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OPTIMISATION OF ANNEALING PARAMETERS AND IMPROVING DIE LIFE FOR NITRIDED ALUMINUM EXTRUSION DIES

M. Sc. Thesis By

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OPTIMISATION OF ANNEALING PARAMETERS AND IMPROVING DIE LIFE FOR NITRIDED ALUMINUM EXTRUSION DIES

ABSTRACT

Aluminum extrusion dies are made of hot work tool steels. After vacuum hardening, dies are processed to a gas nitriding process to improve surface hardness and wear resistance. Before the extrusion process, all dies are annealed at 450°C to reach billet temperature in the furnace. During the annealing process in an air furnace, the nitride layers (white and diffusion layer) oxidize. The amount of oxidation increases as the holding time is increased. The nitride layer is damaged by oxidation, which causes a rough surface and cracks the nitride layer. Microcracks appear on the surface in this case as the aluminum profile slides over it during the extrusion process. Microcracks spread from the surface to the core, causing flaking on the steel. Die surfaces are damaged in this case, and die life are finished.

In this study, specimens were annealed in an air furnace at 450°C for 6,8,12,15,20,25,30,40 hours. As a result, the amount of oxidation was analyzed using an optical and electron microscope, as well as how the white layer was affected by annealing time in an air furnace. Furthermore, two specimens were annealed at 450°C for 6 and 12 hours in an inert furnace (insulated from oxygen). The nitride layers of the two specimens were also studied using an optical and electron microscope. In this case, the oxidation of nitride layers was compared using an air furnace and an inert furnace. Abrasion test was performed on two specimens to determine the white layer's abrasion resistance. The first specimen had a white layer, while the second specimen did not (only have diffusion layer).

Even when nitrided specimens are annealed for 6 hours in an air furnace, an oxide layer forms on the white layer and remains stable. The thickness of the white layer begins to reduce after 12 hours of annealing, but the white layer is still available at the conclusion of 40 hours. Furthermore, after 14 hours, the thickness of the diffusion layer begins to increase. Specimens that anneal for 6 to 12 hours in an inert furnace, on the other hand, are not damaged and do not oxidize as much as those that anneal in an air furnace. When examined under an optical microscope, the nitride layers are same. In an optical microscope, the oxide layers of specimens that anneal in an air furnace at the same time are clearly visible. In an inert furnace, only 5% of the oxide components are present, but this value is 20% for annealed specimens in an air furnace. Specimens with white layers abrade less than specimens without white layers.

As a consequence of this research, inert furnaces are preferred over air furnaces for nitride layer oxidation during annealing before to extrusion. If an air furnace is the only option, the annealing process must be stopped when the die reaches the desired temperature. In this situation, the nitrided layer will be less damaged, resulting in longer die lifetimes. Even though the white layer is brittle as a result of the abrasion test, it is suitable for extrusion dies.

Keywords: Oxidation of nitrided layer, annealed of extrusion dies, holding time of annealing, abrassion test of nitride layer.

OPTIMISATION OF ANNEALING PARAMETERS AND IMPROVING DIE LIFE FOR NITRIDED ALUMINUM EXTRUSION DIES

ÖZET

Alüminyum ekstrüzyon kalıplarında sıcak iş takım çeliği kullanılmaktadır. Yüzey sertliğini ve aşınma direncini artırmak için vakum sertleştirme prosesi sonrası gas nitrasyon prosesi yapılmaktadır. Bütün kalıplar biyet sıcaklığına ön ısıtma fırınında üretim öncesinde ısıtılmaktadır (450 °C). Kalıpların nitrasyon tabakası atmosfer ön ısıtma fırınlarında ısıtma esnasında oksitlenmektedir. Ön ısıtma süresindeki artış, oksidasyon miktarını artırmaktadır. Oksidasyon tabakası nitrasyon tabakasına zarar vererek yüzeyi pürüzlü hale getirmekte ve nitrasyon tabakasının çatlamasına sebep olabilmektedir. Bu durumda üretim esnasında alüminyum kalıp yüzeyinden geçerken yüzeyde mikro çatlak meydana getirebiliyor. Mikro çatlaklar yüzeyden çelik merkezine doğru ilerleyerek çelikten parça kopmasına (pullanma) sebep vermektedir. Bu durumda, zarar gören kalıp yüzeyleri tolerans dışı olduğunda hurdalanmaktadır.

Bu çalışmada; nitrasyon prosesi uygulanmış dokuz numune 6,8,12,15,20,25,30,40 saat 450 °C ön ısıtma fırınında tutulmuştur. Nitrasyon tabakasında oluşan oksidasyon miktarı ve tutma süresinin nitrasyon tabakasına etkisi mikroskop altında incelenmiştir. İki adet numune de korumalı fırında 6 ve 12 saat ön ısıtma yapılarak nitrasyon tabakası gözlemlenerek, korumalı ve korumasız ön ısıtma fırınlarının nitrasyon tabakasına etkisi karşılaştırılmıştır. Nitrasyon tabakasında bulunan beyaz tabakanın aşınma dayanımına katkısı incelenmek için beyaz tabakalı ve beyaz tabakasız olmak üzere 2 numuneye aşınma testi uygulanmıştır.

Korumasız fırında kalıplar 6 saat tutulsa bile, beyaz tabakanın kararlı şekilde oksitlendiği gözlemlenmiştir. 12 saat tutma süresinden sonar beyaz tabaka kalınlığı düşmeye başladı fakat 40 saatin sonunda beyaz tabaka tamamen kaybolmadı. Difüzyon tabakasının 14 saatten sonra arttığı gözlemlenmiştir. Diğer bir taraftan; korumalı ön ısıtma fırınında 6 ve 12 saat tutulan numuneler, korumasız ön ısıtma fırınındaki gibi bozulmamıştır. Optik mikroskopta bakıldığında korumalı atmosferdeki nitrasyon numunesinde bozulma gözlenmemiştir. Elektron mikroskop ile gözlemlendiğinde korumasız atmosferdeki nitrasyon tabakasında ağırlıkça %20 oksit elementine rastlanırken, korumalı atmosferde en fazla ağırlıkça %5 oksit elementi gözlemlenmiştir. Aşınma testi uygulanan 2 numunenin beyaz tabakaya sahip olanının daha az aşındığı gözlenmiştir.

Çalışmanın sonucunda; korumalı atmosferdeki ön ısıtma fırını, korumasız ön ısıtma fırınına göre nitrasyon tabakasına çok daha az zarar verdiği gözlemlenmiştir. Korumasız atmosfer ön ısıtma fırını tek seçenek olduğu durumlarda, 6 saatte bile oksitlenebildiği için kalıp istenen sıcaklığa geldikten sonra hemen fırından çıkartılmalıdır. Önerilere dikkat edildiği taktirde nitrasyon tabakası daha az zarar görerek kalıp ömürleri artacaktır. Aşınma testi sonucunda beyaz tabaka gevrek bir yapıya sahip olmasına rağmen ekstrüzyon kalıplarının geçiş yüzeylerinde aşınma dayanımını artırdığı gözlemlenmiştir.

Anahtar sözcükler: Nitrasyon tabakasının oksitlenmesi, Ekstrüzyon kalıplarının ön ısıtması, ekstrüzyon kalıplarının ön ısıtma süresi

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CHAPTER 1

INTRODUCTION

Extrusion dies, which are often made of tool steels H10, H13, and H11, are subjected to extreme mechanical, thermal, tribological, and chemical loads. [1]. Extrusion dies are made entirely of H13 tool steel. It has a high strength, ductility, and tempering resistance, as well as a moderate cost. After quenching and tempering, the steel is used at hardness values ranging from 450 to 500 HV. It's also suitable for nitriding, which is a surface hardening process. [2].

Extrusion die wear has a significant impact on die life and extruded aluminum surface finish. The most frequent surface treatment for extrusion dies is gas nitriding. This process extends the life of the dies and protects the bearing surfaces. The gas nitriding process includes the diffusion of nitrogen atoms through the surface at temperatures ranging from 450 to 580 °C. This treatment results in a nitride layer of 50–300 microns and a compound layer of 2–10 microns (iron nitride). The hardness of the surface is measured at 1000-1200 HV. Epsilon phase, gamma phase, and mix phase all contribute to the white layer. In the case of hot extrusion dies, a white layer is helpful in terms of wear and corrosion resistance. [2][3].

Aluminum billets and dies are heated between 450 and 520 °C throughout the extrusion process to avoid large temperature gradients. In most cases, dies are annealed in an air furnace. In these furnaces, dies are annealed for 4 to 12 hours, depending on their thickness. During annealing, the white layer on the surface oxidizes, forming an oxidized layer. During the extrusion process, the oxidation layer may break away, leaving a mark on the extrusion's bearing surface. As a result, oxidation causes damage to the white layer. In an investigation performed in 2009, dies were annealed for 4, 16, and 30 hours in air and nitrogen furnaces to observe solutions. To begin, the specimen is heated to 450°C in an air atmosphere for 4 hours, at which point it oxidizes. Only traces of gamma nitrides remain after changing the annealing period to 16/32 hours.

After 4 hours in a nitrogen atmosphere, the white layer oxidized, but not as much as the specimen that had been annealed for 4 hours in an air furnace. Even after 16 hours of annealing, a significant portion of the nitrided layer disappears. The nitride layer almost oxidizes after 30 hours. Finally, a nitrogen furnace is not a complete solution; it just slows oxidation. [4].

The goal of this research is to figure out how to get the best annealing time in a annealing furnace and how to protect the nitriding layer from oxidizing. Nitrided specimens were annealed for 6 hours, 12 hours, 15 hours, 20 hours, 25 hours, 30 hours, and 40 hours. We see how oxidation occurs and grows as the annealing time increases. Furthermore, the specimens are annealed 6 and 12 hours in the nitrogen atmosphere that surrounds the furnace, and oxidation is observed. Abrasion resistance white layer is obtained by performing a pin on plate abrasion test on two specimens, the first of which has a white layer and the second of which does not (only has diffusion layer). The abrasion parameters are forced at 15N, and the sliding distances are 100 and 200 meters. [5].

The importance of this research is that oxidation of the nitriding layer is limited or prevented, die life is extended, die surface is not easily disturbed, and die annealing time is arranged.

CHAPTER 2

LITERATURE REVIEW

2.1 Hot Work Steels

Marcus Grossman and Edgar Bain, in their works published in 1930, learned that tool steels had superior quality attributes compared to other steels. Electric field melting procedures were discovered in powder metallurgy that year, and these processes helped in the development of tool steels with important qualities. Due to their high working temperature, hot work steels are classified as H group steels in the AISI classification system. Deformation resistance at working temperatures, mechanical resistance, thermal resistance, wear resistance, and machinability are the most important properties related to alloys such as chromium, molybdenum, vanadium, and silicon. [4]. Hot work steels are subdivided in three classes and compositions are shown in Table 2.1. In addition, thermal treatment, thermal conductivity, density and elasticity modulus are shown in Table 2.2, Table 2.3, Table 2.4, Table 2.5.

2.1.1 Chromium Hot Work Steels

The amount of chromium in a tool steel affects its properties. High hardenability, toughness, ductility, and wear resistance are the main characteristics. Extrusion tools, drawing punches, and bolt header dies are all frequent uses for chromium tool steels. Chromium tool steels maintain their mechanical characteristics at temperatures up to 538 °C.

2.1.2 Tungsten Hot Work Steels

The main alloy in these tool steels is tungsten, with a low carbon content. The resistance of tungsten alloy to softening at high temperatures is increased. The wear resistance of this alloy is increased. The use of low-carbon steel increases the steel's toughness. Tungsten tool steel has a lower thermal shock resistance than chromium tool steel. Forging tools, extrusion brass, shear blades, and dummy blocks are all frequent uses for tungsten tool steel.

2.1.3 Molybdenum Hot Work Steels

When compared to tungsten tool steel, the main alloy is molybdenum. That steel is preferred because it has excellent softening resistance and high wear resistance. Special claims are made for molybdenum steels. Punches, die inserts, and hot headers are all made with them. [4], [6].

Table 2.1 Composition limits for hot-work tool steels [4]

AISI TYPE	**UNS NO**	**Composition, %**								
		C	**Mn**	**Si**	**Cr**	**Ni**	**Mo**	**W**	**V**	**Co**
Chromium hot-work steels										
H10	T20810	0,35-0,45	0,25-0,70	0,80-1,20	3,00-3,75	0,3 max	2,00-3,00	…	0,25-0,75	…
H11	T20811	0,33-0,43	0,20-0,50	0,80-1,20	4,75-5,50	0,3 max	1,10-1,60	…	0,30-0,60	…
H12	T20812	0,30-0,40	0,20-0,50	0,80-1,20	4,75-5,50	0,3 max	1,25-1,75	1,00-1,70	0,50 max	…
H13	T20813	0,32-0,45	0,20-0,50	0,80-1,20	4,75-5,50	0,3 max	1,10-1,75	…	0,80-1,20	…
H14	T20814	0,35-0,45	0,20-0,50	0,80-1,20	4,75-5,50	0,3 max	…	4,00-5,25	…	…
H19	T20819	0,35-0,45	0,20-0,50	0,20-0,50	4,00-4,75	0,3 max	0,30-0,55	3,75-4,50	1,75-2,00	4,00-4,50
Tungsten hot-work steels										
H21	T20821	0,26-0,36	0,15-0,40	0,15-0,50	3,00-3,75	0,3 max	…	8,5-10,00	0,30-0,60	…
H22	T20822	0,30-0,40	0,15-0,40	0,15-0,40	1,75-3,75	0,3 max	…	10,00-11,75	0,25-0,50	…
H23	T20823	0,25-0,35	0,15-0,40	0,15-0,60	11,00-12,75	0,3 max	…	11,00-12,75	0,75-1,25	…
H24	T20824	0,42-0,53	0,15-0,40	0,15-0,40	2,50-3,50	0,3 max	…	14,00-16,00	0,40-0,60	…
H25	T20825	0,22-0,32	0,15-0,40	0,15-0,40	3,75-4,50	0,3 max	…	14,00-16,00	0,40-0,60	…
H26	T20826	0,45-0,55(b)	0,15-0,40	0,15-0,40	3,75-4,50	0,3 max	…	17,25-19,00	0,75-1,25	…
Molybdenum hot-work steels										
H42	T20842	0,55-0,70(b)	0,15-0,40	…	3,75-4,50	0,3 max	4,50-5,50	5,50-6,75	1,75-2,20	…
(a) 0,25% max Cu, 0,03% max P, and 0,03% max S; where specified, sulfur may be increased to 0,06 to 0,15% to improve machinability. **(b)** Available in several carbon										

Table 2.2 Hot Work Steel Thermal Treatment Temperatures [7]

	Temperature Range	
Treatments	**°C**	**°F**
Types H11, H12, H13, H19, H21		
Forging, start	1095-1135	2000-2075
Forging, start	925 min	1700 min
Annealing	845-870	1550-1600
Stress relieving	650-675	1200-1250
Hardening	1010-1025	1850-1875
Tempering	(a)	(a)
Type H42		
Forging, start	1040-1120	1900-2050
Forging, finish	980 min	1800 min
Annealing	845-870	1550-1600
Hardening, Preheat	790-815	1450-1500
Hardening, quench from	1120-1230	2050-2250
Tempering (a)	540-675	1000-1250
(a)Temperature depends on hardness and other requirements		

Table 2.3 Thermal Conductivity of hot work steels [7]

Temperature		Thermal	Conductivity
°C	**°F**	**W/m. K**	**Btu /ft. H.°F**
Types H11, H13			
215	420	24,6	14,2
350	660	24,4	14,1
475	890	24,3	14
605	1120	24,7	14,3
Type H12			
25	80	17	10,1
205	400	23	13,3
425	800	25	14,5
650	1200	26	15,3
Type H21			
25	80	25,1	14,5
205	400	29,7	17,2
425	800	29,7	17,2
650	1200	28,9	16,7
760	1400	28	16,2

Table 2.4 Density of hot work steels [7]

Steel type	Density	
	g/cm³	lb/in.³
H11, H13	7,8	0,28
H12	7,81	0,282
H21	8,19	0,296

Table 2.5 Elasticity Modulus of H11 and H13 [7]

Temperature		Modulus	
°C	°F	Gpa	〖10〗 ^6 psi
Types H11, H13			
20	70	210	30,5
150	300	192	27,8
260	500	180	26,1
345	650	191	27,7
425	800	188	27,3
480	900	186	27
540	1000	157	22,7
650	1200	114	16,5

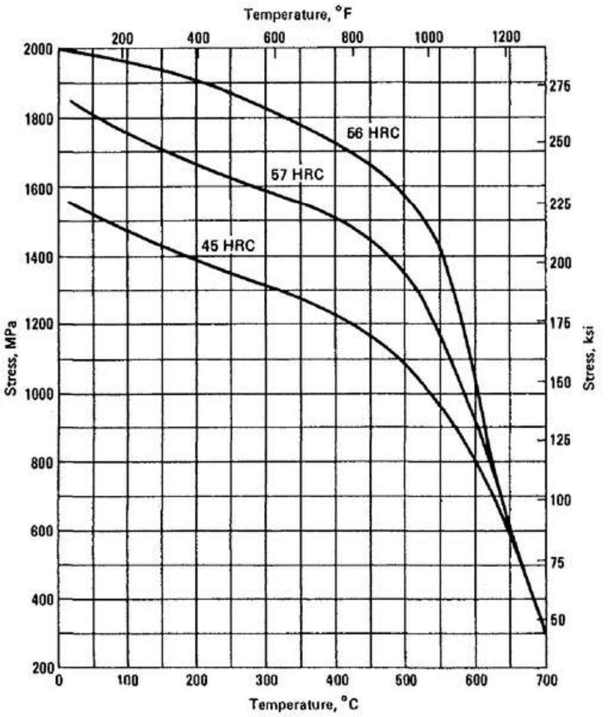


Figure 2.1 Temperature dependence of tensile strength of H11 and H13 steels [7]

2.2 Aluminum Extrusion Die Steels

Process temperature of aluminum extrusion is between 450 and 500 °C. At that temperature, die materials must keep their characteristics. Mechanical qualities such as abrasion resistance, softening resistance, and high toughness are among them. 1.2344 steel is the most popular tool steel. [8]. The 1.2344 has a long service life, high toughness, and high thermal performance. It is well known that the strength of H13 decreases when it is exposure to the temperatures of 600 °C or above. [9]. Chemical composition of H13 is shown in Table 2.6.

