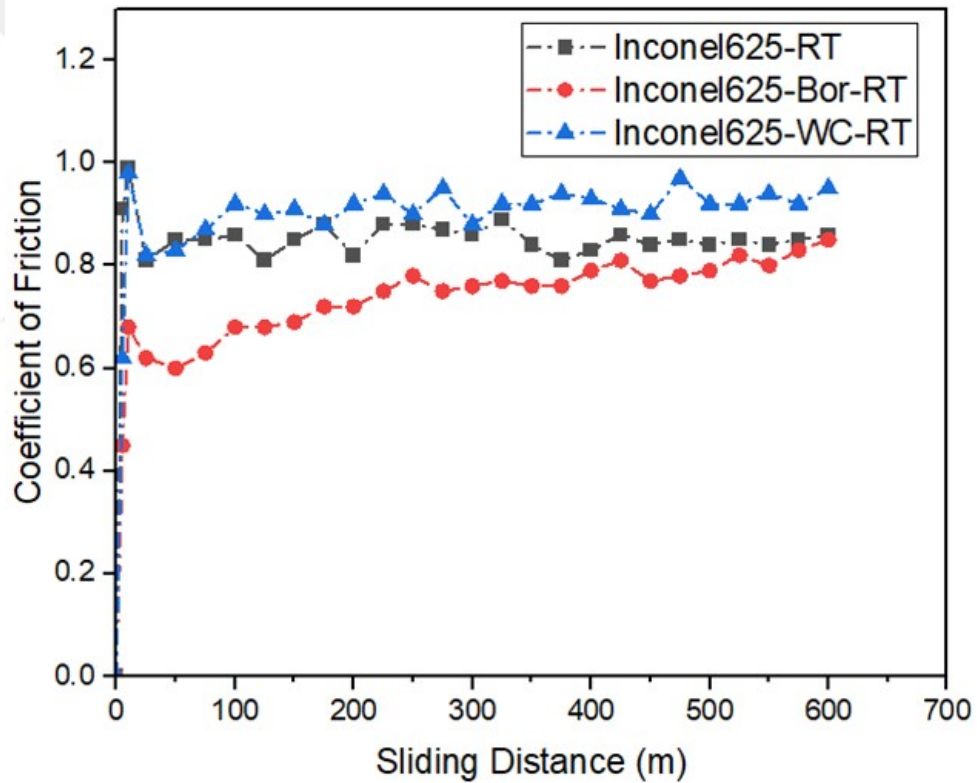


Figure 51: Friction coefficient versus sliding distance of Inconel 625 superalloy at room temperature for different uncoated and coated conditions.

Figure 52 shows the wear rate of all conditions of Inconel 625 at room temperature. Inspection of room temperature wear test results revealed that the wear rates of Inconel 625 uncoated and coated samples were similar to the results of AISI

321. WC coated samples' wear rate was tremendously lower than the others. It can be noticed that the ascending trend of wear rate for uncoated sample is related to increasing the level of material removal by increasing the sliding distance.

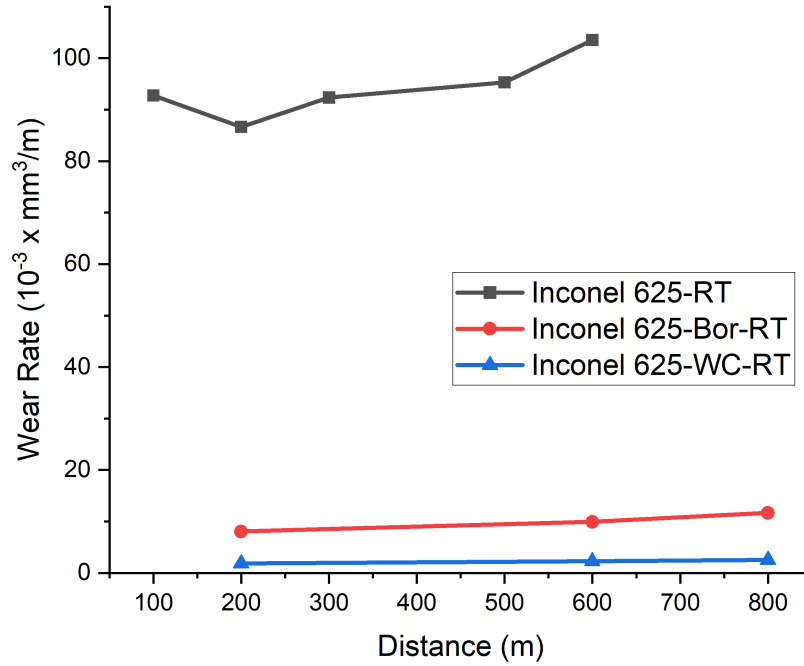


Figure 52: Wear rates of Inconel 625 superalloy samples at room temperature

Figure 53 represent wear track of Inconel 625 samples after 100, 300 and 800 m distances at room temperature wear tests. Additionally, depth of tracks are also given with contour bands. It can be seen that wear track width is increased with the distance but the increasing ratio is higher between 100-300 m interval. It is confirmed that the wear behavior reach the steady-state condition [86].

Similarly, in Figure 54, boronized Inconel 625 samples' wear track observations are given after room temperature tests. It can be seen that there is not much change in the wear trace even if the distance increases. Wear resistance is influenced by the hardness and thickness of the boride layers produced on the surface. Increased hardness and thickness provide better resistance to three-body and two-body abrasion, as well as increased resistance to wear debris formed by

the surface [87]. The penetration depth of abrasive particles in three-body wear have been substantially lower in the presence of hard boride layers, resulting in a reduction in volume losses. When a tangential force is applied to two-body abrasive type wear, particles penetrating the surface can also plow the surface. When the material's hardness is enhanced, it becomes more resistant to plowing [88].

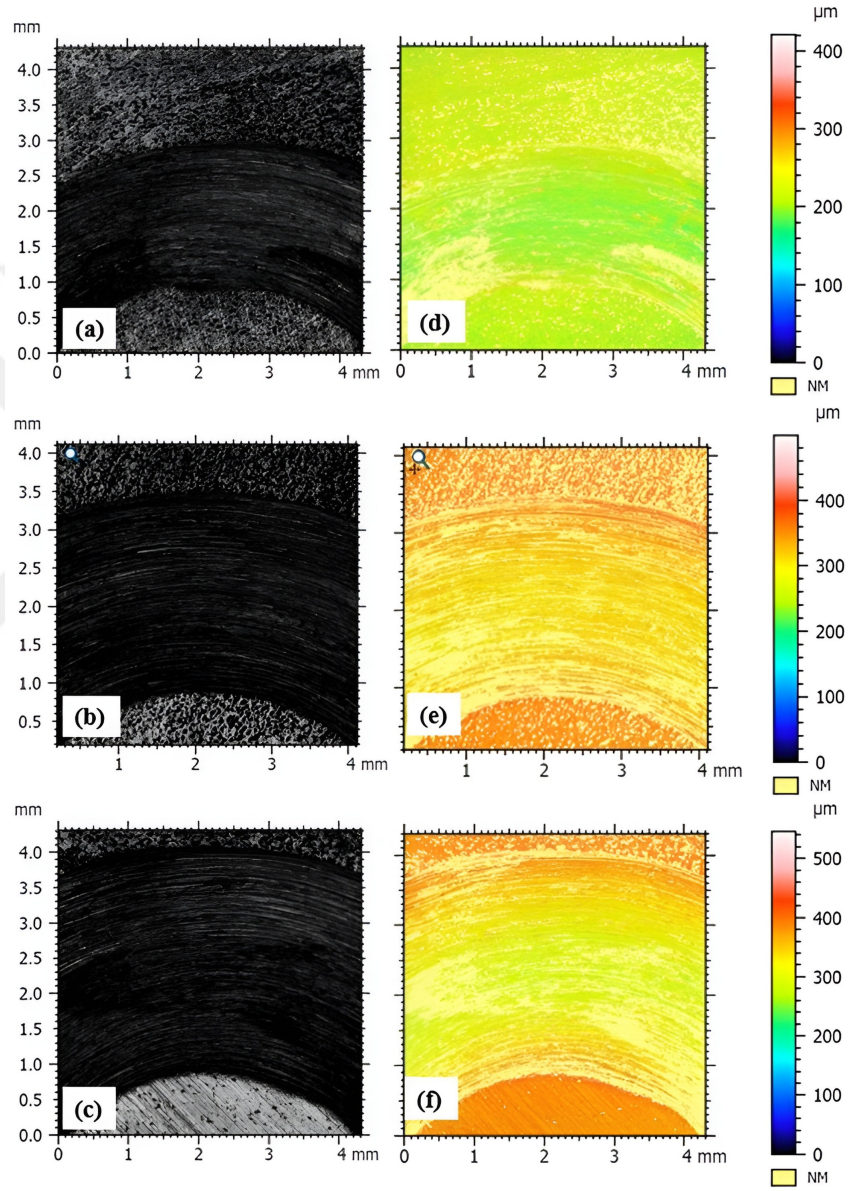


Figure 53: Wear track of Inconel 625 samples at different sliding distances of (a) 100 m, (b) 300 m, and (c) 800 m with color coded depth image of (d) 100 m, (e) 300 m, and (f) 800 m tested at room temperature.

