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FRICTION-WEAR CHARACTERISTICS OF PLASMA NITRIDED COLD WORK TOOL STEELS

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Abstract: D2, D6 and Calmax cold work tool steels were heat treated in vacuum hardening, made grinding and plasma nitrided in 75% N_2 + 25% H_2 atmosphere at 450 °C for 12 h at 2 mbar. Characterization of samples has been carried out by means of surface roughness, microstructure, microhardness and wear-friction. Wear-friction characteristics of samples have been investigated using a ball-on-disc tribosystem with a WC-Co ball. Plasma nitriding increased the surface roughness of samples. However, this effect was decreased with higher surface roughness of base metal. Fe_4N (γ'), Fe_3N (ε) and CrN phases were obtained on the surfaces of samples. Higher surface hardness was obtained in the plasma nitrided D2 and D6 steels. No significant mass loss was observed in the ball on-disc tribosystem. So, the wear of samples was characterized with the worn surfaces using a scanning electron microscope (SEM). Plasma nitriding improved the wear resistance and decreased the friction coefficient of steels. Plasma nitrided D2 steel showed the highest wear resistance, whereas plasma nitrided Calmax steel exhibited the lowest wear resistance. However, plasma nitrided Calmax tool steel had relatively lower friction coefficient than the other plasma nitrided steels. In general, brittle layer fractures have determined the friction coefficient.

Keywords: Cold work tool steels, Plasma nitriding, Microhardness, Friction coefficient, Wear surface characterization

Plazma Nitrürlenmiş Soğuk İş Takım Çeliklerinin Sürtünme-Aşınma Karakteristikleri

Öz: D2, D6 ve Calmax soğuk iş takım çelikleri vakum altında sertleştirildi, taşlandı ve 75% N_2 + 25% H_2 atmosferinde 450 °C sıcaklıkta ve 2 mbar basınç altında 12 saat süre ile plazma nitrürlendi. Numuneler yüzey pürüzlülüğü, mikroyapı, mikrosertlik ve aşınma-sürtünme testleri ile karakterize edildi. Aşınma-sürtünme karakteristiklerinin belirlenmesinde WC-Co bilyasına sahip ball-on-disk sürtünme-aşınma sistemi kullanıldı. Plasma nitrürleme numunelerin yüzey pürüzlülüğünü arttırmıştır. Ancak, bu etki temel malzemenin daha yüksek yüzey pürüzlülüğü ile azalmıştır. Plazma nitrürlenmiş numunelerin yüzeylerinde Fe_4N (γ'), Fe_3N (ε) ve CrN fazları elde edilmiştir. Plazma nitürlenmiş D2 ve D6 çeliklerinde daha yüksek yüzey sertlikleri elde edilmiştir. Ball-on-disk sürtünme-aşınma sisteminde numunelerde önemli bir kütle kaybı meydana gelmemiştir. Bu yüzden, numunelerdeki aşınma taramalı elektron mikroskobu (SEM) ile karakterize edilmiştir. Plazma nitrürleme çeliklerin aşınma dayanımını iyileştirmesinin yanında sürtünme katsayısını da düşürmüştür. Plazma nitürlenmiş D2 çeliği en yüksek aşınma dayanımına sahip olurken, plazma nitrülenmiş Calmax çeliği ise en düşük aşınma dayanımı sergilemiştir. Ancak, plazma nitrürlenmiş Calmax çeliği diğer plazma nitrülenmiş çeliklere nazaran nispeten daha düşük sürtünme katsayısına sahip olmuştur. Genel olarak, aşınma ürünü kırılgan nitrür parçacıkları sürtünme katsayısını belirleyici rol oynamıştır.