Table 2.6 Chemical composition of 1.2344 (%wt) [9]

No	C	Cr	Mo	V	Si	Mn	P	S
Test	0,39	5,09	1,45	0,92	1,07	0,34	0,011	0,0017
Standard	0,32-0,45	4,75-5,50	1,1-1,75	0,8-1,2	0,8-1,2	0,2-0,5	<0,03	<0,03

2.3 Hardening of Extrusion Die Steels

Stress relief is the first step in the hardening process for many key steels. During rough machining, steel is subjected to a great deal of stress. As a result, the material's deformation is reduced by the stress-relieving process. In the stress-relieving procedure, the material is heated to 600-650°C, waited at that temperature for 2 to 3 hours, then cooled in the furnace. The temperature variation regime of stress relieving steps is shown in Figure 2.3.

Preheating cycles are crucial in the hardening of hot work steel. The material is heated to an austenizing temperature in three steps to prevent distortion, thermal shock, and to equalize the temperature of the surface and center of the work piece. The phase transformation must occur throughout the entire material at the same time. The initial preheating temperature is roughly 650 °C. which is the beginning temperature of the austenite phase. The temperature for the second preheating is roughly 850 °C. [10]. Preheating steps are shown in Figure 2.3 schematically.

The material is heated to an austenizing temperature at the end of the second preheating step. The temperature of austenitizing is a particularly delicate step in the hardening process. Alloying elements dissolve in the austenite phase, and if the material is kept at austenite temperature for an extended period of time, grains grow and toughness decreases. Grain growth is limited by residual carbides, which also increase wear resistance. [11]. Austenitizing temperatures of hot work steels are shown in Table 2.7. The most common extrusion die steel H13 has an austenizing temperature of 1000-1040 °C. After the material's core reaches that temperature, it's kept for 15 to 40 minutes to complete the austenitizing. Except for the carbide which forms the vanadium carbide, all carbides in hot work steel dissolve in austenite phase. Dissolved carbides are secondary and undissolved carbides are known primary carbides.

Table 2.7 Austenitizing temperatures for hot work steels [4]

	Hardening						
	Temperature				**Holding time, min**	**Quenching medium (c)**	**Quench hardness, HRC**
	Preheat		**Austenitize**				
	°C	**°F**	**°C**	**°F**			
H10	815	1500	1010-1040	1850-1900	15-40 (dk)	A	56-59
H11	815	1500	995-1025	1825-1875	15-40 (dk)	A	53-55
H12	815	1500	995-1025	1825-1875	15-40 (dk)	A	52-55
H13	815	1500	995-1040	1825-1900	15-40 (dk)	A	49-53
H14	815	1500	1010-1065	1850-1950	15-40 (dk)	A	55-56
H16	815	1500	1120-1175	2050-2150	2_5	A, O	55-58
H19	815	1500	1095-1205	2000-2200	2_5	A, O	52-55

Chromium improves the hardenability of hot work steel, while molybdenum helps martensite formation during air cooling. Vanadium and tungsten alloys have a minor effect on hardenability, although they do prevent austenite grain growth. Due to the presence of alloying elements, hot work steel can be hardened in air cooling.

Cooling rate is chosen with the help of TTT diagram, shown in Figure 2.2. To prevent pearlite microstructure transformation during hardening, an adequate cooling rate is required. Because the temperature of the martensite finish is below zero degree Celsius, the austenite and martensite structures can be observed at room temperature. Furthermore, even if chromium alloys improve hardenability, large crossections cannot entirely transform into martensite structure, therefore a large portion will be bainitic.

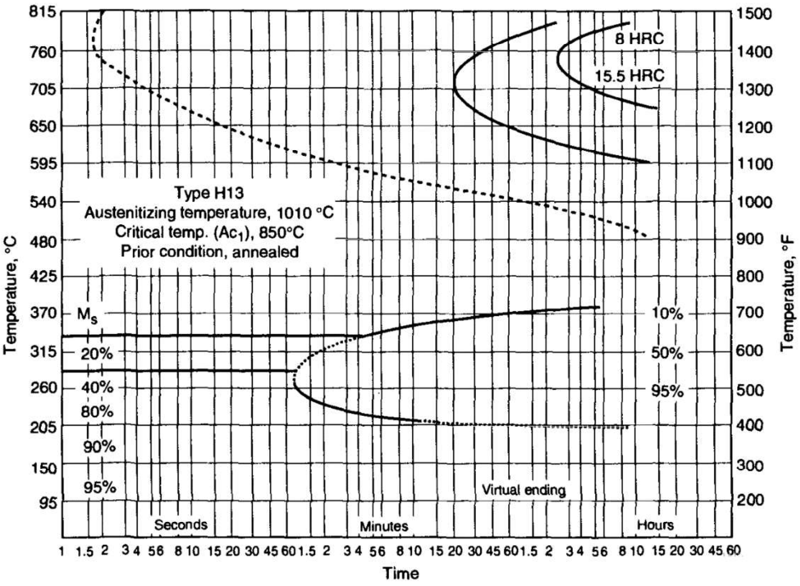


Figure 2.2 TTT diagram of 1.2344 steel [4]

Hot work steel’s hardening cycle is shown in Figure 2.3. The secondary hardening process is known as the first tempering process. As a result of this, dissolved vanadium carbide in the austenite phase precipitates, increases hardness. Residual austenite still exists in structure after rapid cooling. Because of the martensite complete temperature is below zero degrees Celsius, austenite partially converts into martensite structure during the first tempering process.

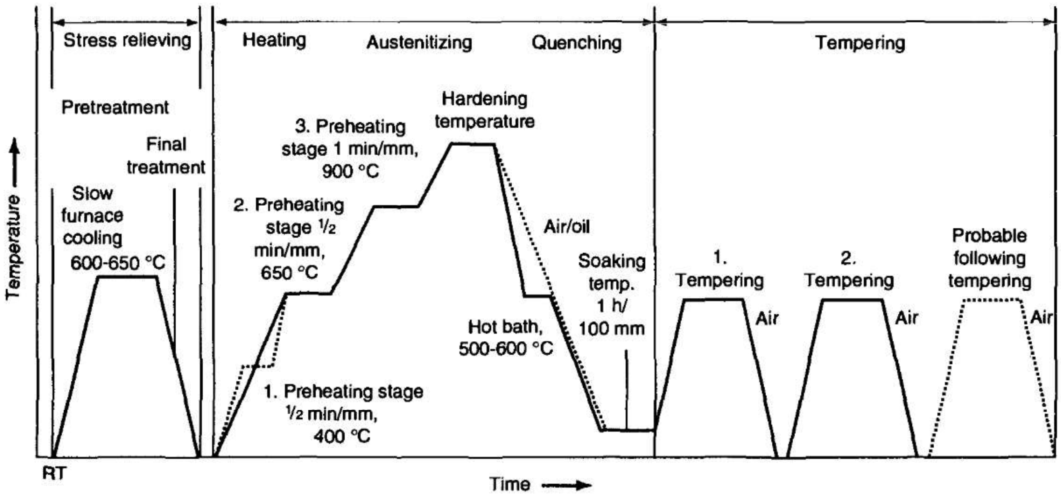


Figure 2.3 Hardening Cycle of 1.2344 Steel [4]

After the first tempering process, there is a hardness peak on temper diagram shown in Figure 2.4. The reason for this hardness increase is Vanadium precipitation dissolved in the austenite phase [12]. The martensite structure at the end of the secondary hardening process is tempered and hardness is optimized in the second tempering process, which is done at a temperature of 580-630 °C. The third tempering procedure reduces hardening stress and prepares it for wire erosion process. [4].

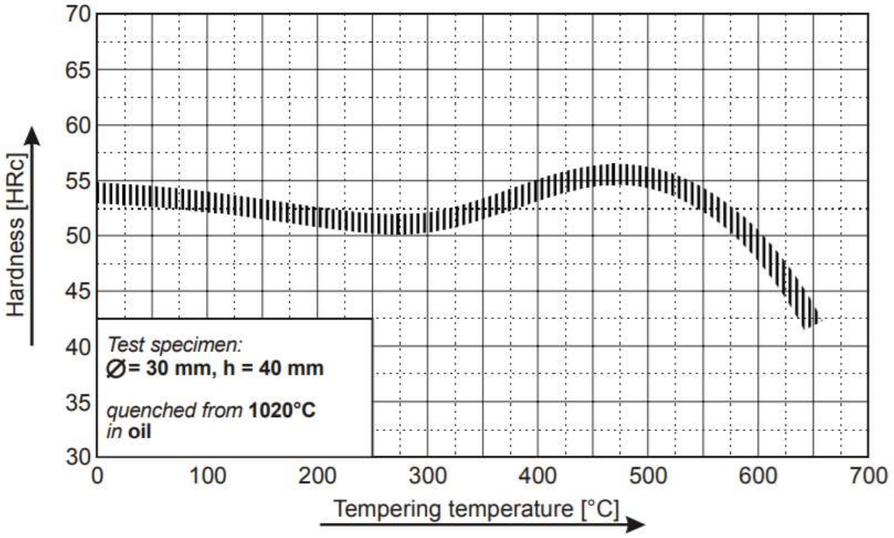


Figure 2.4 Tempering Diagram Of 1.2344 Steel [4]

2.4. Nitriding of Extrusion Dies

For improving the surface qualities of extrusion dies, nitriding is the most common type of surface heat treatment. Decomposition of ammonia gas (NH_3) causes nitrogen atoms to diffuse through steel surfaces.

$$2NH_3 \longrightarrow 6H_2 + 2N \text{ (dissolved in Fe)}$$

In extrusion dies, nitrogen atoms react with alloy components such as chromium, molybdenum, and other stable alloy elements to form nitrides, which separate in steel and improve its characteristics. Nitride layer is divided into white layer and diffusion layer as it is shown in Figure 2.5. Epsilon-$Fe_{2\text{-}3}N$ phase and gamma-Fe_4N phase are found in the white layer, while nitrides are found in the diffusion layer. White coating is avoided in most industrial applications; however, it gives higher wear resistance for the extrusion process. After quenching and tempering, the nitriding process is used. The tempering process must be 30 to 40°C cooler than the nitriding process. Because the hardness of tempered steel must not change as the temperature of the nitriding process rises. The traditional gas nitriding method has drawbacks. Because the gas concentration could not be controlled in this method, the nitriding solutions were unstable. Diffusion and the white layer cannot be controlled using this method. However, the Kn mathematic equation controls the automatic gas control nitriding process. Die performance is improved by using a controlled white layer and a diffusion layer. In general, a white layer of 2-10 microns is desirable. During extrusion, if the white layer is higher than 10-12 microns cracks and exploration can occur. [13]

Kn equation (nitridability of the nitriding atmosphere which contains ammonia);

$$K_n = \frac{p_{NH_3}}{p_{H_2}^{3/2}}$$

Extrusion die's nitriding process temperature is around 530 °C. Microstructure of resultant nitride layer of H13 steel at 530 °C is shown in Figure 2.5. Thickness of white layer is 6 micron and diffusion layer is around 150 microns. White layer includes epsilon -$Fe_{2-3}N$ and gamma Fe_4N. Nitride layer hardness distribution is shown in Figure 2.6. Surface hardness starts from 1068 Vickers and drops through the center of the work piece. Thickness of nitriding layer where it is measured around 540 Vickers shows diffusion layer finish.

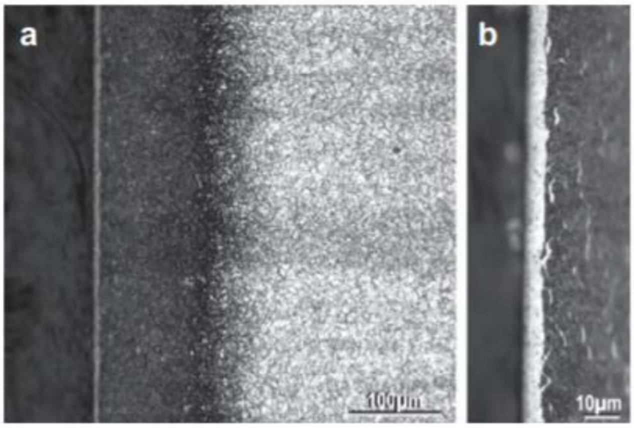


Figure 2.5 Nitride layer **a)** Diffusion zone **b)** White layer [14]

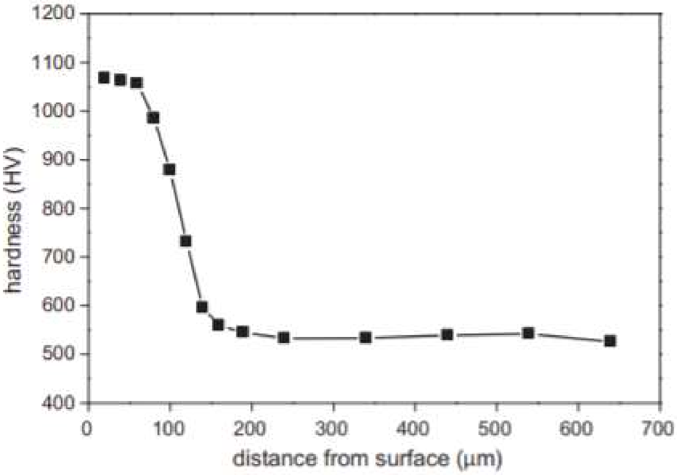


Figure 2.6 Change in hardness with distance from the surface of the nitrided extrusion die [14]

2.5. Preheating (Annealing) of Aluminum Extrusion Dies

During manufacturing, the temperature of an aluminum billet is roughly 450-520 °C. Dies are annealed at 450-470 °C to prevent thermal gradient, die failure, and thermal shock. The annealing time is set between 4 to 8 hours, depending on the thickness of the dies. Air or nitrogen gas surrounding with electric resistance are used in annealing furnaces, however air furnace is used more frequently due to high cost. During annealing of dies, white layer oxidized to Fe_2O_3 and Fe_3O_4 as shown in Figure 2.7. The thickness of the oxidized layer increases fastly as the annealing time and temperature increase. During manufacture, the oxidized layer breaks away, causing micro cracks on the surface. [15].

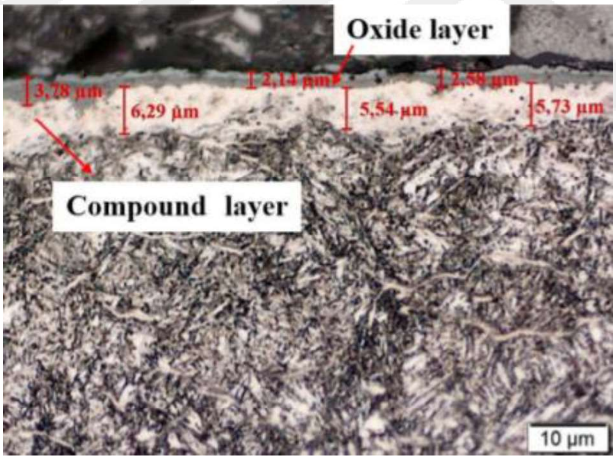


Figure 2.7 Oxidation on compound (white) layer [16]

Experimental research on nitride layer oxidation has been available since 2020. Specimens were nitrided at 530°C, producing a white layer of 7-8 microns and a diffusion layer of 70 microns, as illustrated in Figure 2.3. After that, the specimens are heated to 470°C for 8, 12, 24, 30, 60 hours, and then to 270°C for 20 hours. According to their results, oxidizing begins to build on the white layer after 3 hours, and the oxidized layer becomes stable after 8 hours. The white layer disappeared within 30-60 hours. After 12 hours, the surface hardness drops to 1103 HV, and after 30 hours, it drops to 964 HV. [16].

In an experiment done in 2009, dies were heated for 4, 16, and 30 hours in atmosphere and nitrogen furnace, resulting in the reported solutions. To begin with, the specimen was heated to 450°C in an air environment for 4 hours, causing the specimen to oxidize. Only traces of gamma nitrides remain after changing the annealing time to 16/32 hour. After 4 hours annealing process in a nitrogen atmosphere, the white layer oxidized, but not after 4 hours in an air furnace. Even after 16 hours, a substantial portion of the nitrided layer had left. The nitride layer has almost oxidized after 30 hours. Finally, a nitrogen furnace by itself is not a solution; it only generates oxidized late. [15]

H13 steel is nitrided for 6 hours at 535°C in this experiment. Initially, specimens are annealed in an air furnace at 450°C for 6,8,12,15,20,25,30,40 hours. The oxidation of the white layer is observed, as well as how the thickness of the white layer changes as the annealing time increases. Second, similar specimens are annealed in a nitrogen furnace at 450°C for 6,12 hours to see how successful the nitrogen furnace is at oxidation. In addition, two specimens are subjected to an abrasion test in order to determine the white layer's abrasion resistance. The first specimen has a white layer, while the second specimen does not have any (only have diffusion layer).

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Specimen Selecting and Nitriding process

1.2344 is the most common hot work tool steel used in extrusion dies Some specimens that are used for microstructural characterization shown in Figure 3.1. Nine specimens are nitrided at 535 °C 6 hours (KN:6) in gas furnace that is given in Figure 3.2. Initially, the vacuum and purging processes in the furnace are done. The furnace is then heated to 460°C, and ammonia gas is injected into the furnace environment. Ammonia gas decomposes into nitrogen and hydrogen atoms higher 450 °C. [17] After reaching 535 °C, the furnace is hold at that temperature for 6 hours. Nitrogen atoms diffuse in the steel surface during this process and hydrogen gases are taken away and neutralized by flame. Furnace is slowly cooled to 70-80 °C at the end of the 6-hour period. Temperature profile is shown in Figure 3.3.