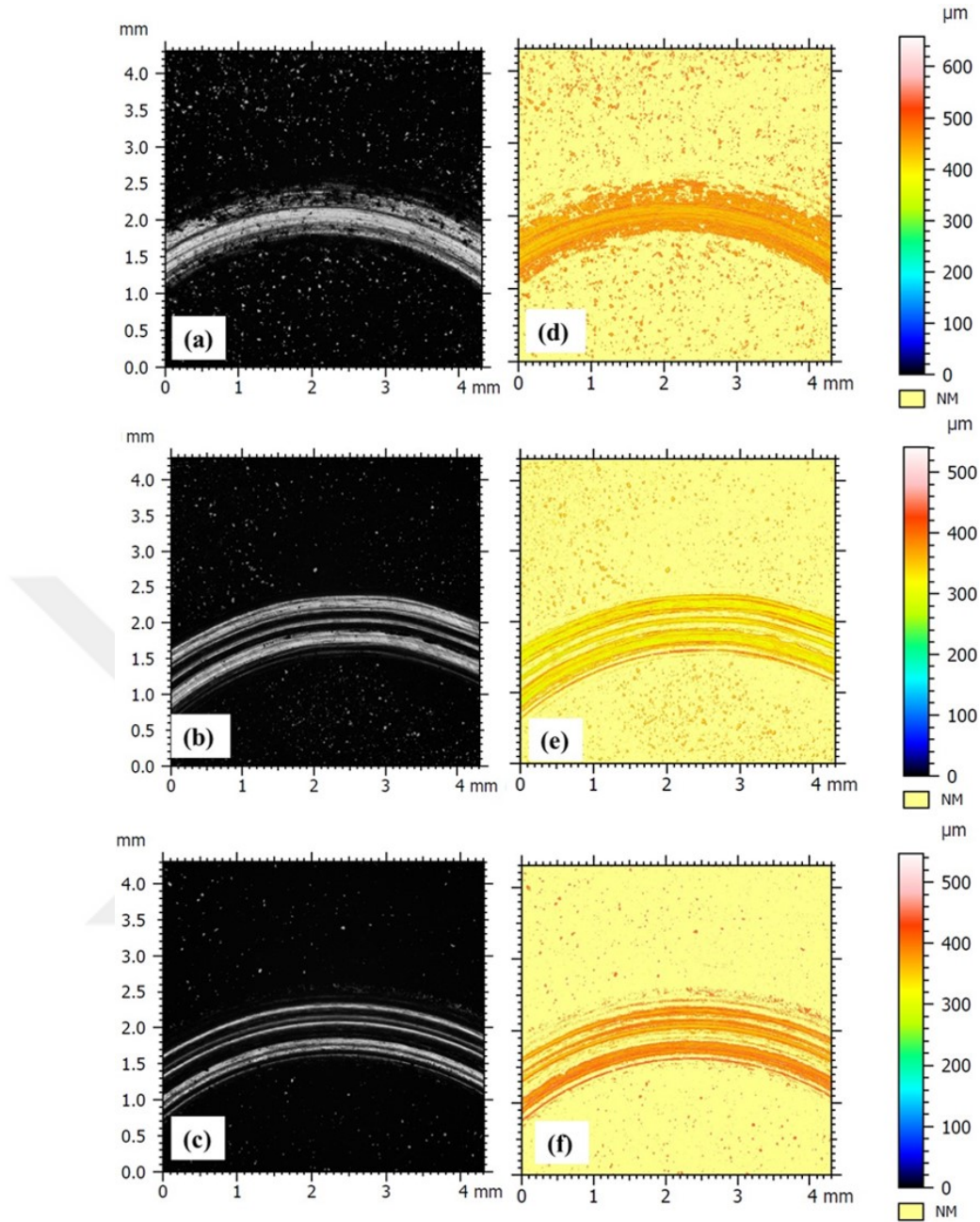


Figure 54: Wear track of boronized Inconel 625 samples at different sliding distances of (a) 200 m, (b) 600 m, and (c) 800 m with color coded depth image of (d) 200 m, (e) 600 m, and (f) 800 m tested at room temperature.

In Figure 55 represents the variation of friction coefficient of Inconel 625 samples for different distances at 600°C. The friction coefficient of all the samples at high temperature is substantially lower than that at room temperature, as can be shown. The running-in stage of coated Inconel 625 samples only lasts a short time

at room temperature, with a very high unstable friction coefficient at the start of the test, but it lasts much longer at 600°C, with the duration reducing as the temperature rises. At the onset of sliding between friction pairs, the interfacial coating will be abraded, and cold welding activity will occur in the contact site. As a result, cutting the "welded" place requires a lot of shear effort, and the friction coefficient is much higher than in the steady wearing stage. The running-in stage takes longer since the cold welding effect is amplified at higher temperatures. For all conditions, there is a downward trend. The variations in friction coefficient behavior could be explained by different wear mechanisms during wear testing. At room temperature, boronized and WC coated samples experience abrasive wear, in which particles wear away from friction pairs and act as abrasive, resulting in a higher friction coefficient. It is easier to cold weld and transport particles between friction pairs when they wear off from friction pairs at high temperature. As a result, the adhesive wear mechanism gains primacy at high temperatures [71].

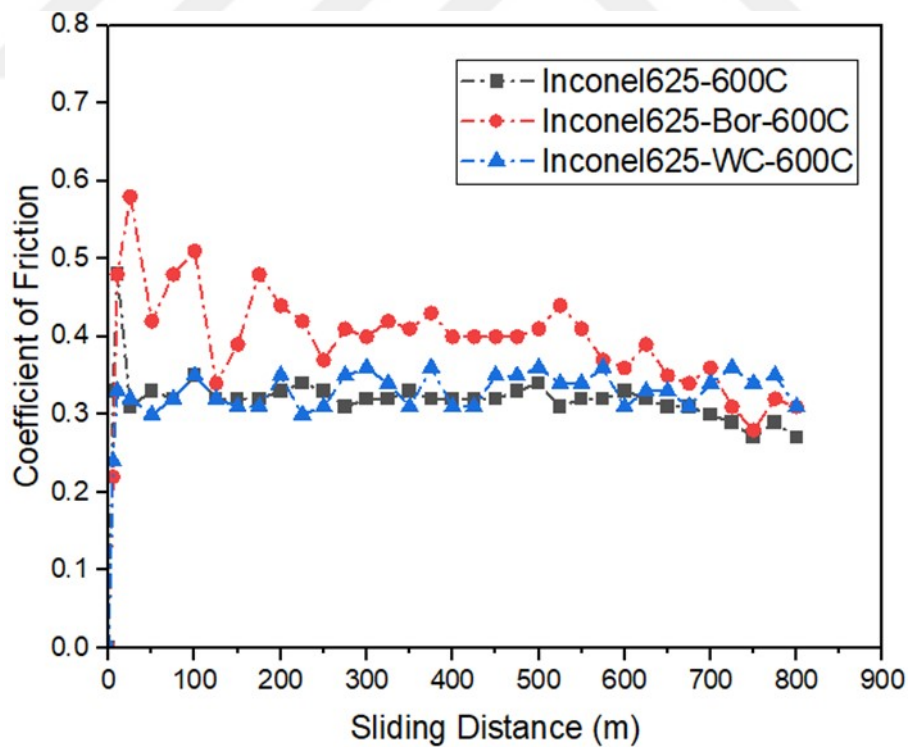


Figure 55: Friction coefficient versus sliding distance of Inconel 625 superalloy at 600°C for different uncoated and coated conditions.