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Anahtar Kelimeler: Soğuk iş takım çelikleri, Plazma nitrürleme, Mikrosertlik, Sürtünme katsayısı, Aşınma yüzeyi karakterizasyonu

1. INTRODUCTION

Cold work tool steels encounter to many different types of forces such as compressive forces, friction induced shear forces, thermal loads (friction energy), chemical loads (environment) during their lifetime (Okonkwo et al., 2016; Groche and Christiany, 2013; Grzesik, 2017; Toboła et al., 2017; Lind et al., 2010). If the tool steel is not durable enough, it can cause damages such as wear, galling/pick-up, chipping, cracking/breakage and plastic deformation. Heat treatment is the most common process to avoid the potential damages. Particularly, contact surface between tool steel and work piece encounters with the largest force. Therefore, surface hardening of the tool steels has critical importance. Plasma nitriding offers many advantages for surface hardening of tool steels. Plasma nitriding process is simply the diffusion of nitrogen atoms to the surface of the material in the vacuum environment at a temperature range of 400-560 °C. Plasma nitriding changes the microstructure of the surface, increases surface hardness and decreases toughness as well (Conci et al., 2014; Alves et al., 2007; Aydın et al., 2013; Zeghni and Hashmi, 2004). The plasma nitriding process takes place below the tempering temperature. In this process, high reproducibility can be achieved in mass production because it does not cause mechanical and dimensional changes in tool steel (Jurci and Panjan, 2006). Plasma nitriding can be applied to all ferrous metals. The toughness of the nitride layer is sufficient for tool steels. Plasma nitriding could increase the polishing and welding capabilities and the corrosion resistance of the materials. In this process, a higher surface roughness can be obtained than the other nitriding methods. M.Uma Devi et al. (1999) reported that plasma nitriding was a suitable method for reducing adhesive wear of D2 cold work tool steel. Akbari et. al. (2010) and Tillmann et. al. (2019) reported that plasma nitriding process increased the surface roughness of the tool steels. Doan et. al. (2016) reported that plasma nitriding increased the surface hardness, wear strength, friction coefficient of 42CrMo4 steel. Manfridini et. al. (2017) stated that nitriding zone needs to identify as two zones, which are white layer and diffusion zone. In this study, wear-friction characteristics of plasma nitrided cold work tool steels (D2, D6 and Calmax) were investigated.

2. MATERIALS AND METHODS

In this study, microstructure, microhardness, the surface roughness, and friction-wear characteristics of the plasma nitrided cold work tool steels (Calmax (1.2358), AISI D2 (1.2379 or Sverker21), AISI D6 (1.2346 or Sverker3)) were investigated. Chemical compositions of cold work tool steels used in experimental studies are given in Table 1.

Table 1. Chemical composition of cold work tool steels used in this study

Trade Name	AISI /W.-Nr.	Chemical composition (wt.%)						
		C%	Si%	Mn%	Cr%	Mo%	W%	V%
Sverker 21	D2/1.2379	1.55	0.30	0.40	11.80	0.80	-	0.80
Sverker3	D6/1.2436	2.05	0.30	0.80	12.70	-	1.10	-
Calmax	- /1.2358	0.60	0.35	0.80	5.30	-	-	0.20

The samples were obtained by slicing from a rod with turning process to 29 mm diameter and 10.5 mm thickness dimensions. In order to prevent possible distortions, heat treatment was performed by applying 3 tempering steps and an average hardness of 58-60 HRC was obtained in each sample. After the heat treatment, the samples were grinded from top and bottom, and the sample thicknesses were reduced to 10 mm. Plasma nitriding process for all samples were carried out in 75% N_2 + 25% H_2 gas mixture at 450 °C for 12 h at a pressure of 2 mbar. Finally, the samples were cooled to room temperature in the plasma nitriding vacuum furnace.

The surface roughness of samples was determined by taking average of six measurements from different regions of the surface. One of the plasma nitrided samples was used for the microstructure characterization and microhardness measurements. The cross-section of samples was ground with SiC emery papers numbered 180-320-600-1000 and finally polished through 6 μm and 1 μm diamond solution. After polishing, the samples were etched with 3% Nital for 10 s to 60 s depending on the cold work tool steel and then examined by optical microscope. Vickers hardness tester was used for the microhardness hardness measurements. In microhardness measurements, 50 g load was performed for 10 s and hardness measurements were performed as 3 different line and 10 measurements were taken from every line with 20 μm step. Schematic illustration for the hardness measurement on the cross-section of samples can be seen Fig. 1.