Figure 3.1 Chosen 9 pieces 1.2344 how work tool steels for study

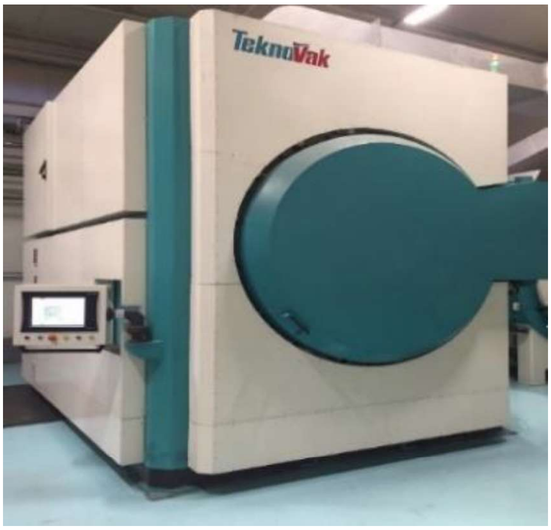
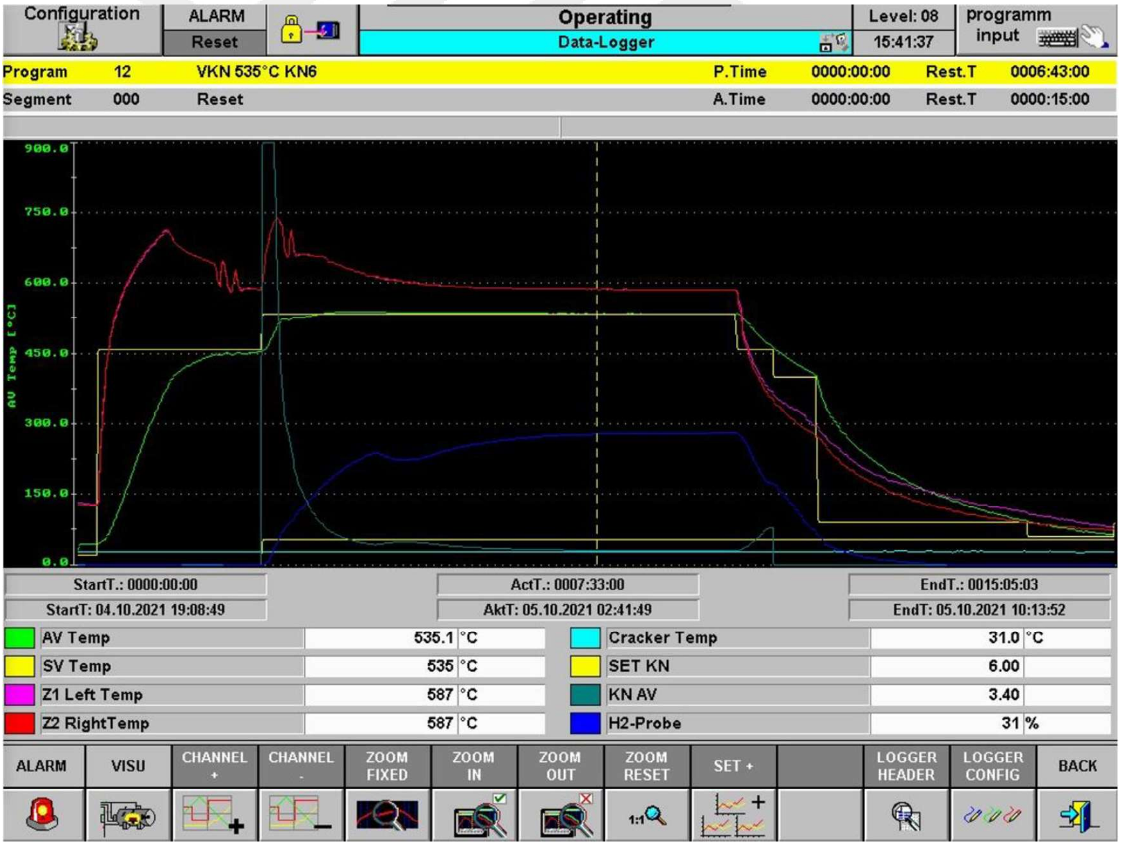


Figure 3.2 Kn controlled gas nitriding furnace



Level: 08
15:41:37

Program 12 VKN 535°C KN6 P.Time 0000:00:00 Rest.T 0006:43:00
Segment 000 Reset A.Time 0000:00:00 Rest.T 0000:15:00

StartT.: 0000:00:00
StartT: 04.10.2021 19:08:49
ActT.: 0007:33:00
AktT: 05.10.2021 02:41:49
EndT.: 0015:05:03
EndT: 05.10.2021 10:13:52

🟩	AV Temp	535.1	°C	🟦	Cracker Temp	31.0	°C
🟨	SV Temp	535	°C	🟨	SET KN	6.00	
🟪	Z1 Left Temp	587	°C	🟦	KN AV	3.40	
🟥	Z2 RightTemp	587	°C	🟦	H2-Probe	31	%

Figure 3.3 Gas nitriding process graphic

Nine study specimens' nitriding results are as it is seen on Figure 3.4. White layer thickness is between 5,2 and 6 microns. Diffusion layer thickness is 118,9 microns. In addition, all specimen's core hardness is 48 HRC as it is given on micrograph given in Figure 3.4.

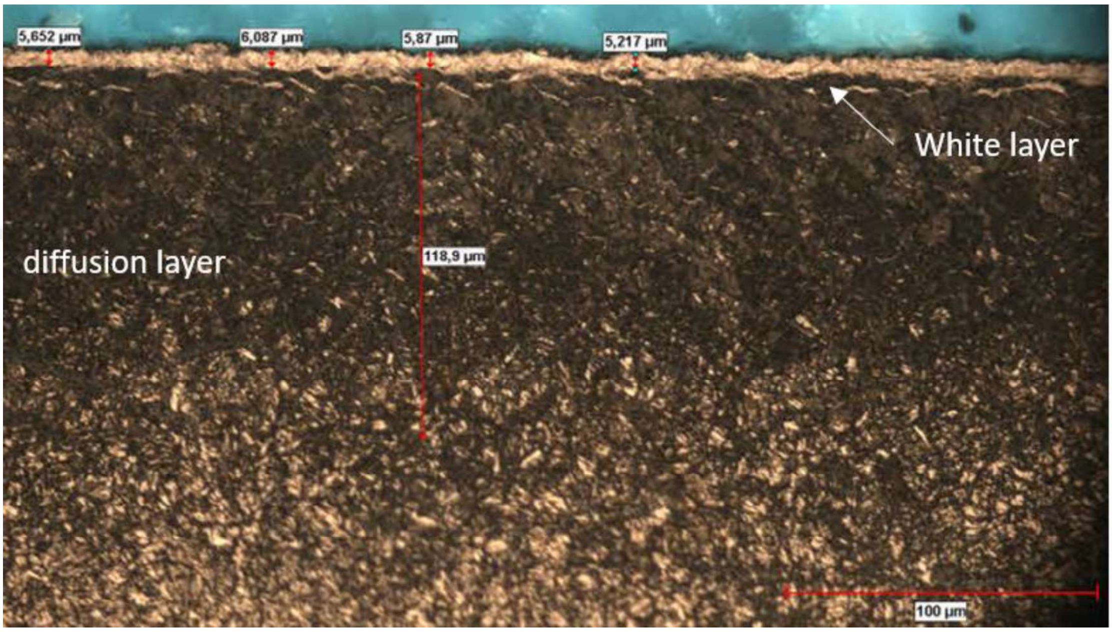


Figure 3.4 Nitriding layer of 1.2344 hot work steel on optical microscope (500X)

3.2. Preheating of 1.2344 Steel Specimens

1.2344 nitrided nine specimens (3cm x 1cm) (for nine different annealing time) are divided into two parts, resulting in nine half specimens that are annealed in an air furnace at 450°C. 6,8,12,14,18,20,25,30, and 40 hours. Specimens are suspended in order to connection with surrounding atmosphere directly in a furnace after being connected to metal as it is shown in Figure 3.5.

Figure 3.5 Nitrided specimens are suspended in air furnace

In nitrogen gas, two pieces 1.2344 nitrided specimens are annealed at 450°C for 6 and 12 hours and furnace is shown in Figure 3.6.

Figure 3.6 Nitrided specimens are suspended in inert gas furnace

3.3. Preparation of Specimens for Analyzing

Specimens are mounted at 200°C under pressure to make the material easier to handle as it is shown in Figure 3.7.

Figure 3.7 Mounting machine

Figure 3.8 At 535 °C 6 hours nitrided and mounted 1.2344 hot work steel specimens

Specimens are grinded by 500, 800, 1200, 2400 scale waterproof grinder and polishing by using alumina powder with machine which is shown in Figure 3.9.

Figure 3.9 Grinding and polishing of nitrided specimens

3.4 Abrasion Test Preparation of Specimens Which Have White Layer and No White Layer on Nitride Layer

Two identical pieces of 1.2344 tool steel are chosen for abrasion testing. The first specimen, which contains a 6 – 8 microns’ white layer and a 121,1-microns diffusion layer, is subjected to a 6-hour 535 °C nitriding process, as illustrated in Figure 3.10. The second specimen is subjected to a 4-hour 535 °C nitriding process, producing an 80-micron diffusion layer (white layer not included). It is shown in Figure 3.11. In addition; abrasion test parameters are shown in Table 3.1.

Table 3.1 Pin-on plate abrasion test parameters and informations

	Sliding Distances(meter)	**Load(N)**	**Ball**
White Layer	100 and 200	15	Aluminum oxide (5mm)
No white layer	100 and 200	15	Aluminum oxide (5mm)

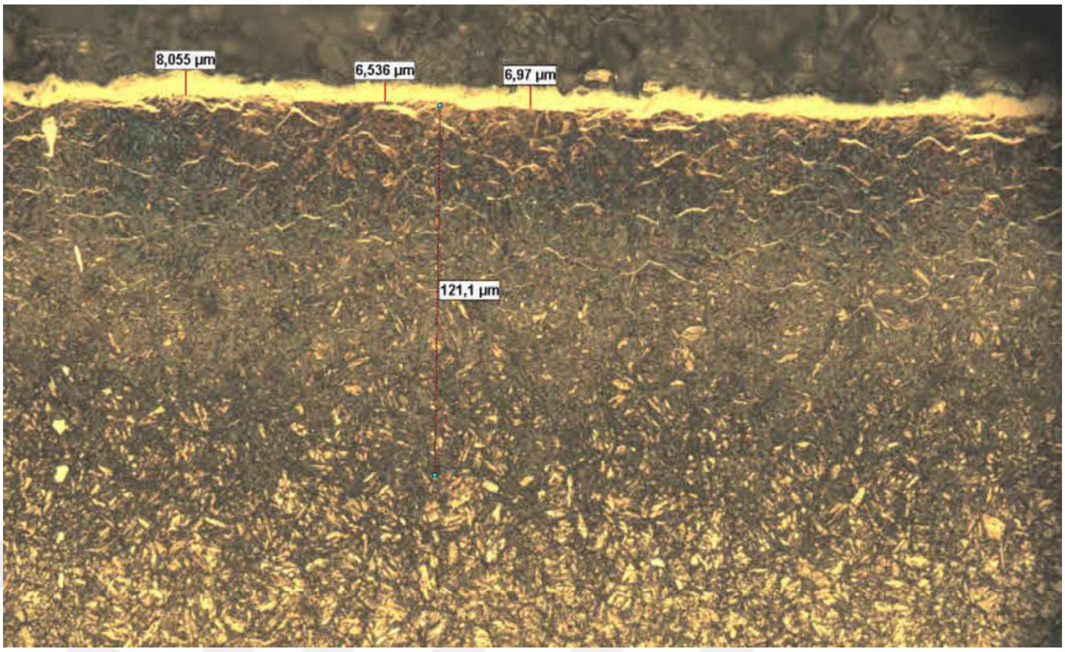


Figure 3.10 1.2344 nitrided specimen with white layer

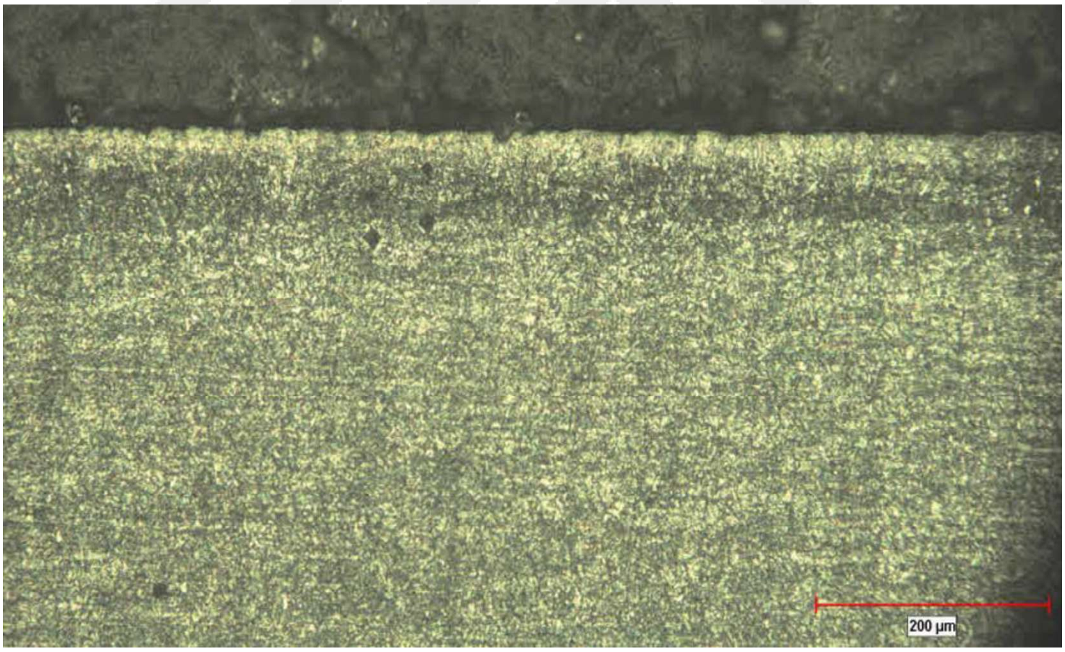


Figure 3.11 1.2344 nitrided specimen without white layer

The abrasion machine is set to 15 N, with sliding distances of 100 and 200 meters. For abrasion, a ceramic ball (aluminum oxide, 5 mm) was chosen and machine is pin-on plate which is shown in Figure 3.11 and 3.12.

Figure 3.12 Pin on plate abrasion test machine

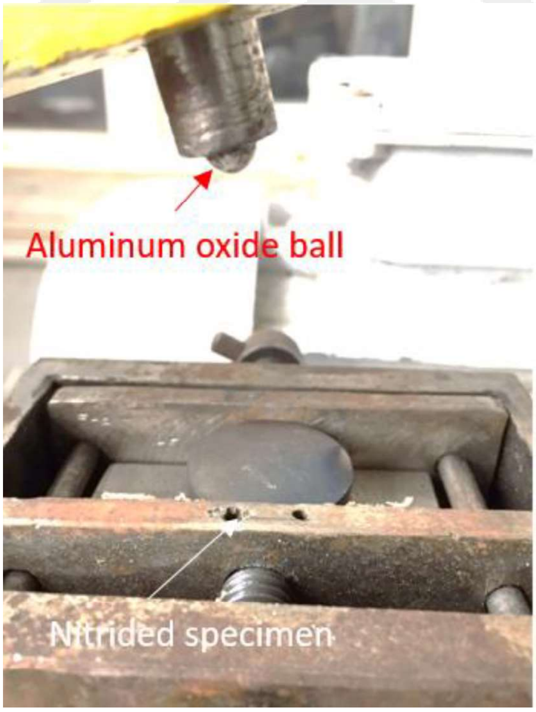


Figure 3.13 Ceramic ball and nitrided specimen

Figure 3.14 Abrassion test direction on nitrided specimens 100 and 200 meters

3.5 Analyzing of Specimens

Polished specimens are measured by means of hardness distribution by micro-Vickers machine. Hardness measurement starts from surface to 50 Vickers higher of core hardness value (540 HV) by 100-gram load and Vickers machine is shown in Figure 3.15.

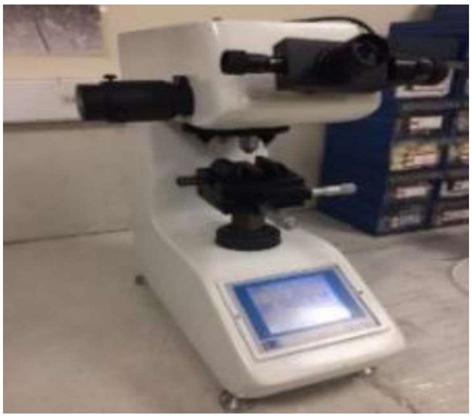


Figure 3.15 Micro Vickers machine

The polished specimens are then etched with $NiO_3$3%-Methanol (nital) solution for nitrided layer examination using an optical microscope, they are also examined by using electron microscope which are given on Figure 3.16, 3.17, 3.18.

Figure 3.16 Optical microscope

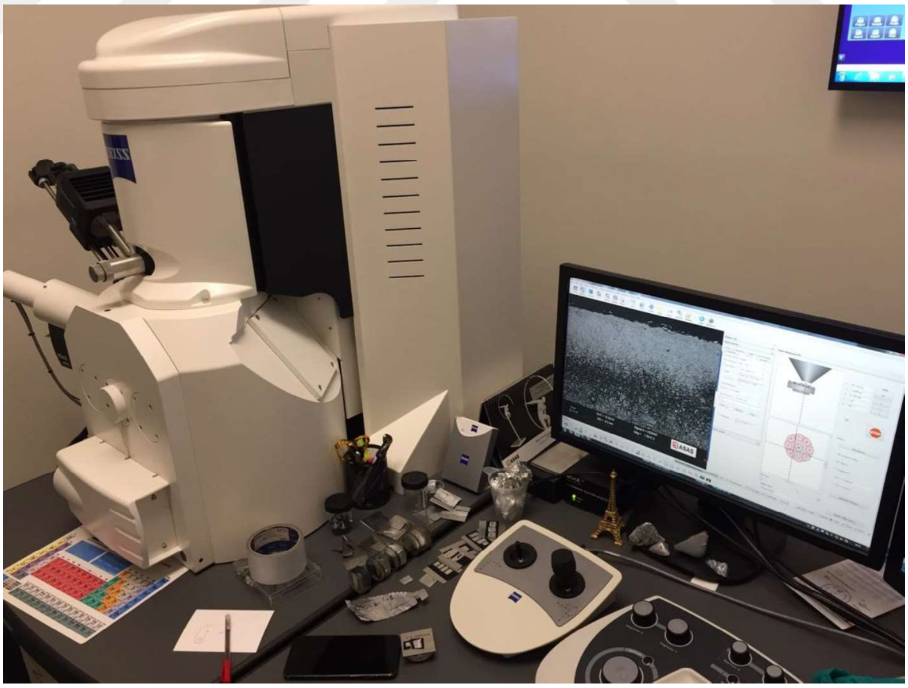


Figure 3.17 Electron microscope

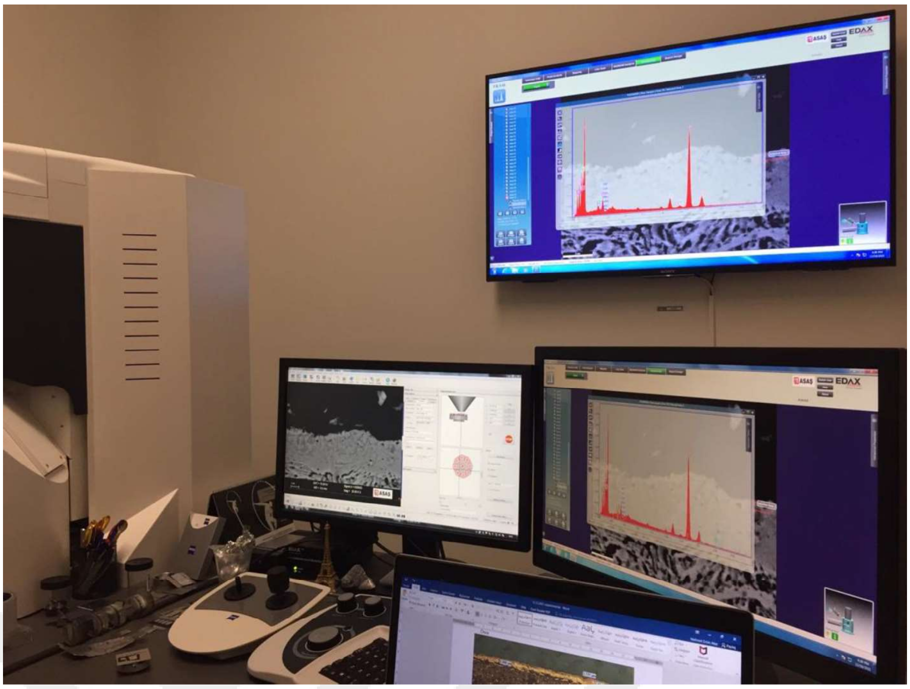


Figure 3.18 Screen appearance of Edx method

Nitrided layer is observed after annealed in air furnace by a xrd machine which is shown in Figure 3.19.