Figure 56 demonstrates the wear rate of Inconel 625 samples at high temperature. The high wear rate was observed after sliding distance of 100 m for WC coated sample and afterwards descending trend can be seen for further sliding distances. This can be attributed to delamination of WC coated layer at the very beginning stage of high temperature wear test. In contrary, a descending trend of wear rates was observed for the uncoated and boronized samples follow-up with a jump from 500 m to 800 m sliding distance. This unusual results is due to decomposition of particles and delamination of the layers after cracking on the boronized and uncoated samples, respectively.

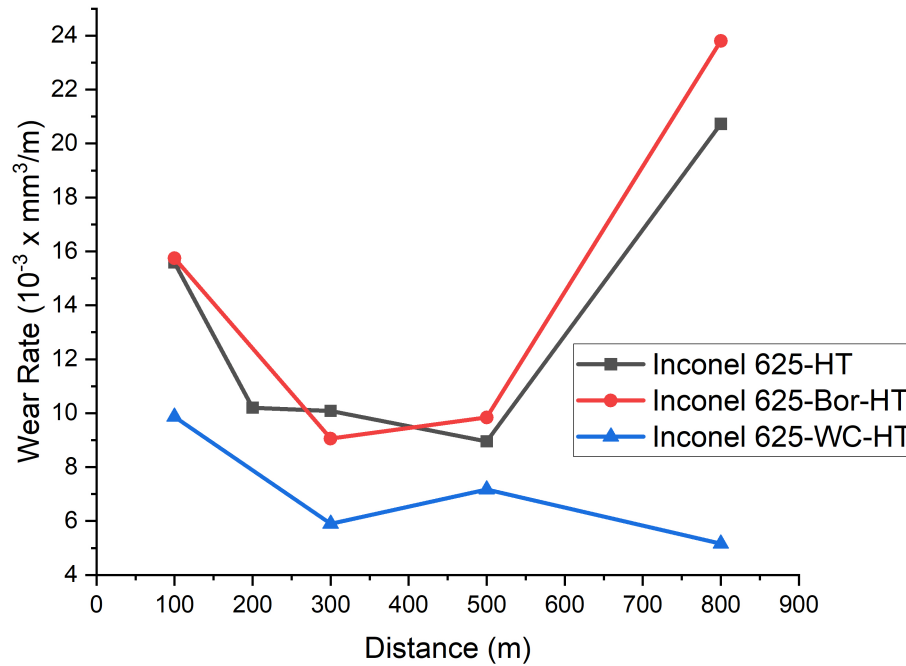


Figure 56: Wear rates of Inconel 625 superalloy samples at 600°C

Figure 57 illustrates the wear track of as-received Inconel 625 sample that was tested at high temperatures. The adhesive wear mechanism at high temperatures could be the cause of the irregular form of the wear track. The material's yield strength falls at this temperature. The yield strength of metals is a major concern in sliding wear, and it is the primary cause of adhesive wear [39].

The wear track of the boronized Inconel 625 sample evaluated at 600°C after various sliding distances is shown in Figure 58. On the samples, non-uniform wear tracks were seen, which could be attributed to the disintegration of boron particles from the surface and the creation of steps on the margins of delaminated layers. The uneven shape of the wear track could also be due to the adhesive wear mechanism at high temperatures [39].

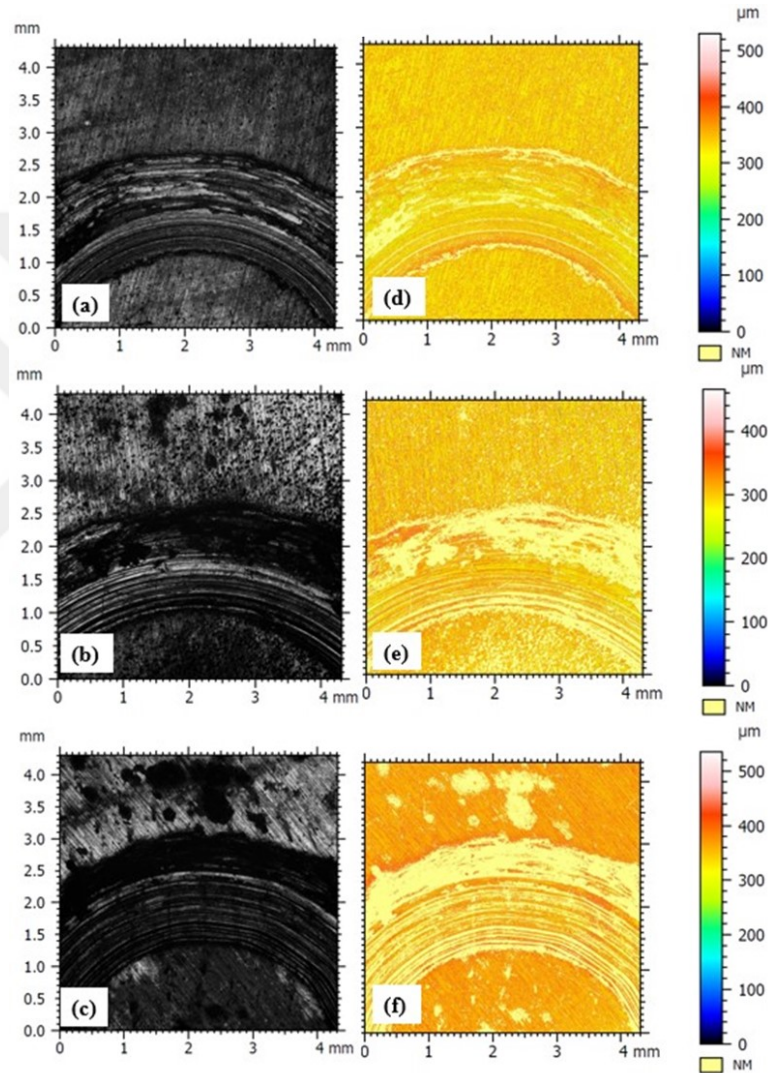


Figure 57: Wear track of Inconel 625 samples at different sliding distances of (a) 100 m, (b) 300 m, and (c) 800 m with color coded depth image of (d) 100 m, (e) 300 m, and (f) 600 m tested at 600°C.

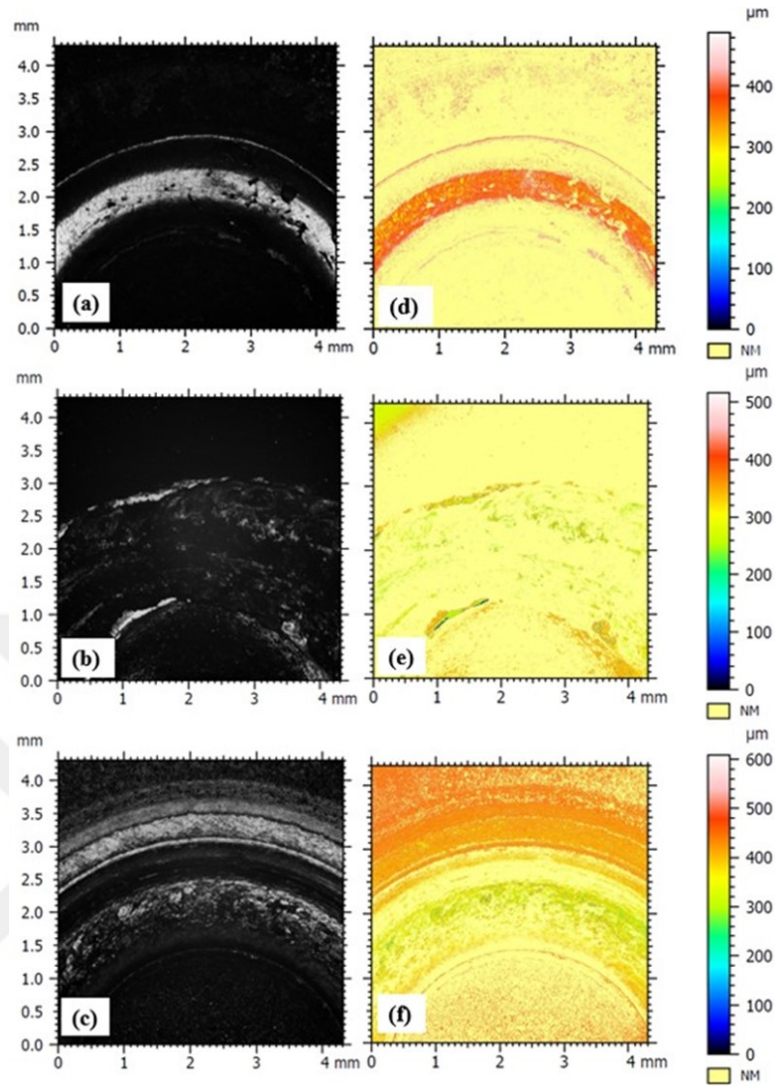


Figure 58: Wear track of boronized Inconel 625 samples at different sliding distances of (a) 300 m, (b) 500 m, and (c) 800 m with color coded depth image of (d) 300 m, (e) 500 m, and (f) 800 m tested at 600°C.