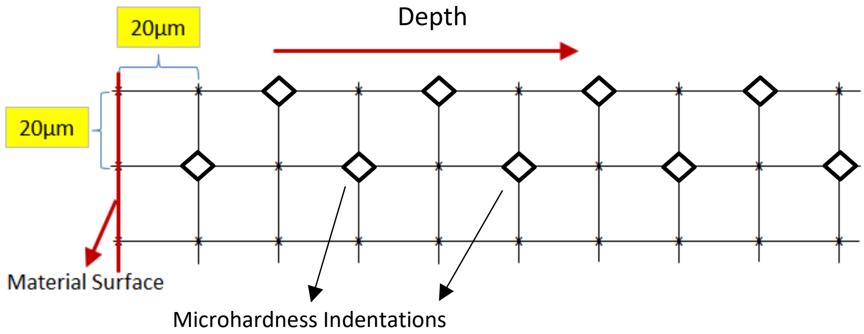


Figure 1:
Vickers hardness measurements in a line on cross-section of the samples

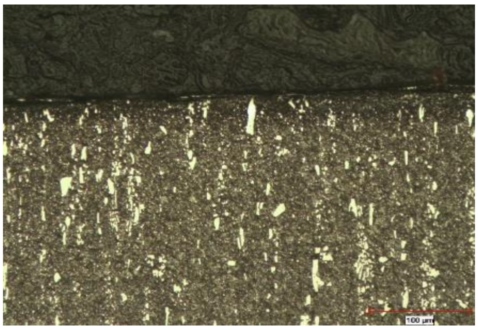
In order to determine wear and friction characteristics of plasma nitrided samples, ball-on-disc test was performed in a way to collect data dynamically under dry friction conditions. Friction coefficient was measured every 4 s and recorded. Samples were used as disc and tungsten carbide (WC/Co (94% WC - 6% Co)) ball with 5 mm diameter was used for the ball. Tungsten carbide balls were placed on top of the plasma nitrided sample to follow a trace, which is diameter of 22.5 mm. Preliminary studies were carried out to determine the appropriate loading during wear tests. Chatter instability arose during the wear tests for loads higher than 1.5 kg. And, this chatter instability significantly affected the wear test results. So, the test load was set at 1.5 kg to avoid chatter instability. Plasma nitrided and unnitrided samples were performed a 4-hour on ball on-disc wear testing at a load of 1.5 kg. The weight loss of samples was measured with the precision electronic balance having 10^{-4} g per hour in a total duration of 4 h. The wear traces of the samples were characterized by Scanning Electron Microscope (SEM). In addition, X-Ray Diffraction (XRD) analysis were conducted for the plasma nitrided samples to determine nitride phases in the microstructure.

3. RESULTS AND DISCUSSION

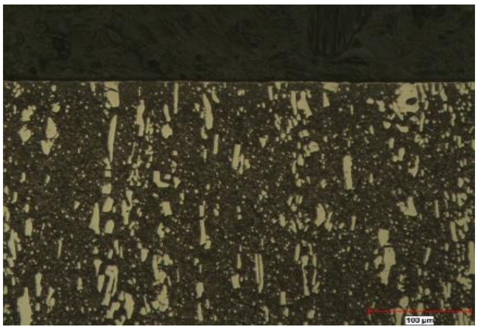
The surface roughness measurements of the test samples are given in Table 2. It was observed that surface roughness values generally increased after plasma nitriding process. However, the surface roughness value of D2 tool steel, which has the highest surface roughness value before plasma nitriding, remained almost same. The increase in the surface roughness through the plasma nitriding process is much more noticeable when the initial surface roughness of the base metals is low.

Table 2. Surface roughness measurements of the samples (PN: Plasma nitrided; UN: Unnitrided; Av.: Average; SD: Standard Deviation)

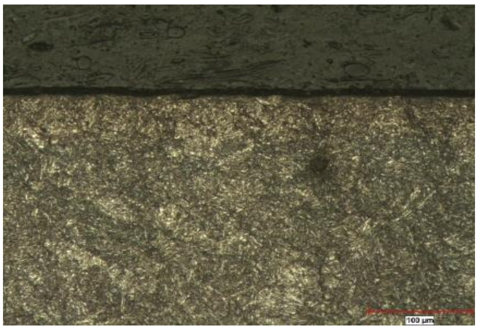
D6 (Ra) [µm]			D2 (Ra) [µm]			Calmax (Ra) [µm]		
No	**UN**	**PN**	**No**	**UN**	**PN**	**No**	**UN**	**PN**
1	0.46	0.64	1	1.14	1.26	1	0.60	0.62
2	0.46	0.62	2	2.18	1.90	2	0.58	0.68
3	0.46	0.58	3	2.28	2.32	3	0.60	0.70
4	0.44	0.62	4	2.62	2.20	4	0.62	0.68
5	0.40	0.56	5	1.92	2.48	5	0.58	0.66
6	0.40	0.54	6	2.06	2.14	6	0.56	0.64
Av.	0.44	0.59	Av.	2.03	2.05	Av.	0.59	0.66
SD	0.03	0.04	SD	0.5	0.43	SD	0.02	0.03