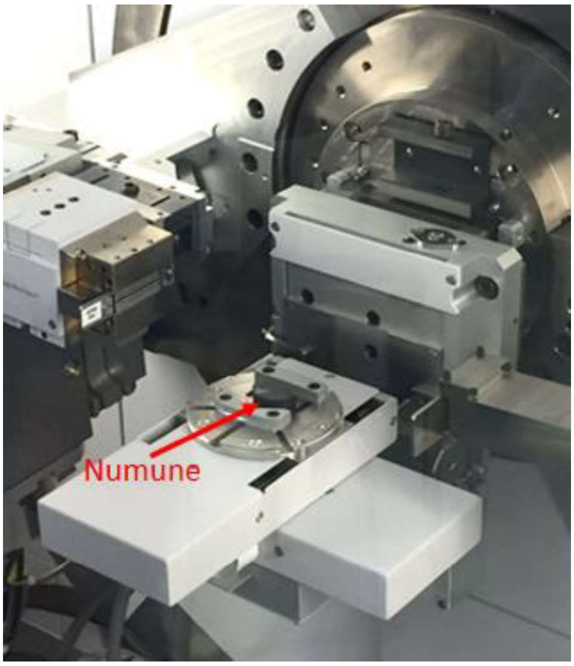


Figure 3.19 XRD method

CHAPTER 4

RESULT AND DISCUSSION

4.1. Analyzing Annealing Time How Effect Nitride Layer in Air Furnace

The white layer thickness of a 6 hour nitrided specimen at 535 °C is between 5,6 and 6 microns, while the diffusion layer is 118,9 microns and shown in Figure 4.1.

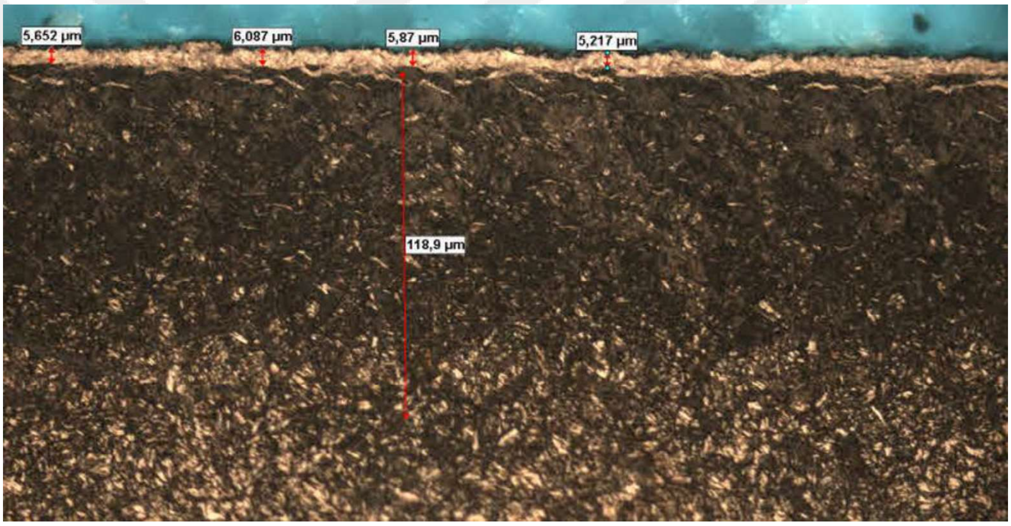


Figure 4.1 1.2344 tool steel nitriding layer before annealing(preheating) by optical microscope 500x

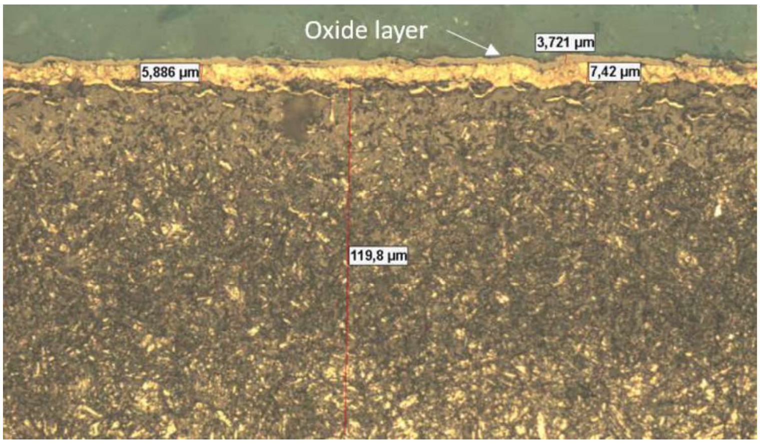


Figure 4.2 1.2344 oxidized nitrided layer of tool steel after 6 hours in air furnace by optical microscope 500x

The white layer is between 5,8 and 7,4 after 6 hours of annealing, while the diffusion layer is 119,8. On the white layer, an oxide layer formed at a thickness of 3,7 microns and stable which are shown in Figure 4.2.

6 hours annealed in air furnace nitride layer's surface is observed by xrd method. Therefore, magnetite (Fe_3O_4) phase (Weight fraction/ %65.1) and iron nitride phase (3/0.97) – Epsilon Weight fraction/ %34.9) are observed on the surface which is shown in Figure 4.3

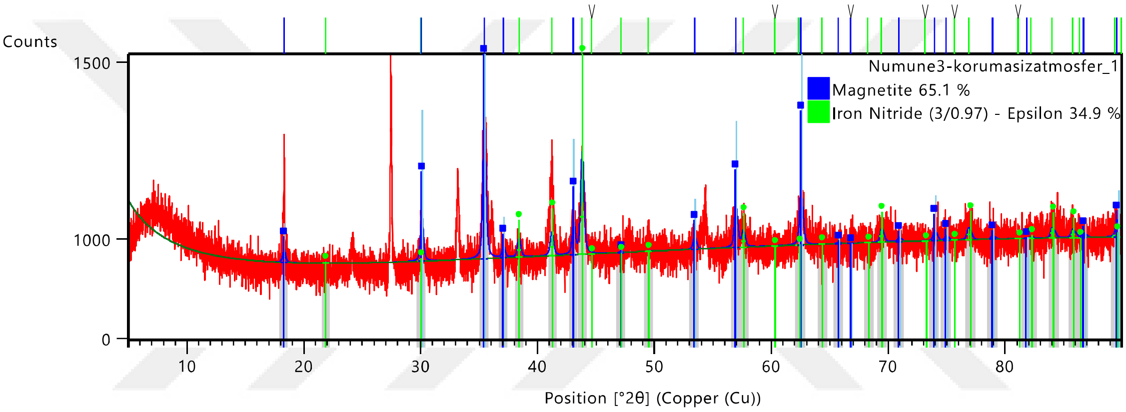


Figure 4.3 XRD Scanning graph of specimen which is held 6 hours for annealing in air furnace

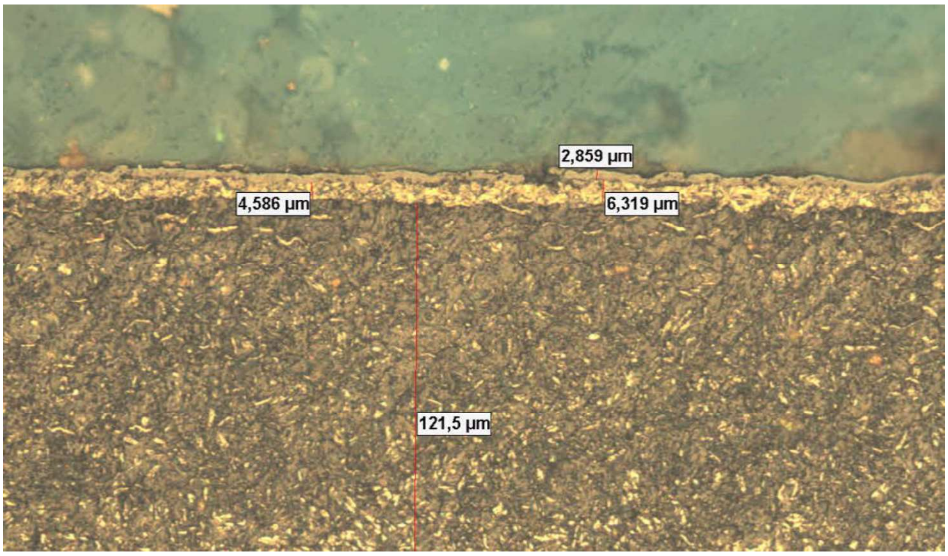


Figure 4.4 1.2344 tool steel after 8 hours annealing in air furnace by optical microscope 500x

After an 8-hour annealing process, the white layer is between 4,5 and 6,3 microns the diffusion layer is 121,5 microns. On the white layer, an oxide layer formed at a thickness of 2,8 microns and stable which are shown in Figure 4.4.

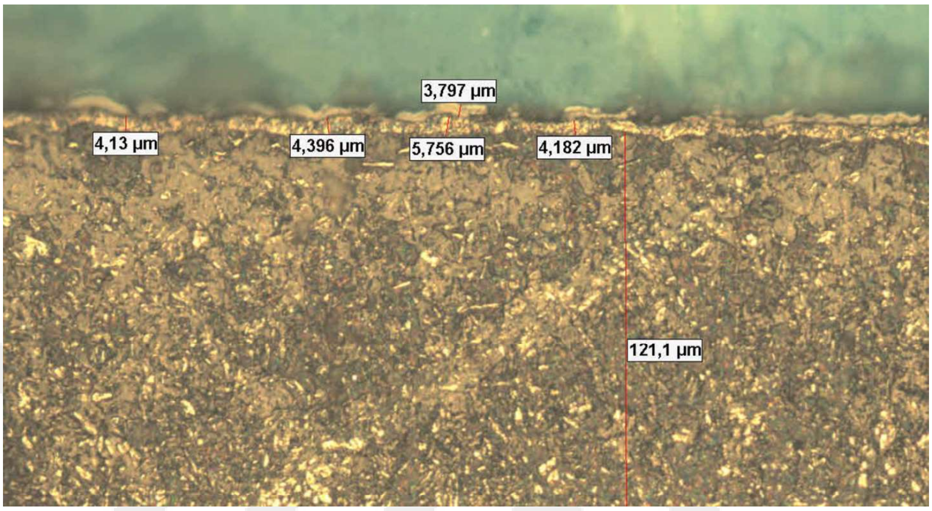


Figure 4.5 1.2344 tool steel after 12 hours annealing in air furnace by optical microscope 500x

After 12 hours of annealing; the white layer is between 4,1 and 5,7, diffusion layer is 121,1 microns. Thus, oxide layer occurred 2,8 microns on white layer and stable which are shown in Figure 4.5.

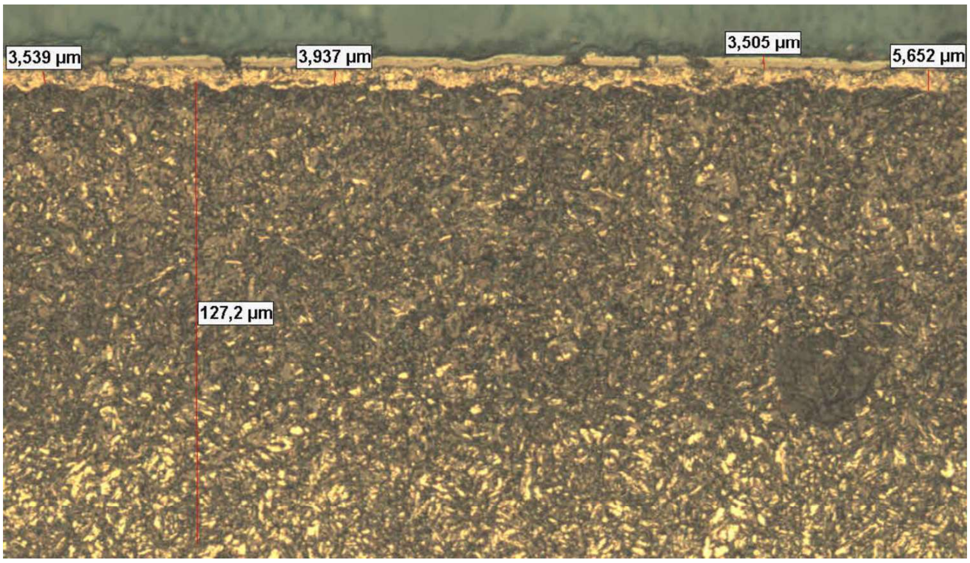


Figure 4.6 1.2344 tool steel after 14 hours of annealing in air furnace by optical microscope 500x

After 14 hours annealing; the white layer is between 3,5 and 5,6, diffusion layer is 127,2 microns measured. As a result, a 3,5-micron oxide layer formed on the white layer and stable which are shown in Figure 4.6.

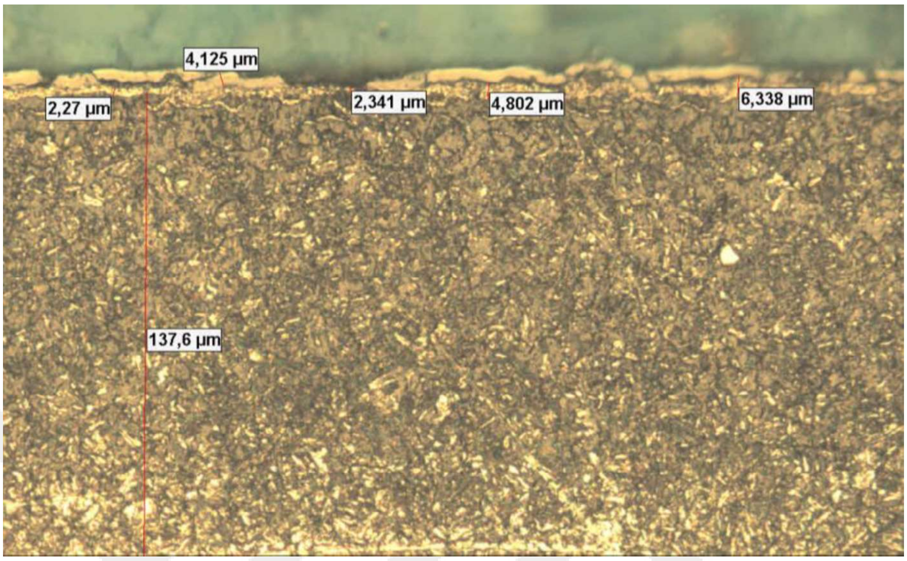


Figure 4.7 1.2344 tool steel after 18 hours annealing in air furnace by optical microscope

After 18 hours of annealing; the white layer is between 2,2 and 6,3, diffusion layer is 137,6 microns. As a result, an oxide layer formed on the white layer at a thickness of 4,1 microns and easily seen which are shown in Figure 4.7.

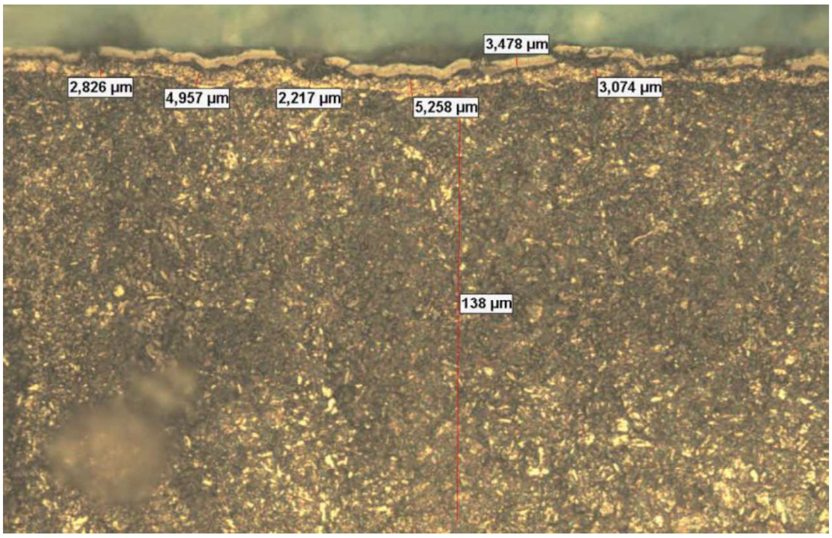


Figure 4.8 1.2344 tool steel after 20 hours of annealing in air furnace by optical microscope

After 20 hours of annealing; the white layer is between 2,2 and 5,2, diffusion layer is 138 microns. On the white layer, an oxide layer formed at a thickness of 3,4 microns and easily seen which are shown in Figure 4.8.