In Figure 60, the SEM images of uncoated and coated Inconel 625 are illustrated after room temperature wear tests. A typical wear tracks were observed on the surface of the uncoated sample (see Figure 60a). Moreover, in some regions delaminated layers and grooves were found after wear test as shown in Figure 60b. Multiple cracks and delamination fractures occurred around the decomposed boron particles due to the sliding and heat generated during the wear test (Figure 60c-d). The EDX analysis of decomposed boron particles can be seen in Figure 59. It

can be seen that boroxide particles on the worn surface. The Nickel base material can be seen on the worn surface. Also, inspection of SEM images revealed the agglomeration of WC particles and WC layers after wear test which is attributed to high wear resistance of WC layer (Figure 60e-f).

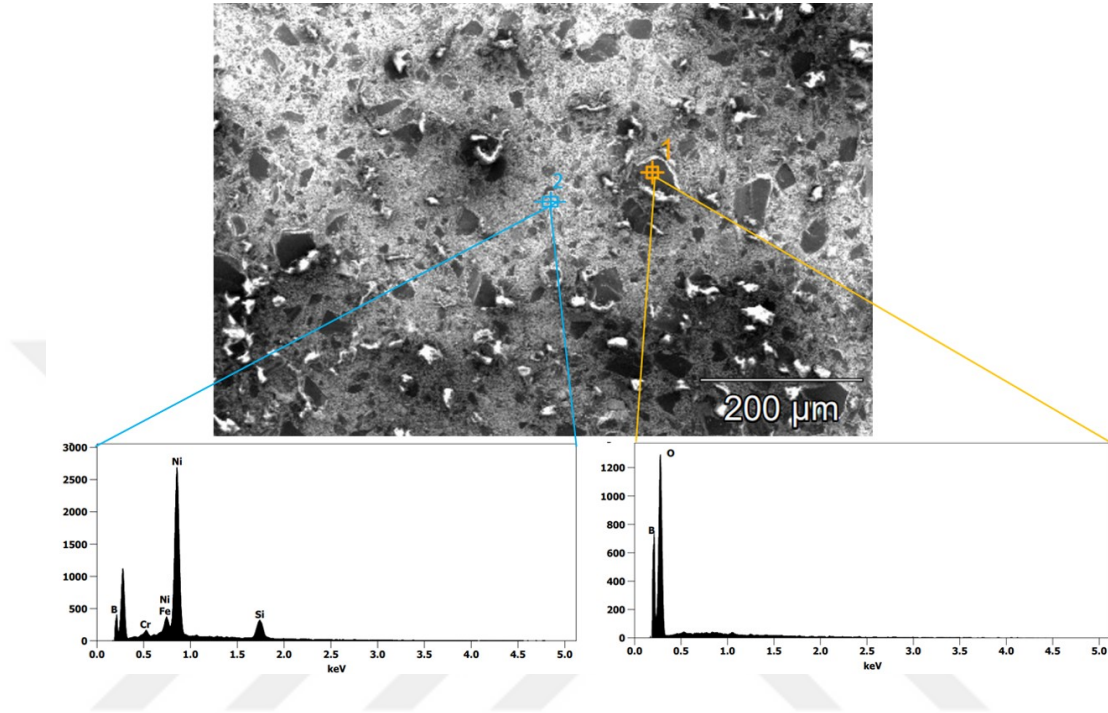


Figure 59: EDX analysis of room temperature tested boronized Inconel 625 sample

The wear tracks of uncoated and coated samples after high temperature wear test observed by SEM are demonstrated in Figure 61. The damaged wear track and oxide layer can be seen in Figure 61a and b due to the high temperature of 600°C were detected on the uncoated samples. Furthermore, after high temperature wear test, shear bands and agglomeration of boron particles were detected on the boronized sample. This can be attributed to high shear force after dispersion of boron particles and strength mismatch between the matrix and coated layer (see Figure 61c and d).

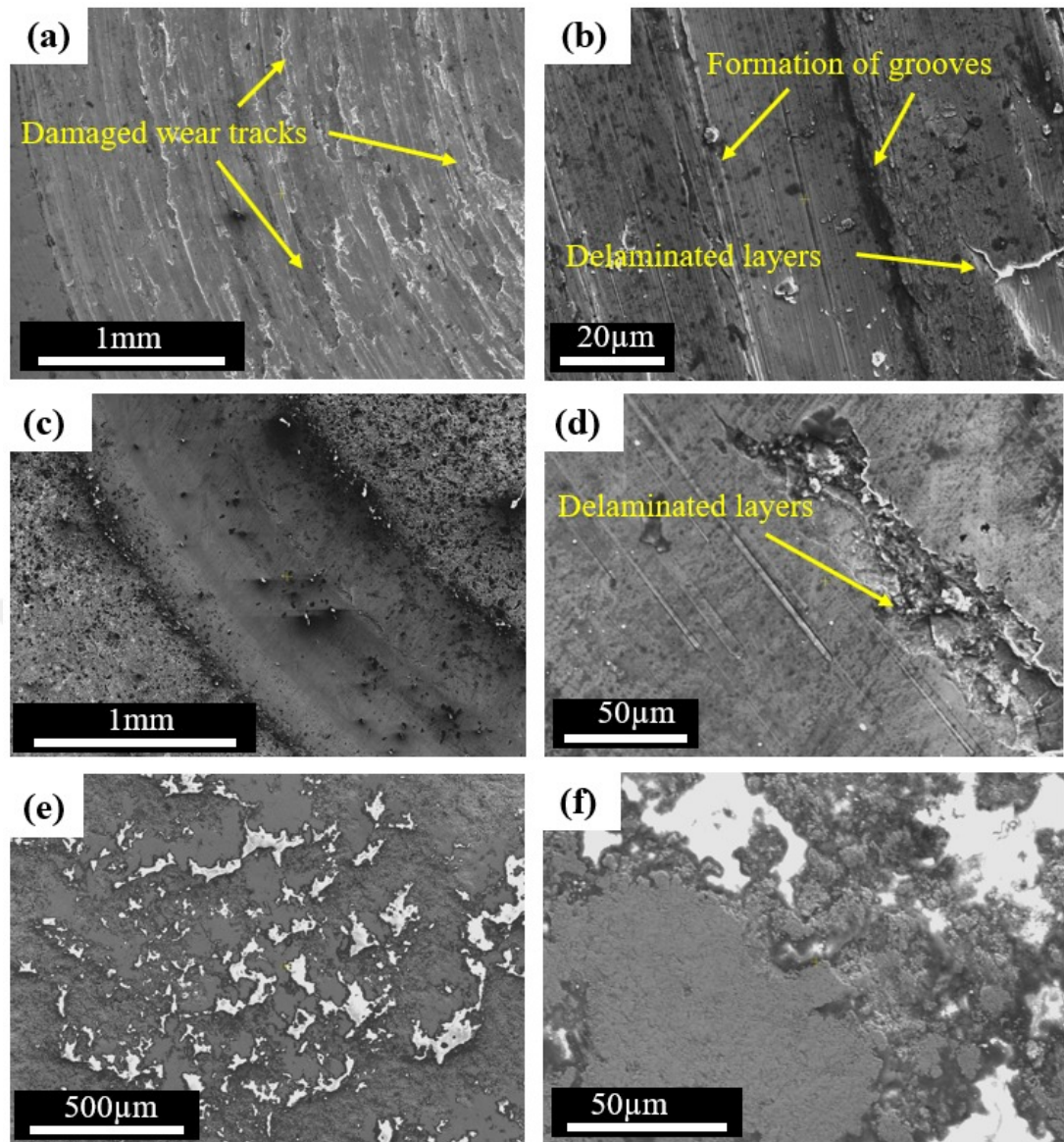


Figure 60: Low and high magnification SEM wear track of Inconel 625 samples at room temperature (a,b) uncoated, (c,d) boronized (e,f) WC coated