a.



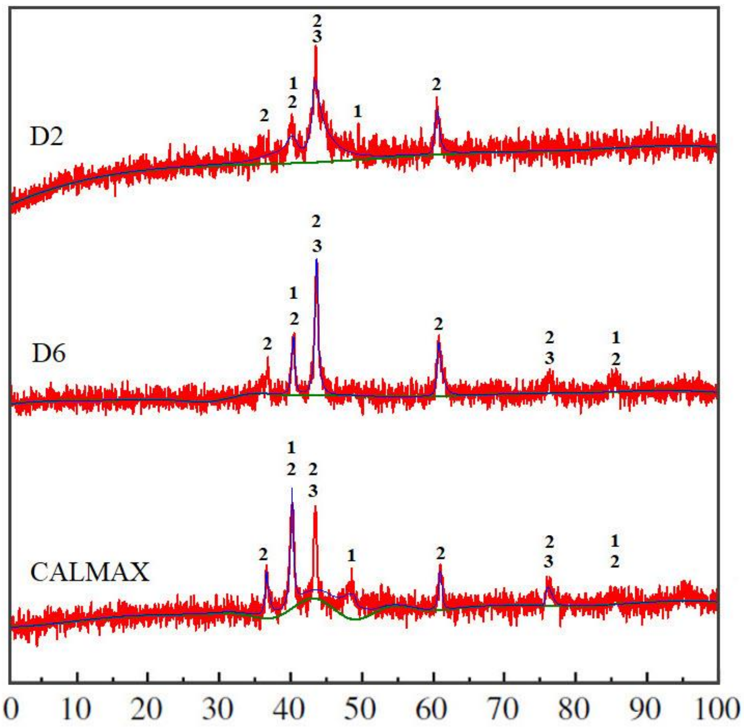
b.



c.

Figure 2:
Optical microscope images (x200)
***a.** Plasma Nitrided D2 steel* ***b.** Plasma Nitrided D6 steel* ***c.** Plasma Nitrided Calmax steel*

The cross-sectional microstructure images of the test samples can be seen in Fig. 2. The D2 and D6 tool steels, which have higher carbon and alloying elements, contain relatively coarse carbide particles, while the Calmax steel, which has lower carbon and alloying elements, has fairly finer carbides. In microstructural images, the diffusion layer was determined more apparent in the D2 and Calmax tool steels, while the diffusion layer in the D6 tool steel could not be observed clearly. Due to the relatively higher carbon and alloying element content in D6 tool steel, the diffusion layer could not be displayed by the existing etching process. However, microhardness measurements may display the depth of the nitrided diffusion layers. Nitride layer and diffusion zone of the nitrided iron-based materials consists of various nitride phases, such as γ’: Fe_4N and ε: Fe_3N and high amount of nitride precipitates (Mashregh et al., 2013; Ahangarania et al., 2009). According to XRD results in this study, Fe_4N (γ'), Fe_3N (ε) and CrN phases were determined in the microstructure of the test samples (Fig. 3).



1 – Fe_4N 2 – Fe_3N 3 – CrN

Figure 3:
XRD results of the nitrided samples at a glancing incident angle 2°

The microhardness values of the test samples measured in the depth direction from the surface are given in Fig. 4. Higher surface hardness was obtained in plasma nitrided D2 and D6 cold work tool steels, which contain higher carbon content and alloying elements. The surface hardness of the Calmax steel, which has lower carbon content and alloying element, was determined relatively low. However, according to the results obtained from the microhardness measurements, the hardening of the nitride layer can be said to be relatively better in Calmax tool steel: it had a higher hardening depth than the other tool steels. This can be attributed to the lower carbon and alloying element contents in Calmax cold work tool steel. In this steel, the nitrogen atoms during plasma nitriding process have been diffused more easily into the microstructure.