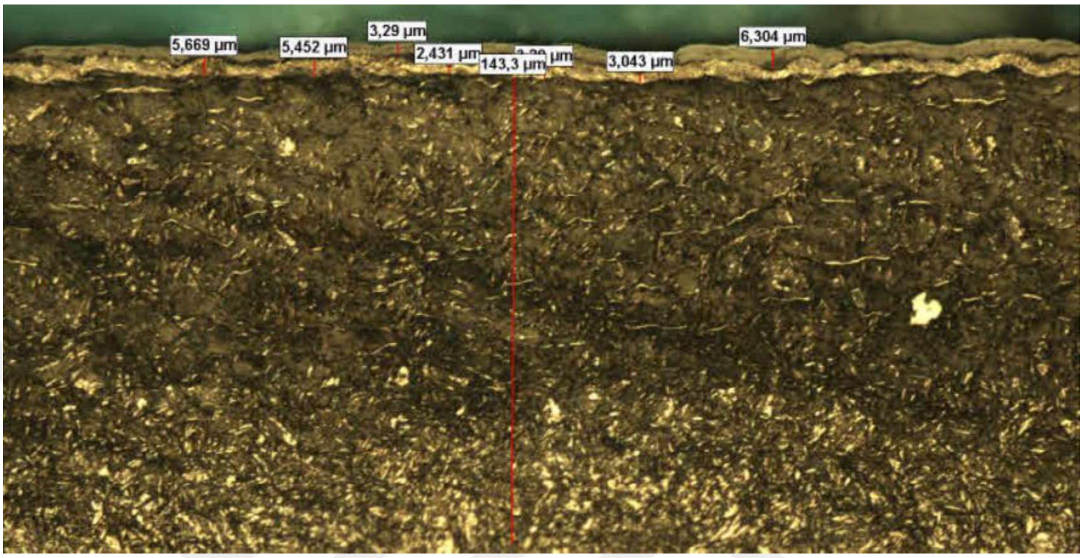


Figure 4.9 1.2344 tool steel after 25 hours of annealing in air furnace by optical microscope 500x

The white layer is between 2,4 and 5,6 microns after 25 hours of annealing, while the diffusion layer is 143,3 microns. On the white layer, an oxide layer formed at a thickness of 6,3 micron and easily seen which are shown in Figure 4.9.

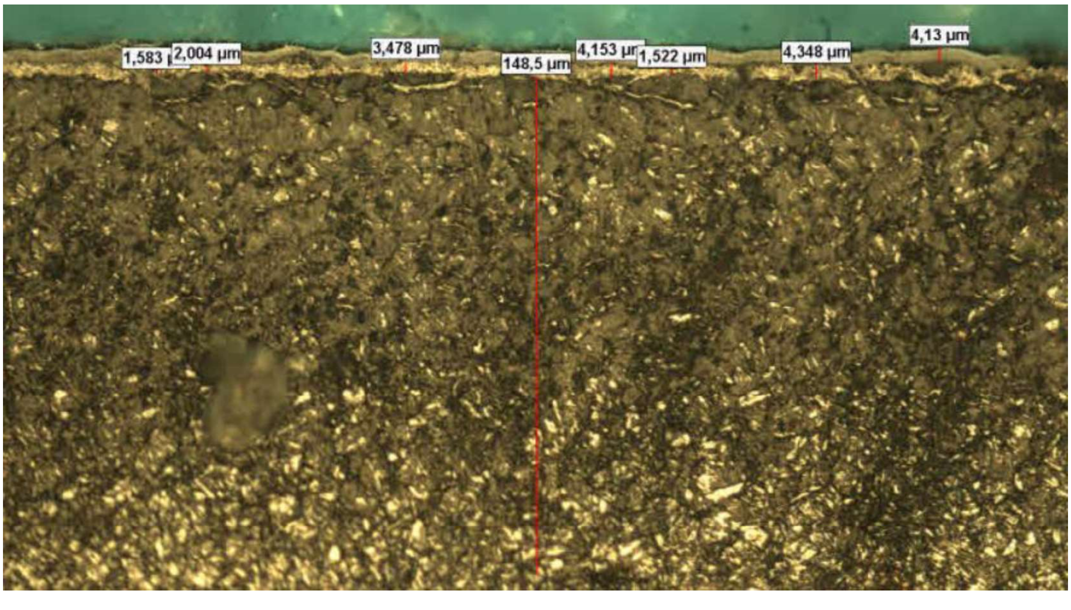


Figure 4.10 1.2344 tool steel After 30 hours annealing in air furnace by optical microscope 500x

The white layer is between 1,5 and 4,3 microns after 30 hours of annealing, and the diffusion layer is 148,5 measured. As a result, a 4.1-micron oxide layer formed on the white layer. and easily seen which are shown in Figure 4.10.

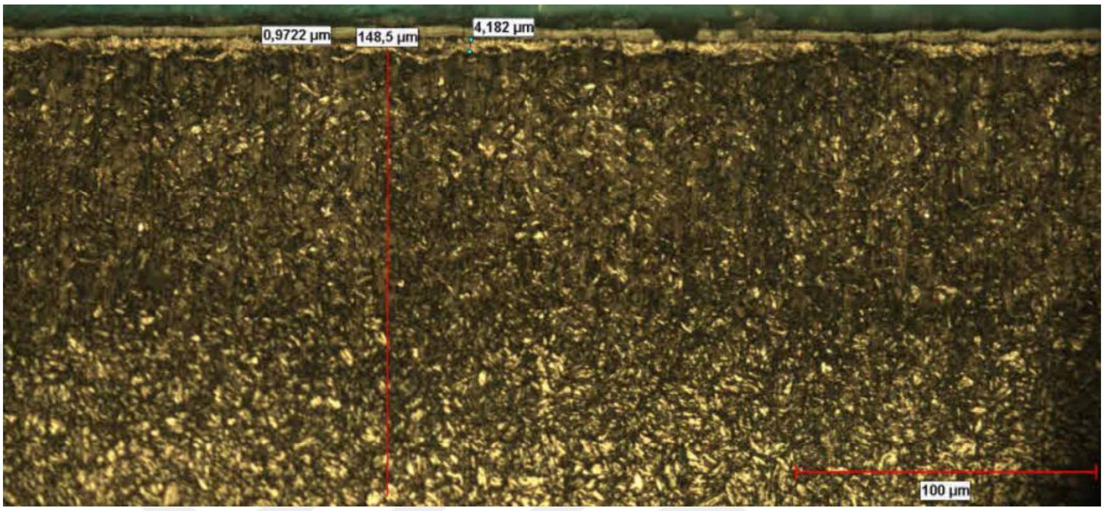


Figure 4.11 1.2344 tool steel after 40 hours annealing in air furnace by optical microscope 500x

The white layer is between 0,9 and 4,1 microns after 40 hours of annealing, while the diffusion layer is 148,5 microns. As a result, a 4.1-micron oxide layer formed on the white layer and stable which are shown in Figure 4.11.

All nine specimens' hardness measured 48 HRC before nitriding. Hardness of core steel does not change end of the 40 hours and 450 °C shown in Figure 4.12.

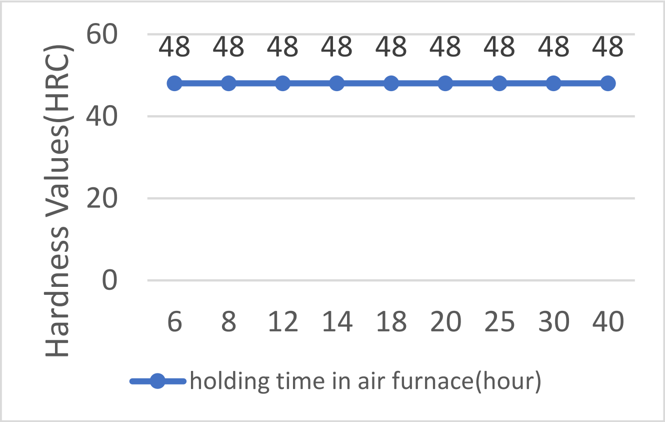


Figure 4.12 1.2344 tool steel hardness changing with annealing time at 450 °C in air furnace

As the annealing time increases, nitrogen atoms diffusing distance from surface increases too, increasing the thickness of the diffusion layer. After the first 12 hours, the diffusion layer thickness begins to increase which are shown in Table 4.1 and Figure 4.13.

Table 4.1 1.2344 tool steel diffusion thickness changing with annealing time at 450 °C atmosphere furnace

Annealing Time (hours)	**Diffusion Layer Thickness (micron meter)**
6	122
8	121
12	120
14	125,9
18	138,6
20	137,2
25	143
30	148,5
40	148,5

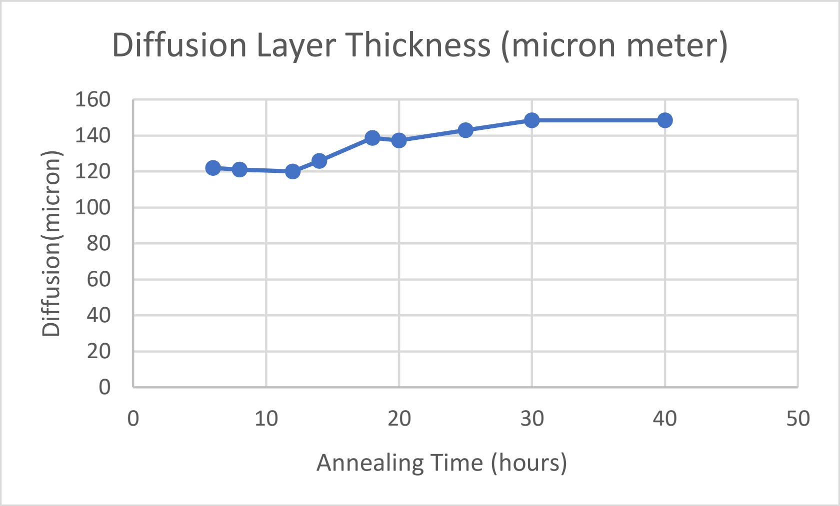


Figure 4.13 1.2344 tool steel diffusion thickness changing with annealing time at 450 °C atmosphere furnace

Table 4.2 Minimum and maximum thickness of white layer and the ratio of change on these thicknesses with respect to annealing time

		White Layer (micron meter)			
		Lower Bound	**Upper Bound**	**Lower bound percantages changing**	**Upper bound percantages changing**
6 hours	**Before annealing**	6,5	7,4	-3%	0%
	After annealing	6,3	7,4		
8 hours	**Before annealing**	5,2	6	-12%	0%
	After annealing	4,6	6		
12hours	**Before annealing**	5,4	6,3	-22%	-10%
	After annealing	4,2	5,7		
14 hours	**Before annealing**	4,6	5,5	-30%	-2%
	After annealing	3,2	5,4		
18 hours	**Before annealing**	6,5	8	-60%	-25%
	After annealing	2,6	6		
20 hours	**Before annealing**	5,5	6,8	-60%	-29%
	After annealing	2,2	4,8		
25 hours	**Before annealing**	7,2	7,8	-69%	-28%
	After annealing	2,2	5,6		
30 hours	**Before annealing**	6,1	6,3	-75%	-32%
	After annealing	1,5	4,3		
40 hours	**Before annealing**	5,9	6,9	-85%	-41%
	After annealing	0,9	4,1		

Lower bound white layer thickness begins to reduce after 12 hours of annealing time, and this quantity of lowering rises as annealing duration increases. Furthermore, by the conclusion of the 40 hours, the lower bound white layer thickness has decreased to 0,9 microns, with 85 percent distraction.

After 12 hours of annealing, the thickness of the upper white layer begins to decrease. This thickness reduces to a maximum of 4,1 micron (% 41). Therefore, it means white layer is still existing for increased annealing time.

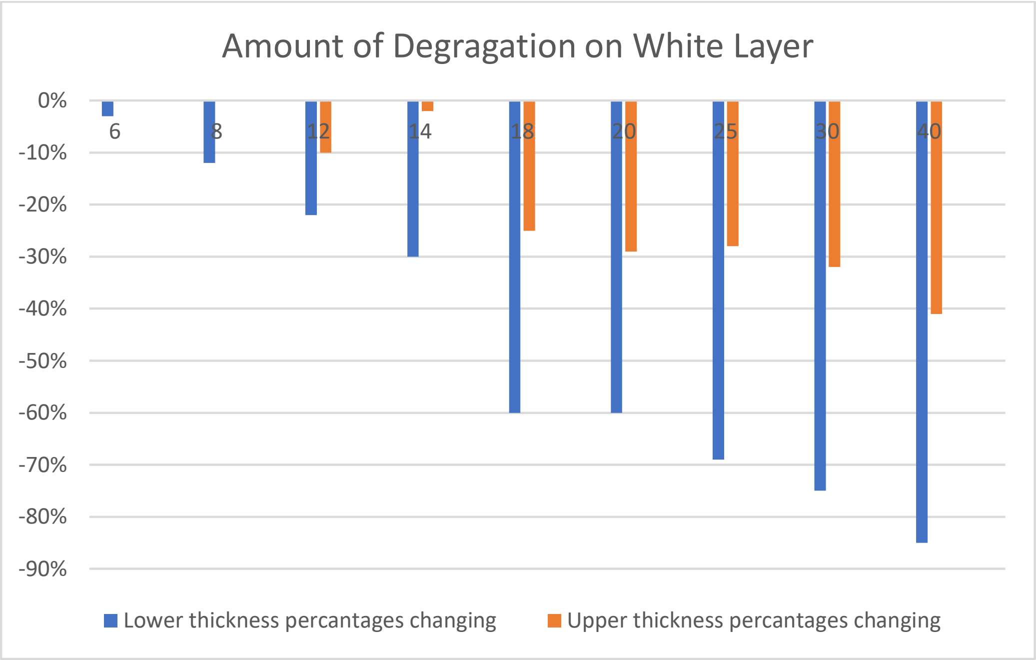


Figure 4.14 White layer thickness changing with annealing time at 450 °C under atmosphere

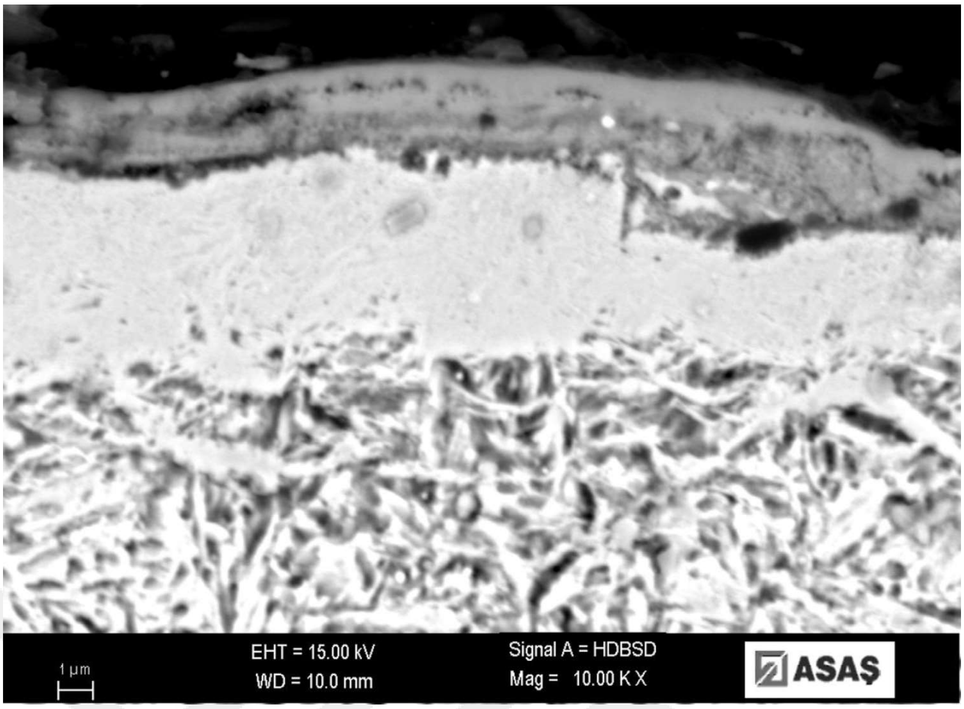


Figure 4.15 1.2344 tool steel after 12 hours of annealing on air (electron microscope) 10. KX

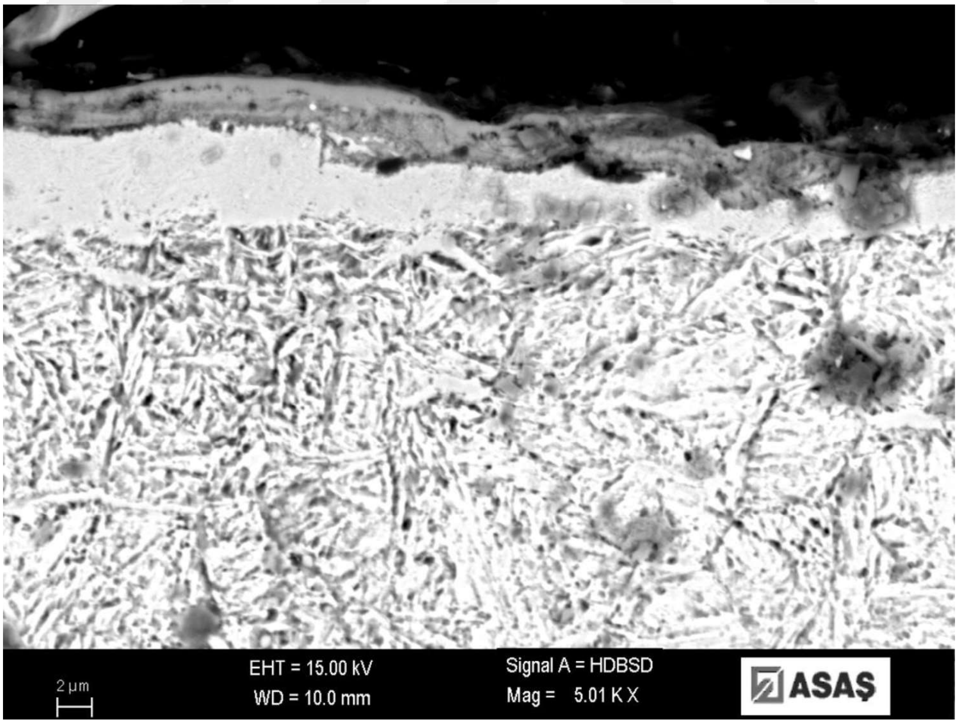


Figure 4.16 1.2344 tool steel after 12 hours of annealing on air (electron microscope) 5. KX

The gray line on the white layer, which is the oxide layer, can easily be seen on Figure 4.15 and 4.16.