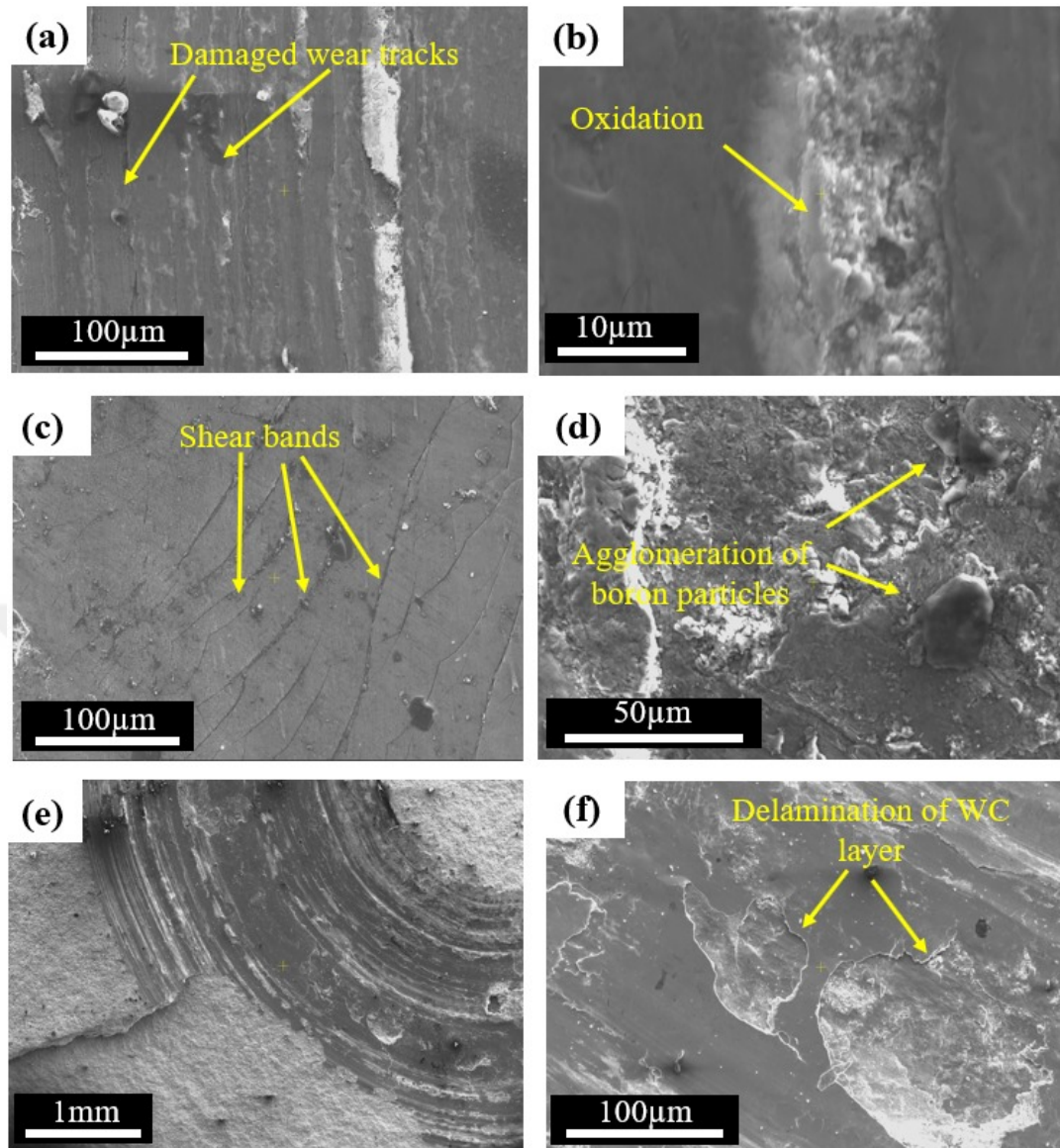


Figure 61: Low and high magnification SEM wear track of Inconel 625 samples at 600°C (a,b) uncoated, (c,d) boronized (e,f) WC coated

Furthermore, with the effect of the high temperature and abrasive wear condition, Alumina ball particles flaked off onto boronized layer (Figure 62). Moreover, delamination of WC layer was detected after wear test due to the softening of inner part and high hardness of WC layer even at high temperature of 600°C (see Figure 61e and f).

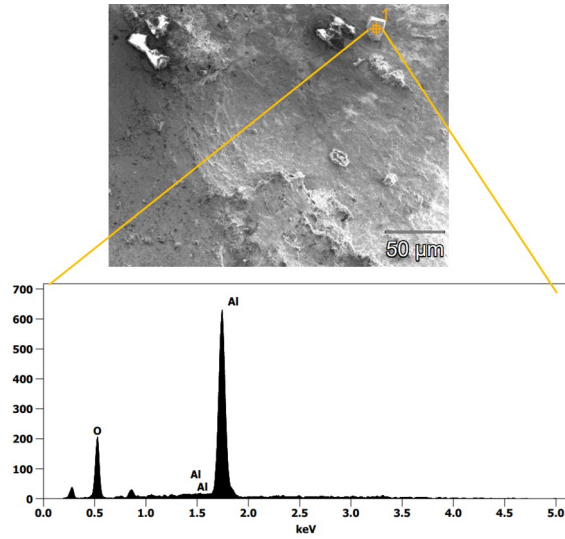


Figure 62: EDX analysis of high temperature tested boronized Inconel 625 sample for spectrum of counterpart (alumina) debris

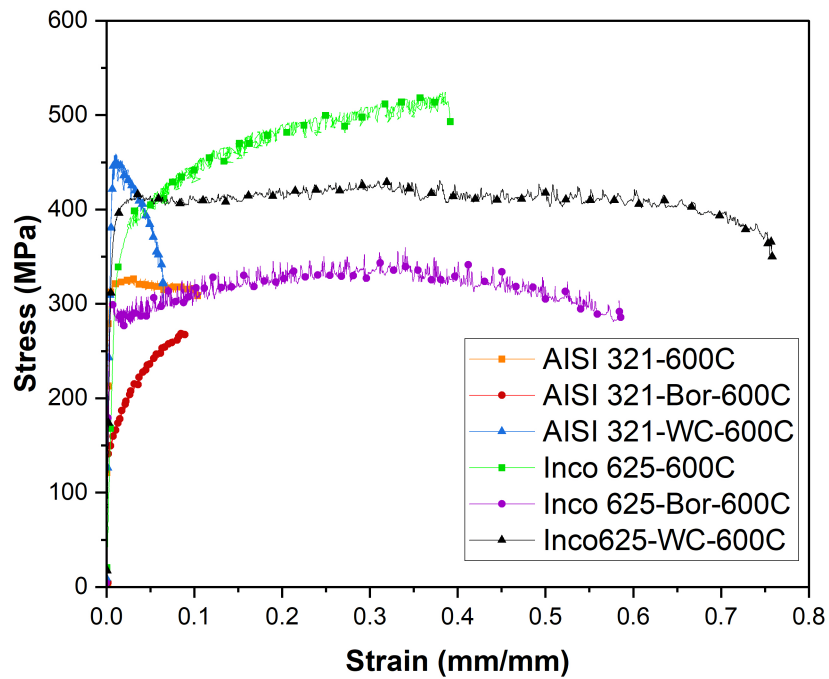


Figure 63: High Temperature Strain-Stress Curves for All AISI 321 and Inconel 625 samples

Table 3: Room temperature tensile results of all AISI 321 and Inconel 625 samples

Material	Yield Strength (MPa)	Tensile Strength (MPa)
AISI 321 as-received	650	723
AISI 321 boronized	280	550
AISI 321 WC Coated	600	610
Inconel 625 as-received	600	980
Inconel 625 boronized	450	730
Inconel 625 WC Coated	700	830

Table 4: High temperature tensile results of all AISI 321 and Inconel 625 samples

Material	Yield Strength (MPa)	Tensile Strength (MPa)
AISI 321 as-received	310	320
AISI 321 boronized	130	250
AISI 321 WC Coated	450	460
Inconel 625 as-received	400	500
Inconel 625 boronized	300	320
Inconel 625 WC Coated	400	410

Figure 63 and Table 4 shows that high temperature strain stress results for all conditions of AISI 321 and Inconel 625. Additionally, room temperature results are given in Table 3 to compare the results easily. YS and UTS of uncoated AISI 321 was 310 and 320 MPa with 10% strain to failure, relatively. For the Inconel 625, these properties were approximately 400 and 500 MPa with 40% elongation. With the boronizing process, YS and UTS values of the boronized 321 sample decreased by about 50% compared to the results at room temperature, to 130 and 250 MPa, respectively. YS and UTS of high temperature boronized Inconel 625 sample were 300 and 320 MPa. The decreasing ratio was same for both materials according to room temperature results. YS of WC coated AISI 321 at high temperature tensile test enhanced to 450 MPa (up to 150 MPa) with the expense of losing ductility to 6%. YS of WC coated Inconel 625 was 400 MPa with the two times higher ductility compared with room temperature, because of the direct effect of the high strength ceramic effect. Moreover, it can be said that bond coating softening can help the increase the ductility. Accordingly, considering both tensile and wear conditions, WC coated Inconel 625 appears to be the best choice for applications at room and 600°C temperature.