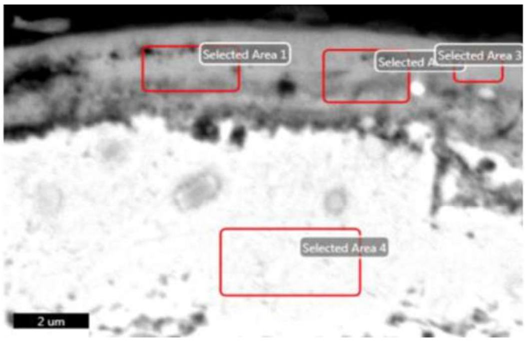


Figure 4.17 Microscopic image of 1.2344 tool steel annealed for 12 hours annealed for 12 hours on air. 4 areas indicated on image to get elemental analysis.

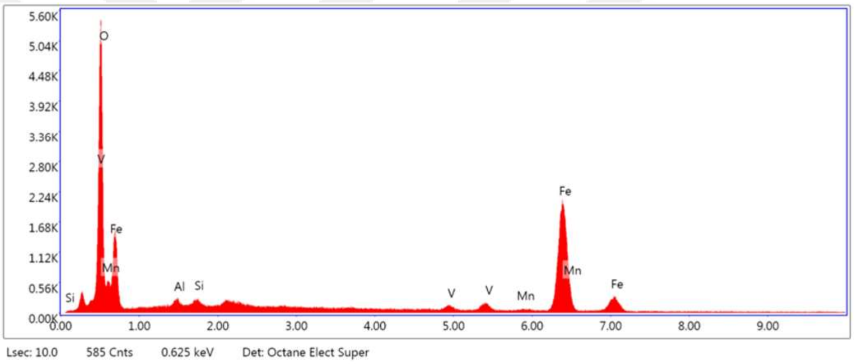


Figure 4.18. Edx Elemental analyze graphic on white layer surface.

Table 4.3 Elemental percentages on selected area

Element	Weight%	Atomic%	Error%
CK	3,64	10,34	12,43
OK	19,55	41,73	6,19
MnL	0,09	0,05	54,52
SiK	1,15	1,4	10,65
VK	0,81	0,54	23,21
CrK	4,75	3,12	7,53
FeK	70,01	42,8	2,85

Table 4.4 Elemental percentages on selected area 2

Element	Weight%	Atomic%	Error%
CK	3,65	10,28	12,45
OK	19,82	41,95	6,29
MnL	0	0	73,35
AlK	1,62	2,03	10,84
VK	1,47	0,98	16,24
CrK	5,28	3,44	7,14
FeK	68,16	41,32	2,92

Table 4.5 Elemental percentages selected area 3

Element	Weight%	Atomic%	Error%
OK	20,94	47,77	5,87
MnL	0,06	0,04	84,31
AlK	0,41	0,55	25,31
SiK	0,3	0,39	28,76
VK	1,42	1,02	8,23
FeK	76,87	50,23	2,85

Table 4.6 Elemental percentages selected area 4

Element	Weight%	Atomic%	Error%
NK	6,68	22,51	9,09
MnL	2,38	2,05	6,2
WM	2,16	0,55	12,8
MoL	1,71	0,84	14,13
VK	1,22	1,13	16,8
CrK	6,3	5,72	6,02
FeK	79,55	67,21	2,83

The element %20 oxide appears on the nitride layer after it has been annealed for 12 hours in an air furnace. Other elemental concentrations in hot work steels are unique.

4.2. Analyzing Annealing Holding Time How Effect Nitride Layer in Nitrogen Purge Furnace

On a controlled atmosphere furnace, nitrided specimens are annealed for 6 to 12 hours. Therefore; those specimens are shown before and after annealing process in Figure 4.19, 4.20, 4.21, 4.22 to analyses how much they are protected from oxidized.

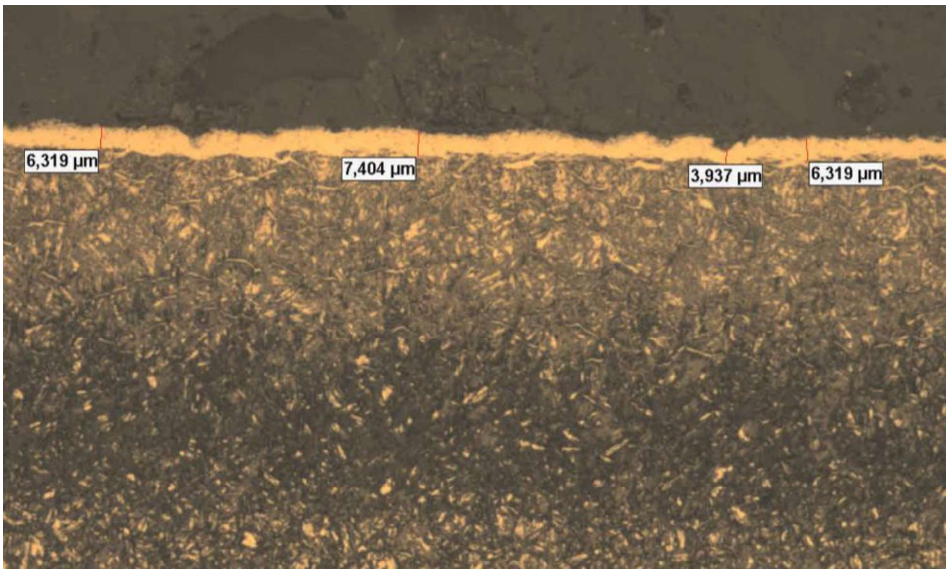


Figure 4.19 The microstructural image 1.2344 nitrided tool steel before annealing (optical microscope 500X)

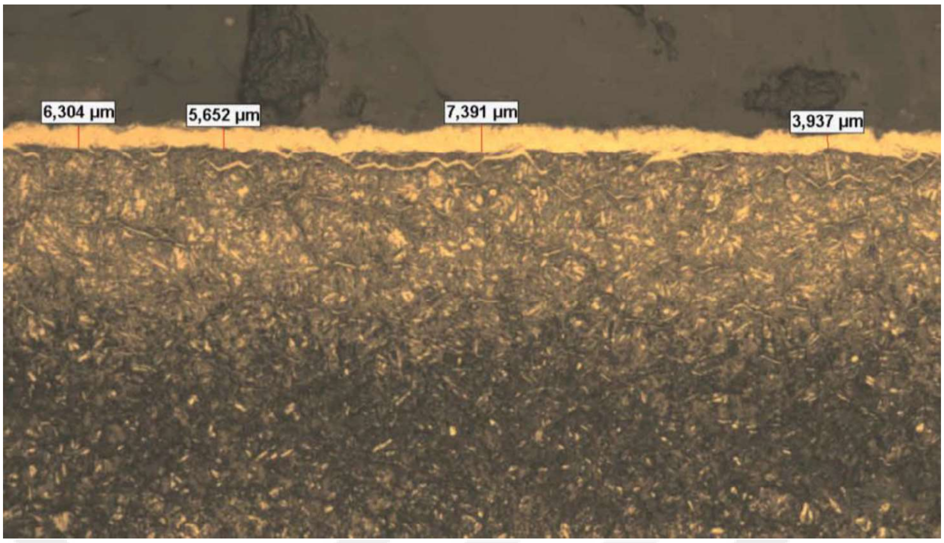


Figure 4.20 The microstructural image 1.2344 tool steel after 6 hours of annealing in protected furnace with nitrogen gas on optical microscope 500X

The nitrided specimen annealed for 6 hours in controlled atmosphere (nitrogen gas) do not show any change by means of microstructure and the oxidized layer is not formed. White layer and diffusion layer are kept same so white layer is between 3,9- and 7,3-micron meters. The microstructural image taken by optical microscope is shown in Figure 4.20.

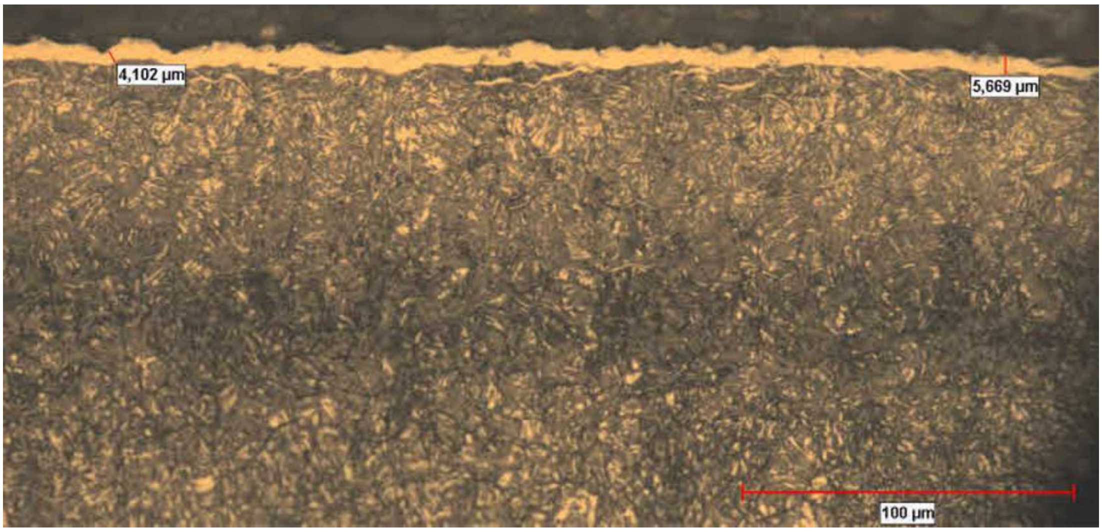


Figure 4.21 The microstructural image 1.2344 tool steel before annealing (optical microscope 500X)

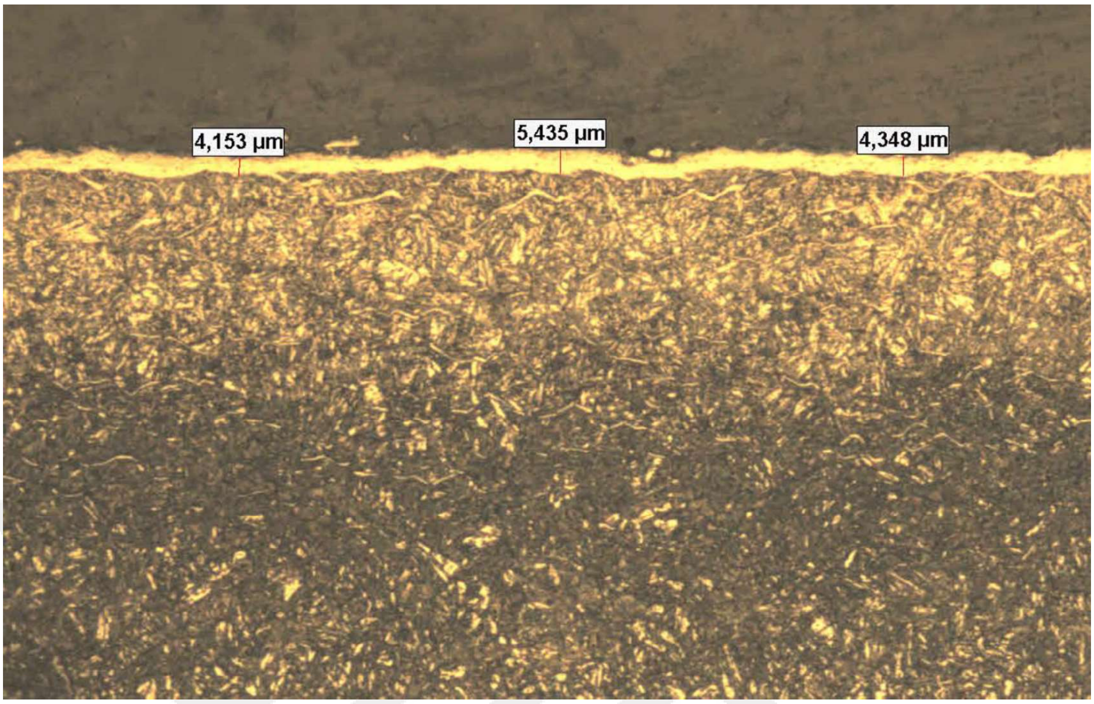


Figure 4.22 The microstructural image of 1.2344 tool steel after 12 hours of annealing in controlled atmosphere (optical microscope 500x)

After nitride, specimen is annealed for 12 hours in a controlled atmosphere, white layer thickness does not change and between 4,3-5,4 microns. It is seen on optical micrograph that oxidized layer does not appear.

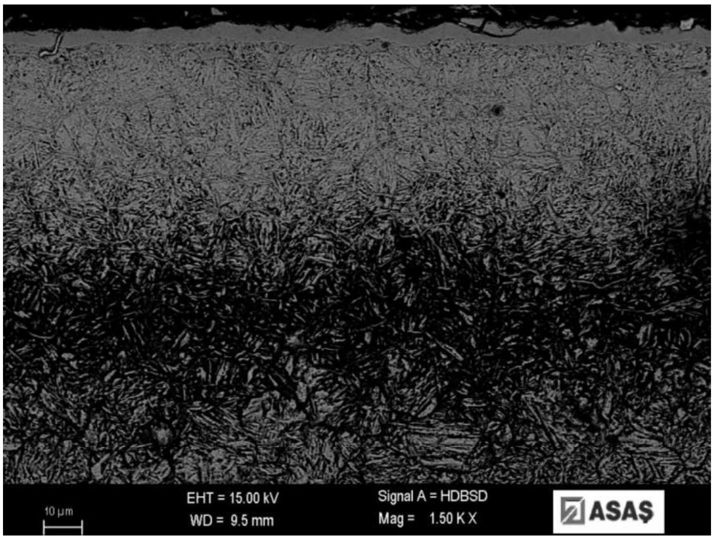


Figure 4.23 The image of specimen after 12 hours of annealing on controlled atmosphere with nitrogen gas (electron microscope)1.5KX

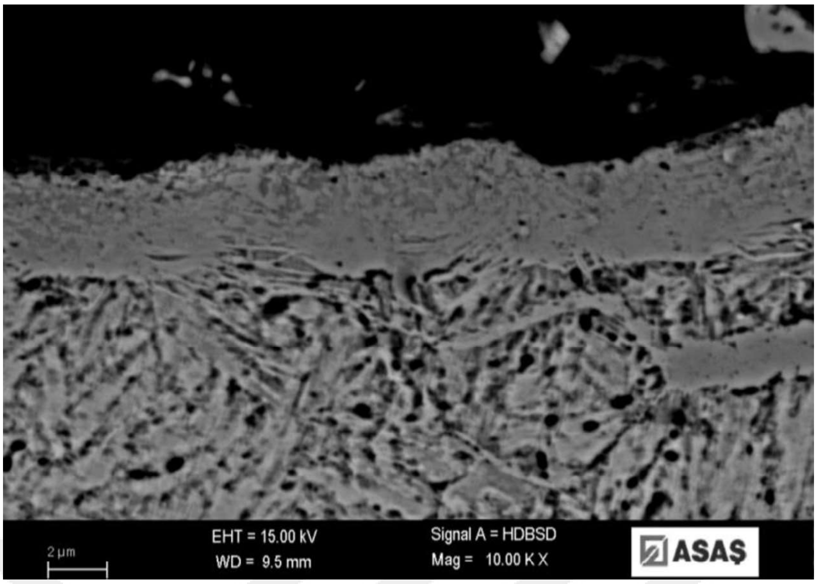


Figure 4.24 The image of specimen after 12 hours of annealing in controlled atmosphere with nitrogen gas (electron microscope) 10.K

On electron microscopy, the white layer of the annealed and nitrided specimen does not change after 12 hours of annealing and as an annealed-on air specimen, the oxide layer does not appear.

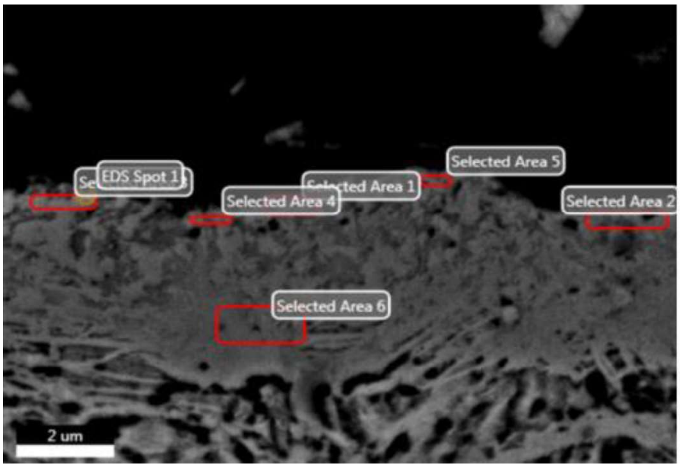


Figure 4.25 The SEM microstructural image of 12 hours annealed on controlled atmosphere specimen's selected areas

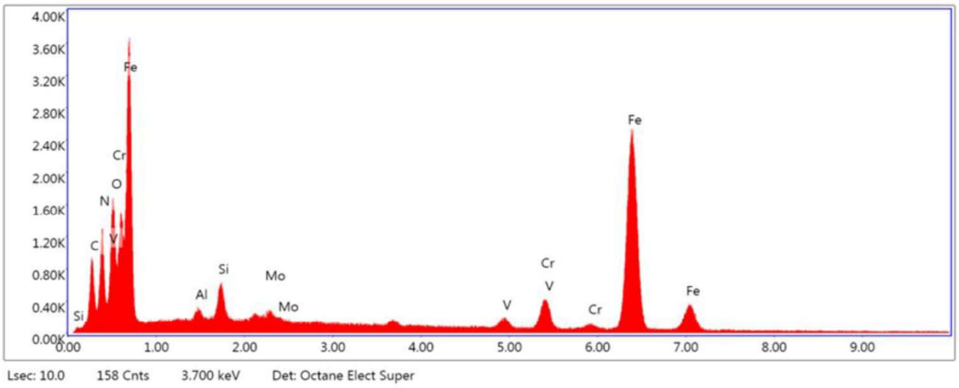


Figure 4.26 Edx Elemental analyze of annealed specimen 12 hours in protected furnace

Table 4.7 Elemental percentages selected area 1

Element	Weight%	Atomic%	Error%
CK	9	24,11	9,9
NK	8,39	19,28	9,59
OK	5,41	10,89	9,06
AlK	0,52	0,63	16,34
SiK	1,64	1,88	8,14
MoL	1,4	0,47	15,74
VK	1,72	1,09	13,6
CrK	5,5	3,41	5,71
FeK	66,4	38,25	2,81

Table 4.8 Elemental percentages selected area 2

Element	Weight%	Atomic%	Error%
CK	7,13	2,68	10,59
NK	6,92	17,21	9,7
OK	4,94	1,76	8,63
SiK	1,27	1,58	8,9
MoL	0,94	0,34	24,76
VK	0,8	0,55	20,59
CrK	5,44	3,64	5,82
FeK	72,55	45,24	2,79

Table 4.9 Elemental percentages selected area 3

Element	Weight%	Atomic%	Error%
CK	8,46	24,68	10,22
NK	9,09	22,74	9,55
AlK	0,3	0,38	26,83
SiK	1,07	1,33	9,54
MoL	1	0,37	25,03
VK	0,54	0,37	27,44
CrK	4,96	3,34	6,36
FeK	74,59	46,79	2,78

Table 4.10 Elemental percentages selected area 4

Element	Weight%	Atomic%	Error%
CK	10,03	28,38	9,88
NK	8,76	21,25	9,76
AlK	0,37	0,46	22,5
SiK	1,26	1,52	9,32
MoL	1,2	0,43	21,14
VK	0,6	0,4	24,97
CrK	5,15	3,37	5,95
FeK	72,63	44,19	2,75

Table 4.11 Elemental percentages selected area 5

Element	Weight%	Atomic%	Error%
CK	8,29	22,86	10,4
NK	8,1	19,16	9,63
OK	4,83	10	8,81
AlK	0,27	0,34	31,21
SiK	1,29	1,52	9,02
VK	1,01	0,66	17,45
CrK	5,54	3,53	5,83
FeK	70,67	41,93	2,78

Table 4.12 Elemental percentages selected area 6

Element	Weight%	Atomic%	Error%
CK	4,88	16,01	11,06
NK	7,71	21,66	9,3
SiK	0,97	1,35	10,73
MoL	1,06	0,43	24,21
VK	1,2	0,93	16,18
CrK	5,11	3,87	6,27
FeK	79,07	55,74	2,78

After annealing the nitrided specimen for 12 hours in a protected furnace, the electron microscope showed that 3 of the 6 selected areas have a maximum of 5% oxide element.

Even after 6 hours of annealing in an air furnace, the oxide layer forms and remains stable on the white layer. The thickness of the white layer begins to decrease after 12 hours of annealing, but the white layer was still available at the end of the 40 hours in the air furnace. In addition, diffusion layer starts to increase after 14 hours. At the similar experimental study, they found that white layer disappears after 30 hours and diffusion layer increases after 30 hours later [15].

Specimens annealed for 6 and 12 hours in an inert furnace are not affected in the same way as specimens annealed in an air furnace are. On the white layer, no difference can be seen using an optical microscope. When specimens are annealed in an air furnace for the same amount of time, their oxide layers are easily visible under an optical microscope. 3 and 6 selected areas on the white layer have a maximum of 5% oxide element, however this value is 20% for annealed specimens in an air furnace.

4.3. Abrasion Comparison Between with White Layer and without White Layer on Nitride Layer

Nitrided samples were subjected to abrasion test to check their abrasion resistance. Abrasion tests were carried out on a pin on plate machine the samples that are with white layer and without white later on before test are shown in Figure 4.27. and 4.30.

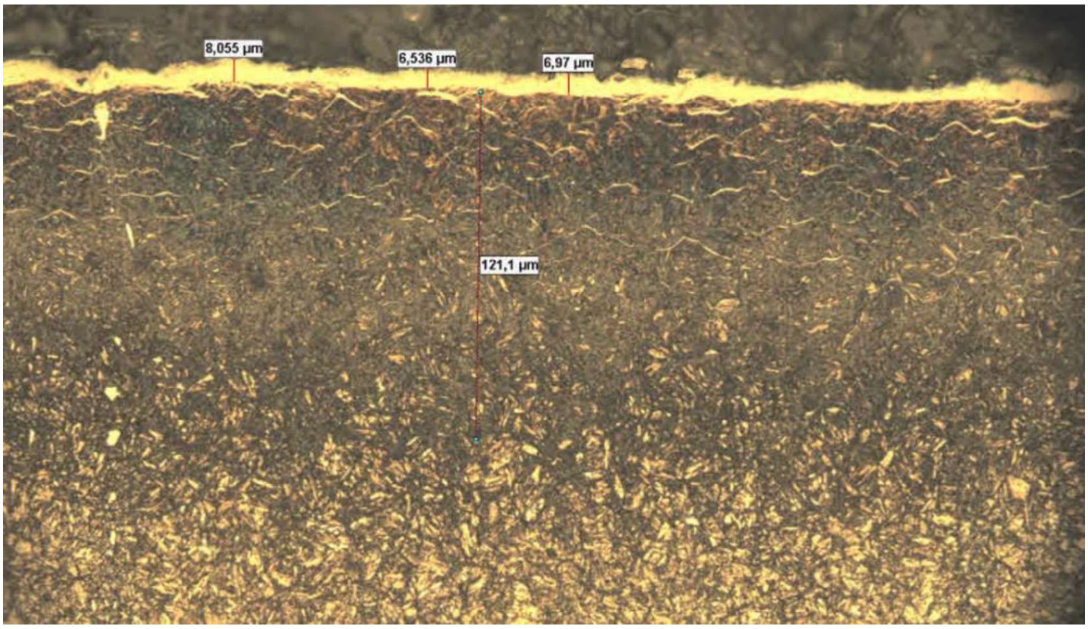


Figure 4.27 1.2344 nitrided specimen with white layer before abrasion test

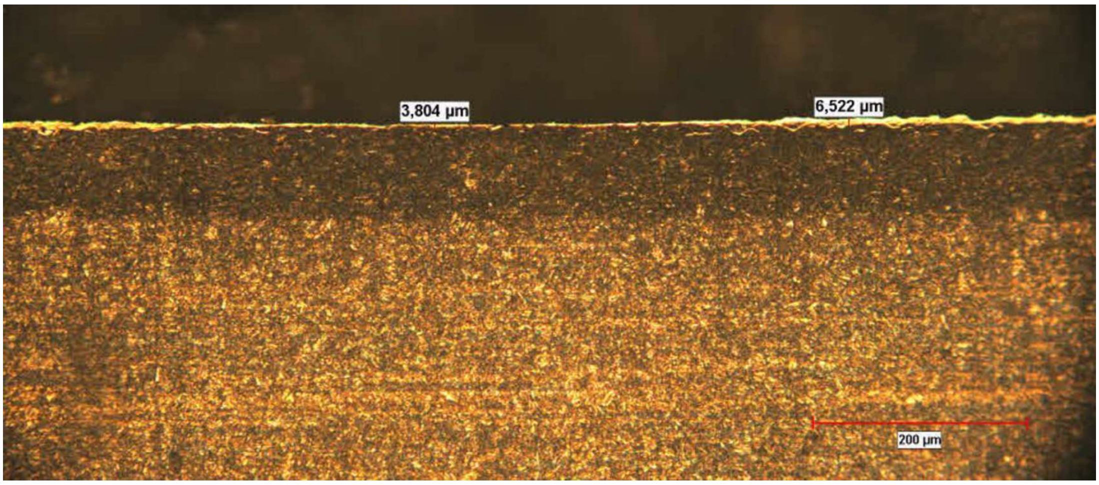


Figure 4.28 1.2344 nitrided specimen with white layer after 100 meters' abrasion (Etched)(400X) (Optical Image)

Abraded area’s white layer thickness drops to 3,8 microns from 6- 8 microns after 100 meters sliding distance as shown in Figure 4.28.

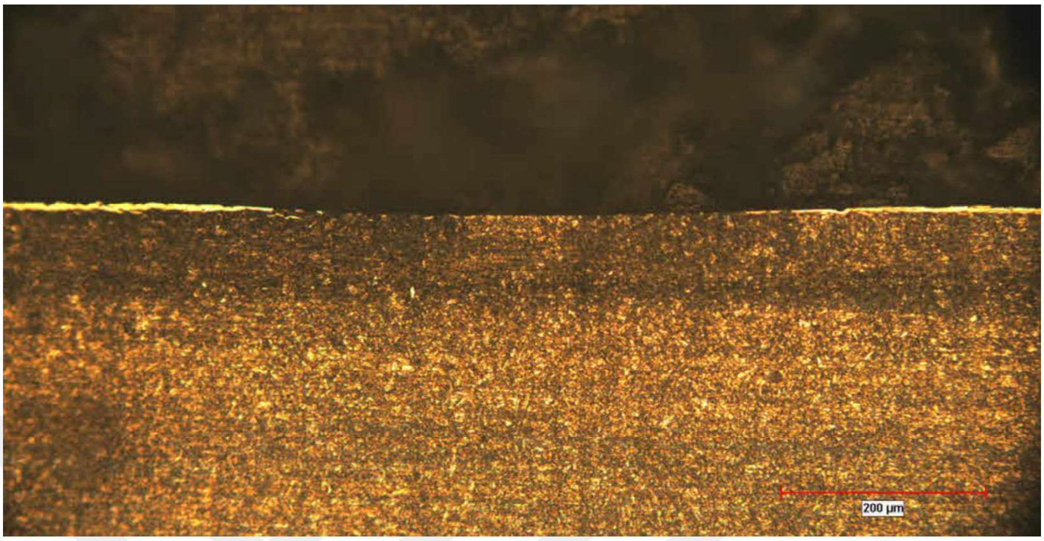


Figure 4.29 1.2344 nitrided specimen with white layer after 200 meters’ abrasion (Etched)(400X) (optical Imagine)

Abraded area’s white layer thickness is completely wear out from 6- 8 microns after 200 meters sliding distance as shown in Figure 4.29.

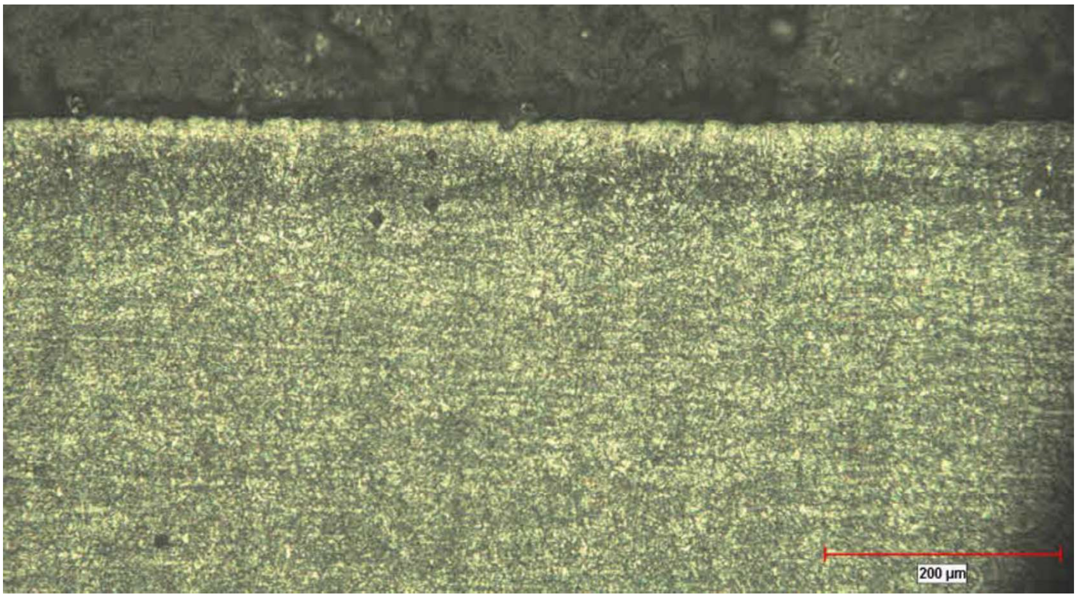


Figure 4.30 1.2344 nitrided specimen without white layer before abrasion (Etched) (Optical Imagine)

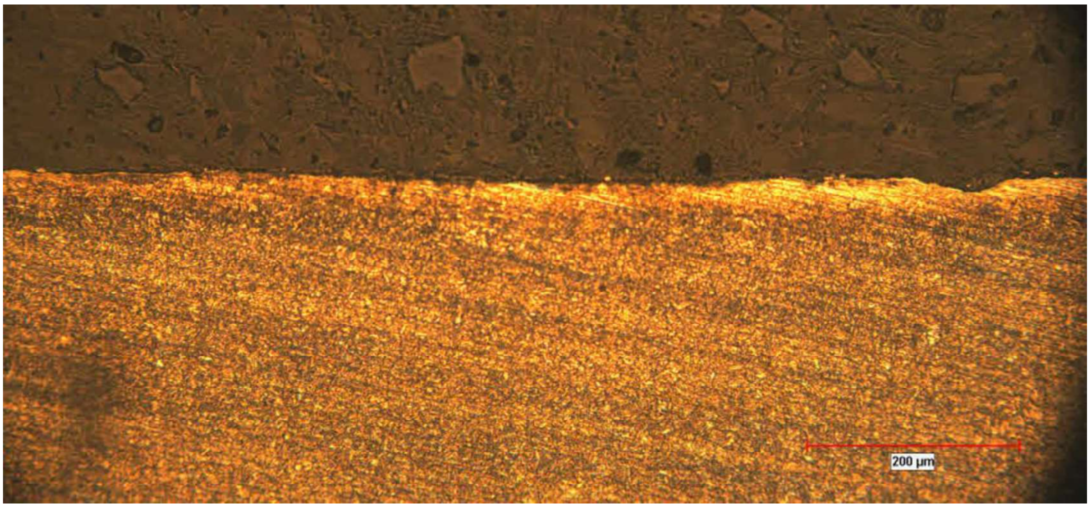


Figure 4.31 1.2344 nitrided specimen without white layer after 100 meters' abrasion (Etched) (Optical Imagine)

Abraded area's diffusion layer decreases and shown in Table 4.14 after 200 meters sliding distance and it's easily seen on Figure 4.31.

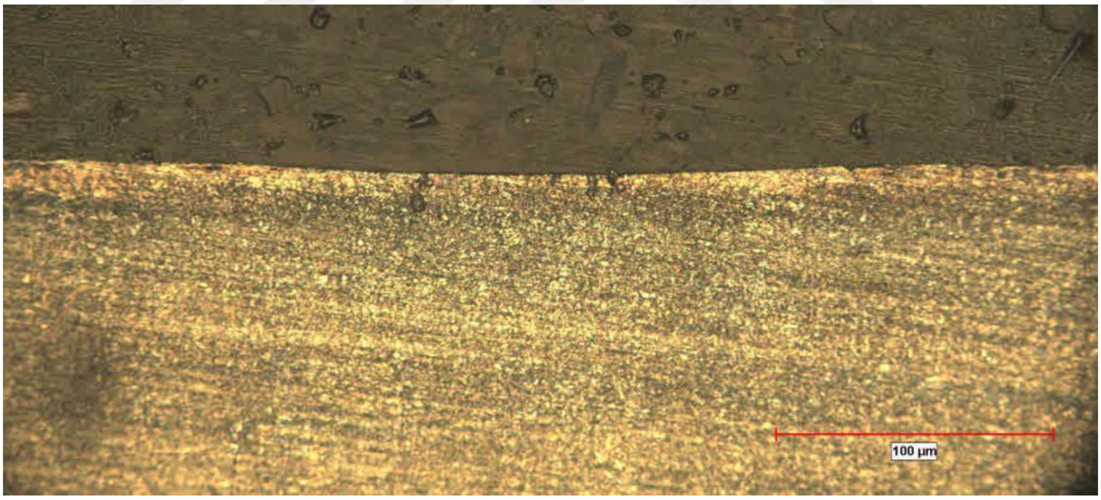


Figure 4.32 1.2344 nitrided specimen without white layer after 200 meters' abrasion (400X) (Optical Imagine)

Abraded area's diffusion layer decreases and shown in Table 4.14 after 200 meters sliding distance and it's easily seen on Figure 4.32.

As a conclusion, it is clearly understood from the abrasion test that white later have more resistance for abrasion. Two specimens were subjected to same load and distances (15N, 100meters-15N200 meters). Amount of abrasions are shown in Figure 4.27. ,4.28, 4.29., 4.30. and comparison are shown in Table 4.13.

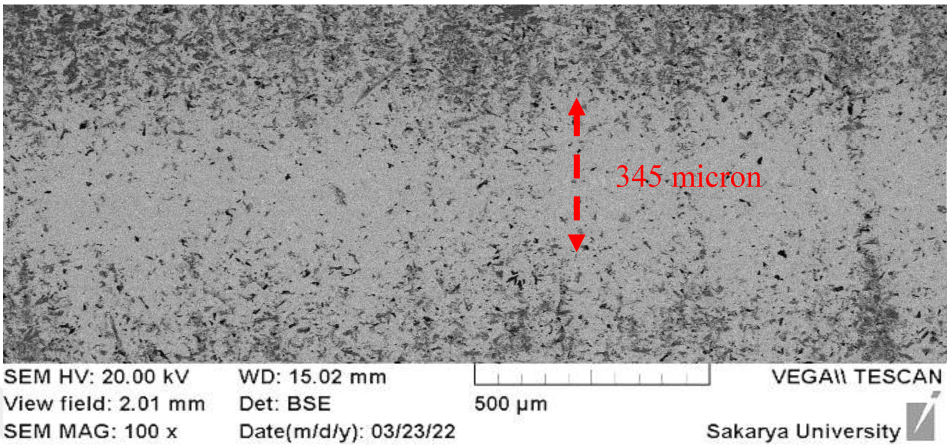


Figure 4.33 SEM image of the sample15N forced white layer and 100 meters sliding distance of pin on plate abrasion test

Specimen with white layer is abraded 345 microns after 100 meters sliding distance as shown in Figure 4.33.

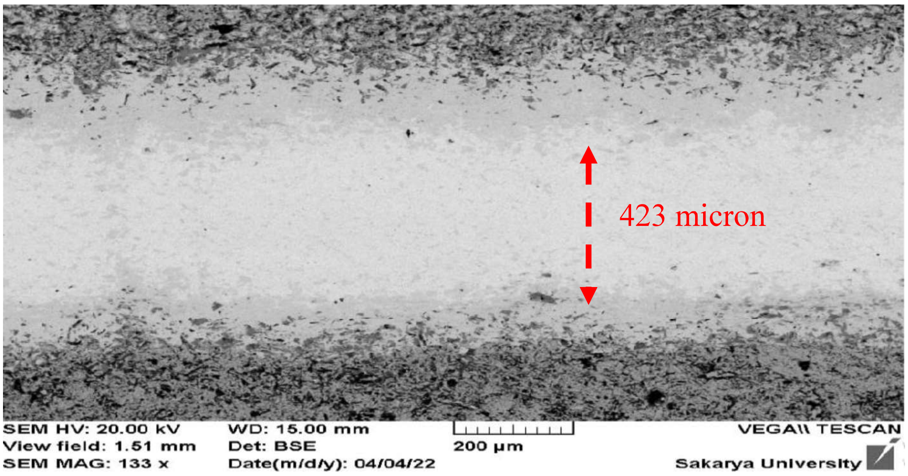


Figure 4.34 SEM image of the sample 15N forced with white layer and 200 meters' sliding distance of pin on plate abrasion test

Specimen with white layer is abraded 423-micron meters after 100 meters sliding distance as shown Figure 4.34.