CHAPTER V

CONCLUSION AND FUTURE WORKS

In the current thesis the effect of boronizing and WC coating on the room temperature and high temperature wear and tensile behavior of AISI 321 and Inconel 625 samples were investigated using mechanical and fracture morphology observations. The main conclusions of the work are listed as below according to base material.

Specific to AISI 321:

1. Room temperature YS of the boronized AISI 321 was diminished to 280 MPa (56% decrease), due to the softening at high temperature diffusion process. However, dispersion of boron particles improved the work hardening and strain to failure of the sample up to 64%. Additionally, the strain at failure of the WC-coated sample (30%) was lower than the uncoated and boronized conditions, which may be related to the hardness mismatch between the WC layer and the AISI321 layer beneath it. Which is may be a high probability region for crack initiation and rapid fracture.

2. YS of WC coated AISI 321 at high temperature tensile test enhanced to 450 MPa (up to 150 MPa) with the expense of losing ductility to 6%. Strain stress curve of WC coated AISI 321 is typically similar to pure ceramic curve except for the micro-fluctuations. Furthermore, due to the decomposition of boron particles at high temperature, the YS and UTS values of the boronized sample decreased by 50% compared to the results at room temperature, to 130 and 250 MPa, respectively.

3. Fracture surface morphology of the boronized AISI 321 at high temperature, revealed a ductile fracture mode represented by deep dimples and high density of serpentine sliding on the inner walls of the dimples while the WC coated AISI 321

illustrated a brittle fracture with a surface covered by cleavage features and facets.

4. Particle decomposition was observed on the fracture surface of the boronized AISI 321 on the edges of the dimples after failure at high temperature.

5. The results of room temperature wear tests revealed that the friction coefficient of WC coated AISI 321 was higher than that of boronized and uncoated samples due to the high hardness and heat resistance of WC coated layer. At the high temperature wear tests, it was observed that the friction coefficients of the WC coated samples decreased more than the boronized samples. This may be due to the softening of the bond layer due to high temperature.

6. The fracture morphology of worn AISI 321 samples disclosed the formation of cracks and shallow ploughing on the uncoated AISI 321 while the entrapment of boron particles and agglomeration of WC coated layer were detected on the coated samples.

Specific to Inconel 625:

1. The room temperature YS and UTS of the boronized Inconel 625 were reduced significantly due to the grain growth and subsequently softening of the sample throughout the boronizing process at 1100°C for 8 hours. In contrary, WC coated Inconel 625 has high YS and low strain to failure at room temperature due to the fast fracture of outer WC coated layer.

2. High temperature tensile results shows that the ductility of Inconel 625 has been reduced for all conditions in compare with room temperature results. The YS and UTS of the uncoated sample were reduced by about 40% relative to room temperature, down to about 370 and 515 MPa, respectively. The decreasing ratio of the boronized samples is also similar to uncoated condition. YS and UTS of high temperature boronized sample were 300 and 320 MPa, respectively. On the other hand, YS of WC coated Inconel 625 was 400 MPa with the two times higher ductility compared with room temperature, because of the direct effect of the high strength ceramic effect.

3. Portevin-Le Chatelier (PLC) phenomena characterized by serrated yielding

at high temperature was observed in boronized Inconel 625 attributed to dynamic strain aging (DSA).

4. A typical ductile fracture was observed on the fracture surface of the uncoated Inconel 625 sample tested at room temperature while a mixture of relatively brittle fracture mechanism manifested by cleavages on the near surface layer and ductile fracture manner on the core section were detected on the boronized sample.

5. The cleavage and facets representing brittle fracture were covered the surface of WC coated Inconel 625 sample tested at room and high temperature. This is attributed to dominated effect of hard WC particles on the outside region of the samples.

6. The friction coefficient of boronized Inconel 625 was higher than of the WC coated sample at high temperature. Also, the fluctuation was observed which is related to agglomeration of boron particles on the surface at high temperature.

To sum up, two different coating methods utilized on two different base materials at room and high temperature applications. The results and effectiveness of coating methods should be investigated in case of tensile and wear performance at the room and high temperatures, separately.

The AISI 321 room temperature tensile results show that, both of coating methods negatively effected the YS and UTS, but boronizing can be increase the ductility of material. For high temperature tensile results of AISI 321 show that WC coating can be increase the bulk properties of material. On the other hand, at room temperature, boronizing and WC coating can be extremely increase the wear resistance of material. But at the high temperature, WC coating is more effective coating method.

Inconel 625 room temperature tensile results shows that, uncoated condition can be the best choice at room temperature in tensile condition. At high temperature, uncoated version exhibits good bulk properties but WC coating can be increase the ductility. At room temperature wear condition, similar with AISI 321, boronizing and WC coating can be very good coating methods for the Inconel 625.

At the high temperature, it has been observed that the WC coating maintains its performance even under long-term wear condition.

For the future works, the effect of other coating methods on the bulk mechanical properties of AISI 321 and Inconel 625 can be investigated in detail such as elasticity and micro-hardness distribution. Furthermore, the other bulk mechanical properties such as compressive strength, impact, and low and high cycle fatigue can be examined. In addition, the results of experimental studies can be validated in numerical studies and then in simulation software, saving time and cost and being widely used in the industry.



REFERENCES

- [1] A. Calik, F. Taylan, and O. S. B. . N. Ucar, “Comparison of mechanical properties of boronized and vanadium carbide coated aisi 1040 steels,” 2009.
- [2] A. Gopkalo and A. Rutkovskyy, “The effect of pvd coatings on the tensile strength and low-cycle fatigue resistance of stainless steel and titanium alloys,” *Fatigue & Fracture of Engineering Materials & Structures*, vol. 34, no. 12, pp. 1012–1020, 2011.
- [3] B. Saini and V. Gupta, “Effect of wc/c pvd coating on fatigue behaviour of case carburized sae8620 steel,” *Surface and Coatings Technology*, vol. 205, pp. 511–518, 10 2010.
- [4] W. Li, H. Chen, C. Li, W. Huang, J. Chen, L. Zuo, Y. Ren, J. He, and S. Zhang, “Microstructure and tensile properties of aisi 321 stainless steel with aluminizing and annealing treatment,” *Materials & Design*, vol. 205, p. 109729, 2021.
- [5] I. O. Tugay, A. Hosseinzadeh, and G. G. Yapici, “Hardness and wear resistance of roller burnished 316l stainless steel,” *Materials Today: Proceedings*, vol. 47, pp. 2405–2409, 2021.
- [6] H. M. Tehrani, R. Shoja-Razavi, M. Erfanmanesh, S. H. Hashemi, and M. Barekat, “Evaluation of the mechanical properties of wc-ni composite coating on an aisi 321 steel substrate,” *Optics & Laser Technology*, vol. 127, p. 106138, 2020.
- [7] J.-S. Xu, X.-C. Zhang, F.-Z. Xuan, F.-Q. Tian, Z.-D. Wang, and S.-T. Tu, “Tensile properties and fracture behavior of laser cladde wc/ni composite coatings with different contents of wc particle studied by in-situ tensile testing,” *Materials Science and Engineering: A*, vol. 560, pp. 744–751, 2013.
- [8] C. Leyens and M. Peters, *Titanium an Titanium Alloys*. 2003.
- [9] H. Dinç, *Inconel 718 süperalaşımının termokimyasal borlanması*. PhD thesis, Fen Bilimleri Enstitüsü, 2013.
- [10] J. D. WHITTENBERGER, “A review of: “superalloys ii” edited by ct. sims, n.s. stoloff, and w.c. hagel a wiley-interscience publication john wiley sons, new york, ny 615 pages, hardcover, 1987,” *Materials and Manufacturing Processes*, vol. 7, pp. 463–468, 1992.
- [11] X. F. Yuan, J. X. Song, Y. R. Zheng, Q. Huang, K. Yagi, C. B. Xiao, and Q. Feng, “Quantitative microstructural evolution and corresponding stress rupture property of k465 superalloy,” *Materials Science and Engineering: A*, vol. 651, pp. 734–744, 2016.