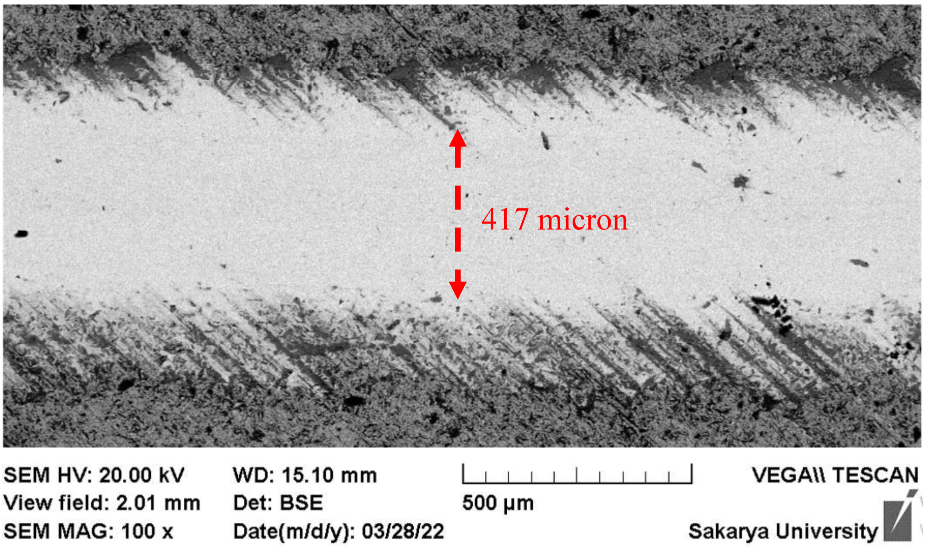


Figure 4.35 SEM image of the sample 15N forced without white layer and 100 meters' sliding distance of pin on plate abrasion test

Specimen without white layer is abraded 417-micron meters after 100 meters sliding distance as shown Figure 4.35.

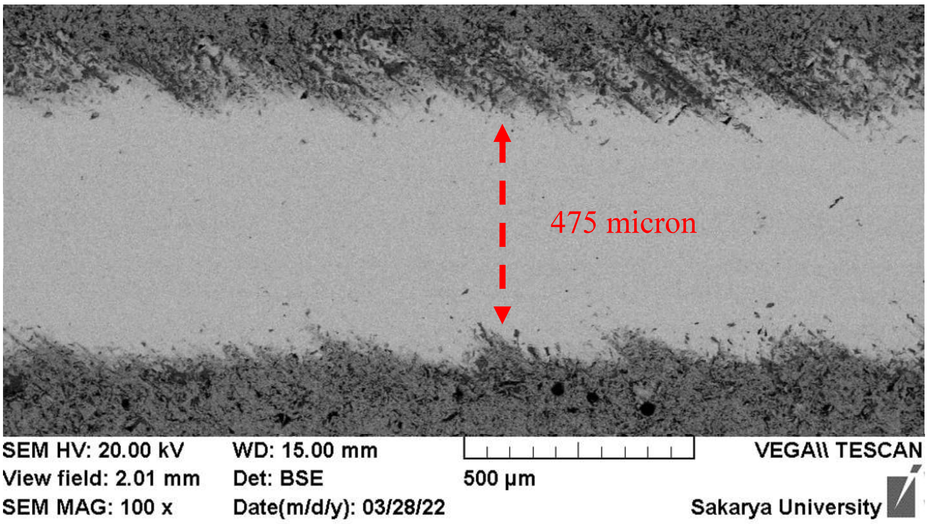


Figure 4.36 SEM image of the sample 15N forced without white layer and 200 meters sliding distance of pin on plate abrasion test

Specimen without white layer is abraded 475-micron meters after 200 meters sliding distance as shown Figure 4.36.

As a conclusion, it is clearly understood from the abrasion test that that the white later has more resistance for abrasion as shown in Table 4.13. White layer is desired for extrusion process in order to have more wear resistance.

Table 4.13 Amount of Abrasion Wide (micron)

	Amount of Abrasion Wide (micron meter)	
	With White Layer	**Without White Layer**
100 meters sliding	345	417
200 meters sliding	423	475

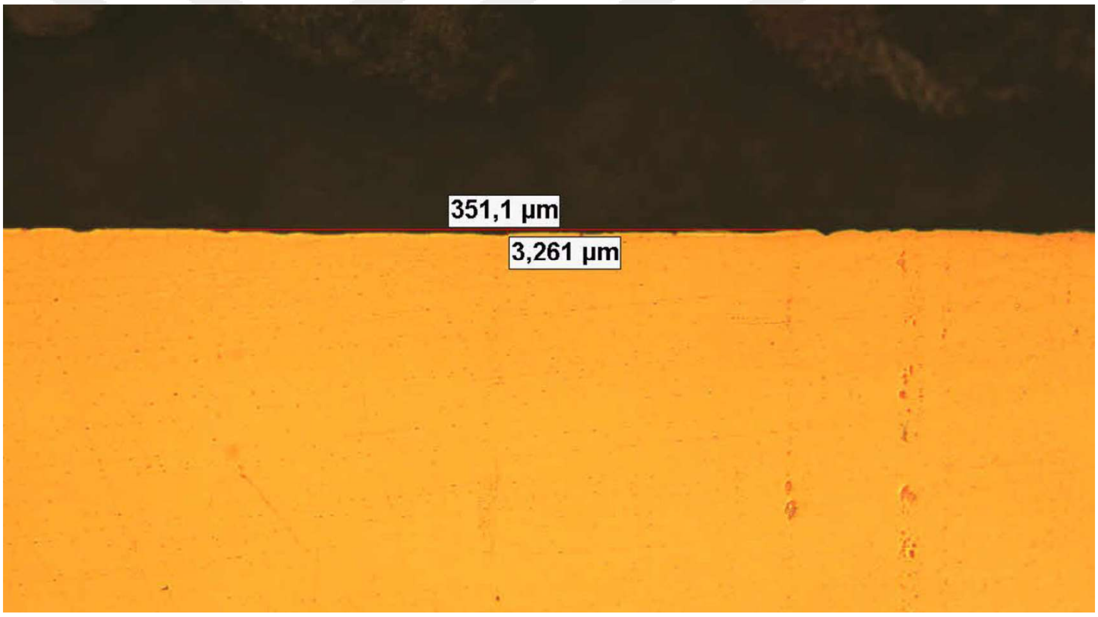


Figure 4.37 1.2344 nitrided specimen with white layer after 100 meters' abrasion (polished)

Specimen with white layer is abraded 351 microns wide and 3,261 microns' depth after 100 meters sliding distance which is shown in Figure 4.37.

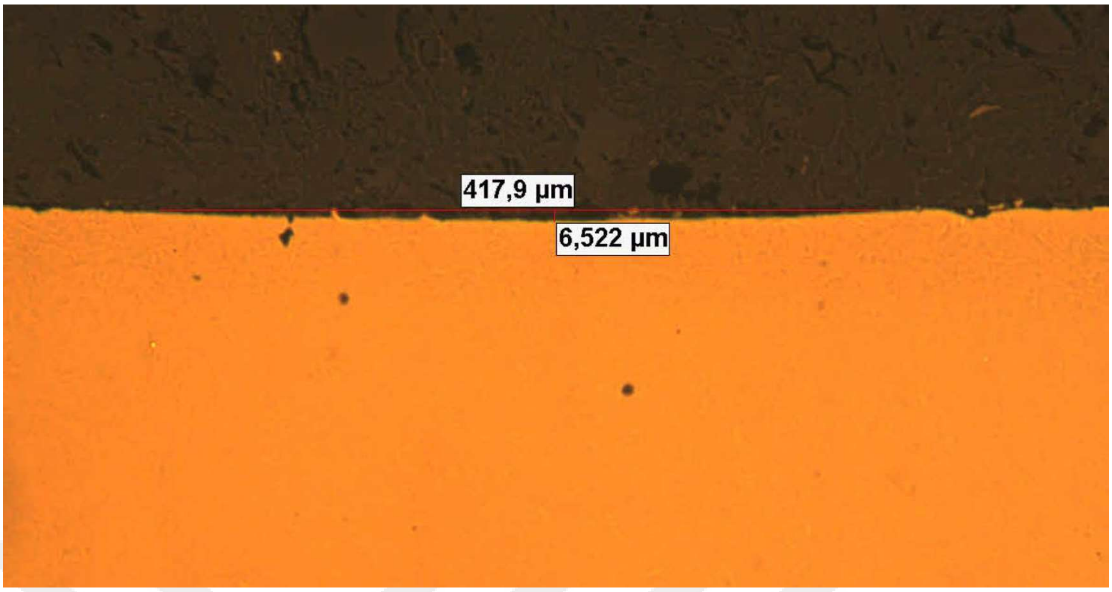


Figure 4.38 1.2344 nitrided specimen without white layer after 100 meters' abrasion test

Specimen without white layer is abraded 417 microns wide and 6.522 microns' depth after 100 meters sliding distance which is shown in Figure 4.38.

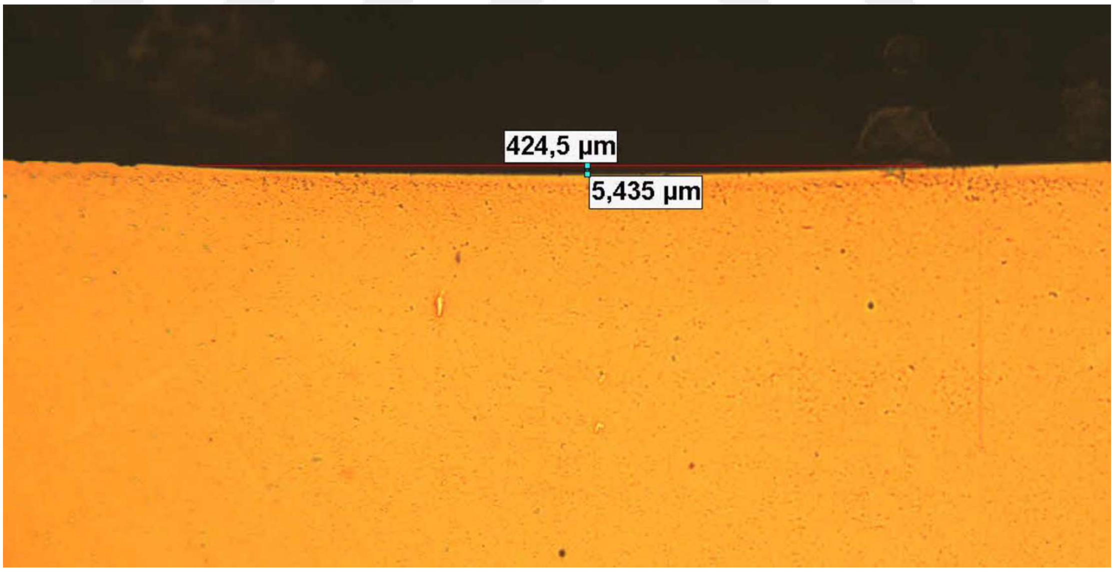


Figure 4.39 1.2344 nitrided specimen without white layer after 200 meters' abrasion test (polished)

Specimen with white layer is abraded 424,5 microns wide and 5,435 microns' depth after 100 meters sliding distance which is shown in Figure 4.39.

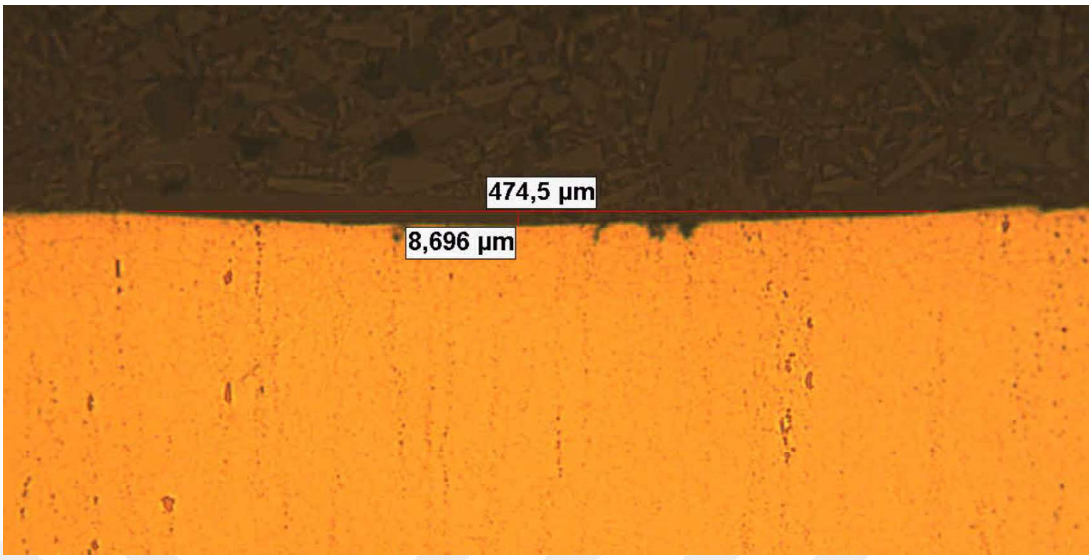


Figure 4.40 1.2344 nitrided specimen without white layer after 200 meters' abrasion test (polished)

Specimen with white layer is abraded 475,5 microns wide and 8,696 microns' depth after 200 meters sliding distance which is shown in Figure 4.40.

As a conclusion, it is clearly understood from the abrasion test that white later has more resistance for abrasion as shown in Table 4.14.

Table 4.14 Amount of Abrasion Depth (micron meter)

	Amount of Abrasion Depth (micron meter)	
	With White Layer	Without White Layer
100 meters sliding distances	3,26	6,52
200 meters sliding distances	5,43	8,69

Abrasion tests show that specimens with a white layer have higher abrasion resistance.

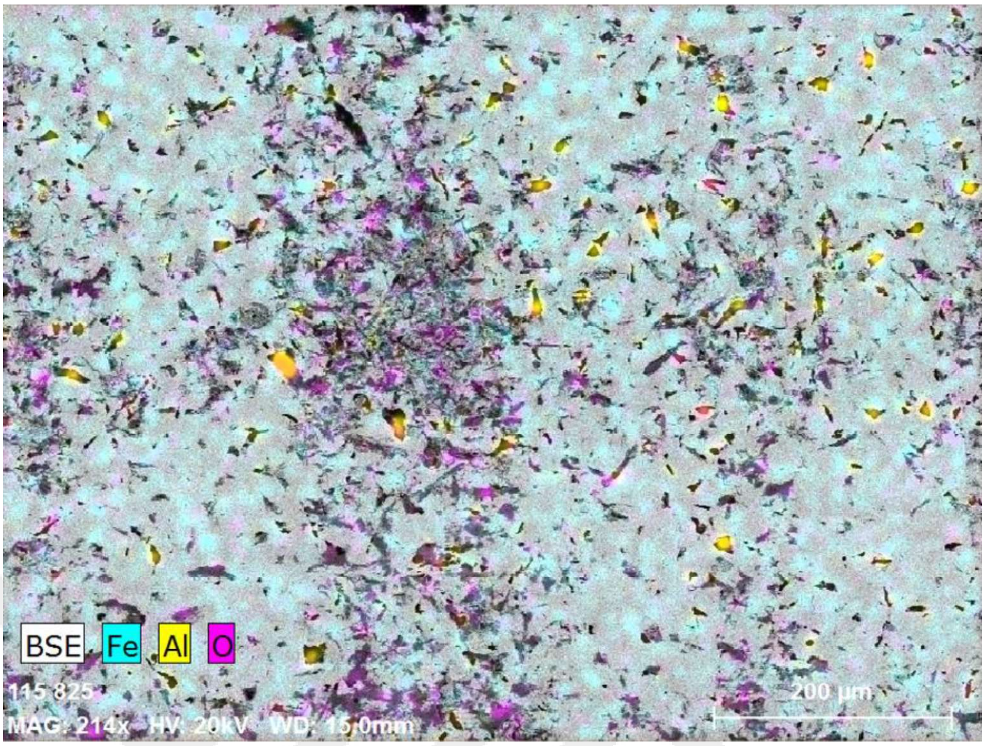


Figure 4.41 Element imagine map of abraded 1.2344 nitrided specimen with white layer

The aluminum oxide ball used in the 1.2344 nitrided specimen abrasion test is elementally examined. As a result, both the specimen and the aluminum oxide ball have been abraded, and the elements Al and O can be seen on the map in Figure 4.41.

CHAPTER 5

CONCLUSION AND FUTURE WORKS

5.1 Conclusion

The influence of annealing time on the nitride layer is investigated in this study using an air furnace and inert furnace is compared with air furnace for nitride layer of extrusion dies.

It has been understood from the experiment that after 12 hours of annealing the extrusion die, the white layer thickness begins to decrease and the diffusion layer begins to increase in the air furnace. The nitride layer is degrading in this case. The annealing time should not be longer than 12 hours.

Second, even after 6 hours of annealing in an air furnace, a stable oxide layer appears on the extrusion die. On the annealing process, when die reachs annealing temperature, die must be getting out of furnace for protecting from oxygen. The corrosion of the white layer is caused by the oxide layer. While aluminum is sliding through the surface, the rough surface might cause flaking, causing the die life ended earlier than expected.

The use of an inert furnace helps in the reduction of nitride layer oxidation. Even after being annealed for 12 hours, the nitride surface is essentially unaffected by oxidation. In this case, selected areas on white layer have a maximum of 5% oxide element. Nitrogen gas purge to oxygen gas, and oxygen is disconnected from the surface.

In the abrasion test study, even white layer is brittle, it is useful and provides more wear resistance on nitride layer of extrusion dies.

5.2 Future Work

Some other coating methods should be investigated instead of gas nitriding process to get more effective protection.

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