- [12] X. Ren, F. Wang, and X. Wang, "High-temperature oxidation and hot corrosion behaviors of the nicr-cral coating on a nickel-based superalloy," *Surface and Coatings Technology*, vol. 198, pp. 425–431, 8 2005.
- [13] P. M. Rogers, I. M. Hutchings, and J. A. Little, "Coatings and surface treatments for protection against low-velocity erosion-corrosion in fluidized beds," *Wear*, vol. 186, pp. 238–246, 1995.
- [14] R. C. Reed, *The superalloys: fundamentals and applications*. Cambridge university press, 2008.
- [15] Y. Gao, Y. Ding, J. Chen, J. Xu, Y. Ma, and X. Wang, "Effect of twin boundaries on the microstructure and mechanical properties of inconel 625 alloy," *Materials Science and Engineering: A*, vol. 767, p. 138361, 11 2019.
- [16] R. R. Kumar and K. M. Pandey, "Static structural and modal analysis of gas turbine blade," *IOP Conference Series: Materials Science and Engineering*, vol. 225, p. 012102, 8 2017.
- [17] H. M. Cobb, "The naming and numbering of stainless steels: the variety of numbering and naming systems for stainless steels is based on the fact that the alloys were developed by companies all over the world," *Advanced Materials & Processes*, vol. 165, no. 9, pp. 39–45, 2007.
- [18] B. M. Tordoir and E. J. Moeys, "The treatment of certain types of fractures: With a v2a steel nail in the medullary cavity (the kÜntscher method)," *Journal of the American Medical Association*, vol. 128, pp. 792–794, 1945.
- [19] H. Li, S. Yang, S. Zhang, B. Zhang, Z. Jiang, H. Feng, P. Han, and J. Li, "Microstructure evolution and mechanical properties of friction stir welding super-austenitic stainless steel s32654," *Materials Design*, vol. C, pp. 207–217, 3 2017.
- [20] H. Khatak and B. Raj, *Corrosion of austenitic stainless steels: mechanism, mitigation and monitoring*. Woodhead publishing, 2002.
- [21] W.-B. Lee and S.-B. Jung, "Effect of microstructure on mechanical properties of friction-welded joints between ti and aisi 321 stainless steel," *Materials transactions*, vol. 45, pp. 2805–2811, 2004.
- [22] S. Pour-Ali, M. Weiser, N. T. Nguyen, A.-R. Kiani-Rashid, A. Babakhani, and S. Virtanen, "High temperature oxidation behaviour of aisi 321 stainless steel with an ultrafine-grained surface at 800° c in ar-20 vol.% o2," *Corrosion Science*, vol. 163, p. 108282, 2020.
- [23] I. Hutchings and P. Shipway, *Tribology: Friction and wear of engineering materials: Second Edition*. 2017.
- [24] J. S. Selvan, K. Subramanian, and A. K. Nath, "Effect of laser surface hardening on en18 (aisi 5135) steel," *Journal of Materials Processing Technology*, vol. 91, pp. 29–36, 1999.

- [25] R. Carrera-Espinoza, U. Figueroa-López, J. Martínez-Trinidad, I. Campos-Silva, E. Hernández-Sánchez, and A. Motalebzadeh, “Tribological behavior of borided aisi 1018 steel under linear reciprocating sliding conditions,” *Wear*, vol. 362-363, pp. 1–7, 9 2016.
- [26] W. Fichtl, “Boronizing and its practical applications,” *Materials Design*, vol. 2, pp. 276–286, 12 1981.
- [27] R. S. Petrova, N. Suwattananont, and V. Samardzic, “The effect of boronizing on metallic alloys for automotive applications,” *Journal of Materials Engineering and Performance*, vol. 17, pp. 340–345, 6 2008.
- [28] D. N. Tsipas and J. Rus, “Boronizing of alloy steels,” 1987.
- [29] N. Kiratli and F. Findik, “Research on wear characteristics of aisi 1035 steel boronized at various parameters,” *Industrial Lubrication and Tribology*, vol. 63, pp. 127–133, 3 2011.
- [30] M. Ulutan, M. M. Yildirim, O. N. Çelik, and S. Buytoz, “Tribological properties of borided aisi 4140 steel with the powder pack-boriding method,” *Tribology letters*, vol. 38, no. 3, pp. 231–239, 2010.
- [31] T. Schopphoven, A. Gasser, K. Wissenbach, and R. Poprawe, “Investigations on ultra-high-speed laser material deposition as alternative for hard chrome plating and thermal spraying,” *Journal of Laser Applications*, vol. 28, p. 022501, 2016.
- [32] A. Ibrahim and C. C. Berndt, “Fatigue and deformation of hvof sprayed wc-co coatings and hard chrome plating,” *Materials Science and Engineering: A*, vol. 456, pp. 114–119, 2007.
- [33] K. Holmberg and A. Matthews, *Coatings tribology: properties, mechanisms, techniques and applications in surface engineering*. Elsevier, 2009.
- [34] H. Holleck, , and V. Schier, “Multilayer pvd coatings for wear protection,” *Surface and Coatings Technology*, vol. 76, pp. 328–336, 1995.
- [35] R. B. Heimann, *Plasma-spray coating: principles and applications*. John Wiley Sons, 2008.
- [36] B. Bhushan, *Modern Tribology Handbook, Two Volume Set*. CRC Press, 12 2000.
- [37] M. Nosonovsky and V. Mortazavi, *Friction-induced vibrations and self-organization: mechanics and non-equilibrium thermodynamics of sliding contact*. CRC Press, 2013.
- [38] Y. Wang, S. Zhao, Z. Jia, J. Ji, D. Liu, T. Guo, and Y. Ding, “Study on friction and wear behavior of inconel 625 superalloy during hot extrusion,” *Advances in Materials Science and Engineering*, vol. 2020, 2020.

- [39] K. Thirugnanasambantham and S. Natarajan, "Mechanistic studies on degradation in sliding wear behavior of in718 and hastelloy x superalloys at 500° c," *Tribology International*, vol. 101, pp. 324–330, 2016.
- [40] G. E. Totten, Batchelor, *et al.*, "Asm handbook w," *Materials Park, Ohio*, pp. 44073–0002, 2017.
- [41] P. Olander and S. Jacobson, "Scuffing resistance testing of piston ring materials for marine two-stroke diesel engines and mapping of the operating mechanisms," *Wear*, vol. 330-331, pp. 42–48, 2015.
- [42] M. F. Jensen, J. Bøttiger, H. H. Reitz, and M. E. Benzon, "Simulation of wear characteristics of engine cylinders," *Wear*, vol. 253, pp. 1044–1056, 2002.
- [43] S. Wan, D. Li, G. Zhang, A. K. Tieu, and B. Zhang, "Comparison of the scuffing behaviour and wear resistance of candidate engineered coatings for automotive piston rings," *Tribology International*, vol. 106, pp. 10–22, 2017.
- [44] A. Cameron, "Principles of tribology: Edited by prof. s. halling published by macmillan press ltd, london," *Tribology International*, vol. 8, p. 229, 1975.
- [45] D. R. Jones and M. F. Ashby, *Engineering materials 1: An introduction to properties, applications and design*. Butterworth-Heinemann, 2018.
- [46] J. Stokes, "The theory and application of the hvof thermal spray process," 2008.
- [47] S. K. Mishra, S. Biswas, and A. Satapathy, "A study on processing, characterization and erosion wear behavior of silicon carbide particle filled za-27 metal matrix composites," *Materials Design*, vol. 55, pp. 958–965, 2014.
- [48] C. Azevedo and A. Sinátorá, "Erosion-fatigue of steam turbine blades," *Engineering Failure Analysis*, vol. 16, no. 7, pp. 2290–2303, 2009.
- [49] L. A. Sosnovskiy, *Tribo-fatigue: wear-fatigue damage and its prediction*. Springer Science Business Media, 2010.
- [50] K. C. Radil, "Test method to evaluate cylinder liner-piston ring coatings for advanced heat engines.," 1997.
- [51] H. Güleriyüz and H. Çimenoglu, "Effect of thermal oxidation on corrosion and corrosion–wear behaviour of a ti–6al–4v alloy," *Biomaterials*, vol. 25, pp. 3325–3333, 2004.
- [52] E. Rezaei, "A new model for the optimization of periodic inspection intervals with failure interaction: A case study for a turbine rotor," *Case Studies in Engineering Failure Analysis*, vol. 9, pp. 148–156, 10 2017.
- [53] A. Molinari and M. Nouari, "Modeling of tool wear by diffusion in metal cutting," *Wear*, vol. 252, pp. 135–149, 2002.
- [54] R. B. Waterhouse, "Fretting wear," *Wear*, vol. 100, pp. 107–118, 1984.

- [55] J. A. McGeehan, E. S. Yamaguchi, and J. Q. Adams, “Some effects of zinc dithiophosphates and detergents on controlling engine wear,” *SAE transactions*, pp. 879–892, 1985.
- [56] N. P. Suh, “An overview of the delamination theory of wear,” *Wear*, vol. 44, pp. 1–16, 1977.
- [57] H. Hertz, “Über die berührung fester elastischer körper,” *Journal für die reine und angewandte Mathematik*, vol. 92, no. 156-171, p. 22, 1882.
- [58] A. C. Fischer-Cripps, *Introduction to contact mechanics*, vol. 101. Springer, 2007.
- [59] K. Skalski, A. Morawski, and W. Przybylski, “Analysis of contact elastic-plastic strains during the process of burnishing,” *International journal of mechanical sciences*, vol. 37, no. 5, pp. 461–472, 1995.
- [60] J. E. Shigley, *Shigley’s mechanical engineering design*. Tata McGraw-Hill Education, 2011.
- [61] Y. ALEMDAĞ, G. PÜRÇEK, and M. BEDER, “Effect of age hardening on the wear behaviour of the al-7si-4zn-3cu alloy,”
- [62] I. Hutchings and P. Shipway, *Tribology: friction and wear of engineering materials*. Butterworth-Heinemann, 2017.
- [63] L. Dan and J. Mathew, “Tool wear and failure monitoring techniques for turning—a review,” *International Journal of Machine Tools and Manufacture*, vol. 30, no. 4, pp. 579–598, 1990.
- [64] Ç. Albayrak, İ. Hacısalihoğlu, A. Alsaran, *et al.*, “Tribocorrosion behavior of duplex treated pure titanium in simulated body fluid,” *Wear*, vol. 302, no. 1-2, pp. 1642–1648, 2013.
- [65] B. Mordyuk, G. Prokopenko, M. Vasylyev, and M. Iefimov, “Effect of structure evolution induced by ultrasonic peening on the corrosion behavior of aisi-321 stainless steel,” *Materials Science and Engineering: A*, vol. 458, pp. 253–261, 6 2007.
- [66] M. S. Ghazani and B. Eghbali, “Characterization of the hot deformation microstructure of aisi 321 austenitic stainless steel,” *Materials Science and Engineering: A*, vol. 730, pp. 380–390, 7 2018.
- [67] A. Hosseinzadeh and G. G. Yapici, “On the high-temperature flow response of friction stir processed magnesium metal matrix composites,” *Journal of Engineering Materials and Technology*, vol. 143, 1 2021.
- [68] A. Hosseinzadeh and G. G. Yapici, “High temperature characteristics of al2024/sic metal matrix composite fabricated by friction stir processing,” *Materials Science and Engineering: A*, vol. 731, pp. 487–494, 7 2018.

- [69] U. Sen and S. Sen, “The fracture toughness of borides formed on boronized cold work tool steels,” *Materials Characterization*, vol. 50, pp. 261–267, 6 2003.
- [70] J.-S. Xu, X.-C. Zhang, F.-Z. Xuan, F.-Q. Tian, Z.-D. Wang, and S.-T. Tu, “Tensile properties and fracture behavior of laser cladded wc/ni composite coatings with different contents of wc particle studied by in-situ tensile testing,” *Materials Science and Engineering: A*, vol. 560, pp. 744–751, 1 2013.
- [71] K. Feng, Y. Chen, P. Deng, Y. Li, H. Zhao, F. Lu, R. Li, J. Huang, and Z. Li, “Improved high-temperature hardness and wear resistance of inconel 625 coatings fabricated by laser cladding,” *Journal of Materials Processing Technology*, vol. 243, pp. 82–91, 2017.
- [72] H. Attar, K. Prashanth, A. Chaubey, M. Calin, L. C. Zhang, S. Scudino, and J. Eckert, “Comparison of wear properties of commercially pure titanium prepared by selective laser melting and casting processes,” *Materials Letters*, vol. 142, pp. 38–41, 2015.
- [73] F. Jiang, S. Kexing, and L. Shuhua, “Design and research progress of guoxiuhua hybrid reinforced copper matrix composites,” *Journal of Materials Heat Treatment*, vol. 39, pp. 1–9, 2018.
- [74] H. Bai, L. Zhong, L. Kang, J. Liu, W. Zhuang, Z. Lv, and Y. Xu, “A review on wear-resistant coating with high hardness and high toughness on the surface of titanium alloy,” *Journal of Alloys and Compounds*, vol. 882, p. 160645, 11 2021.
- [75] L. Qian, S. Zhu, Y. Kagawa, and T. Kubo, “Tensile damage evolution behavior in plasma-sprayed thermal barrier coating system,” *Surface and Coatings Technology*, vol. 173, no. 2, pp. 178–184, 2003.
- [76] C. Tian, C. Cui, L. Xu, Y. Gu, and X. Sun, “Dynamic strain aging in a newly developed ni-co-base superalloy with low stacking fault energy,” *Journal of Materials Science Technology*, vol. 29, pp. 873–878, 9 2013.
- [77] P. Maj, J. Zdunek, J. Mizera, K. J. Kurzydowski, B. Sakowicz, and M. Kaminski, “Microstructure and strain-stress analysis of the dynamic strain aging in inconel 625 at high temperature,” *Metals and Materials International*, vol. 23, pp. 54–67, 1 2017.
- [78] A. Chatterjee, G. Sharma, R. Tewari, and J. K. Chakravartty, “Investigation of the dynamic strain aging and mechanical properties in alloy-625 with different microstructures,” *Metallurgical and Materials Transactions A*, vol. 46, pp. 1097–1107, 3 2015.
- [79] M. M. De Oliveira, A. A. Couto, G. F. Almeida, D. A. Reis, N. B. De Lima, and R. Baldan, “Mechanical behavior of inconel 625 at elevated temperatures,” *Metals*, vol. 9, no. 3, p. 301, 2019.
- [80] K. Shojaei, S. Sajadifar, and G. Yapici, “On the mechanical behavior of cold deformed aluminum 7075 alloy at elevated temperatures,” *Materials Science and Engineering: A*, vol. 670, pp. 81–89, 7 2016.

- [81] A. Calik, N. Ucar, K. Delikanli, M. Carkci, and . S. Karakas, “Boriding kinetics and mechanical properties of borided commercial-purity nickel,” 2017.
- [82] P. J. Blau, “The significance and use of the friction coefficient,” *Tribology International*, vol. 34, no. 9, pp. 585–591, 2001.
- [83] P. J. Blau, “Four great challenges confronting our understanding and modeling of sliding friction,” in *Tribology Series*, vol. 34, pp. 117–128, Elsevier, 1998.
- [84] G. Ostermeyer, “On the dynamics of the friction coefficient,” *Wear*, vol. 254, no. 9, pp. 852–858, 2003.
- [85] A. Piasecki, M. Kotkowiak, N. Makuch, and M. Kulka, “Wear behavior of self-lubricating boride layers produced on inconel 600-alloy by laser alloying,” *Wear*, vol. 426, pp. 919–933, 2019.
- [86] J. D. Bressan, G. A. Battiston, R. Gerbasi, D. P. Daros, and L. M. Gilapa, “Wear on tool steel aisi m2, d6 and 52100 coated with al₂o₃ by the mocvd process,” *Journal of materials processing technology*, vol. 179, no. 1-3, pp. 81–86, 2006.
- [87] B. Kurt, Y. Küçük, and M. Sabri Gök, “Microabrasion wear behavior of vc and crc coatings deposited by thermoreactive diffusion technique,” *Tribology Transactions*, vol. 57, no. 2, pp. 345–352, 2014.
- [88] A. Günen, “Properties and high temperature dry sliding wear behavior of boronized inconel 718,” *Metallurgical and Materials Transactions A*, vol. 51, no. 2, pp. 927–939, 2020.