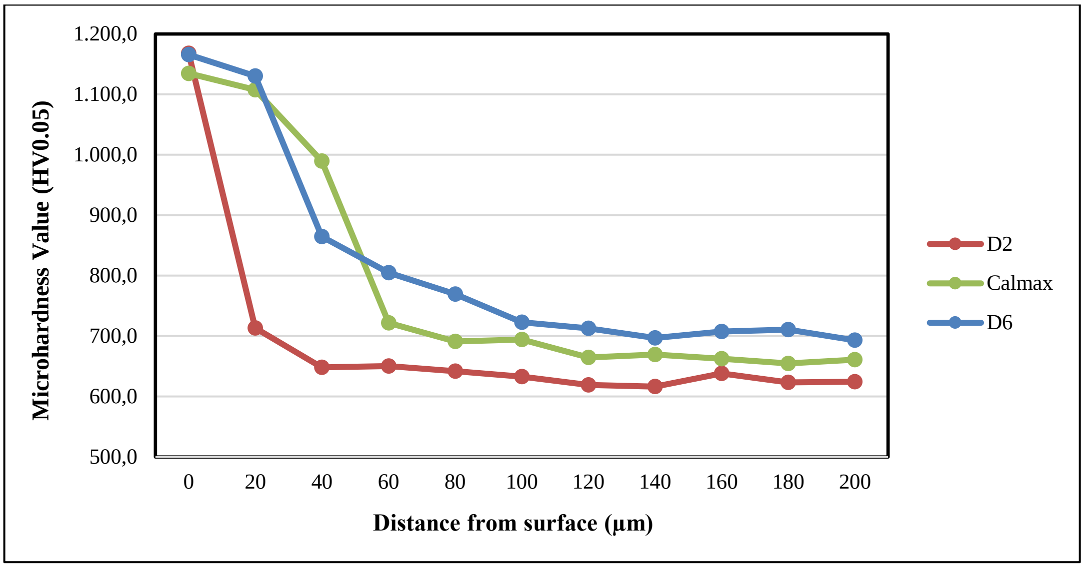


Figure 4:
Microhardness profiles measured along depth on the cross-section of the test samples

Wear tests were carried out in ball-on-disc experimental setup for 4 hours and weight measurements were conducted to determine the mass loss and evaluate the wear amount. But, as a result of the tests, no significant mass loss was observed in the tests to characterize the wear of the test samples because the wear amount caused by just the point contact of the abrasive ball was so much low. Therefore, the wear characteristics of the test samples were examined by evaluating the friction coefficients and worn surfaces. Significant deviations in the friction coefficient graphs of the samples were caused by disassembling and reattaching the samples for mass loss measurements. The contact point of the abrasive ball on the wear trace of the samples after disassembling and reattaching could not be matched to the same point. The friction coefficient of the unnitrided D6 tool steel was initially quite low (Fig. 5). The initial low friction coefficient in this sample may be attributed to the plastic deformation occurring in the contact point of the ball through sudden initial loading. With the increase of wear, the friction coefficient of the unnitrided D6 tool steel has increased gradually up to almost 1 (Fig. 5). This increase in the friction coefficient may associated with the increase of the surface roughness through wearing and the non-removal of the worn particles from the system (dry friction conditions). In the case of the plasma nitrided D6 tool steel, the initial friction coefficient was quite high, because brittle nitrided layer was broken to worn particles in the contact point of the ball through sudden initial loading (Fig. 6). The friction coefficient in this sample was generally around 0.8. From Fig. 7, it can be clearly seen that plasma nitriding process increased the wear resistance of D6 cold work tool steel. The wear depth was greater in the unnitrided sample, whereas it was shallower in plasma nitrided sample. In addition, the plasma nitrided sample exhibited a wear characteristic of hard layer exfoliation, while the the unnitrided sample exhibited a wear characteristic in the form of scratching. This wear characteristic of the unnitrided sample may be attributed to pull-out of coarse carbides in the microstructure. These carbides taken out from the microstructure increased the wear amount in this sample.

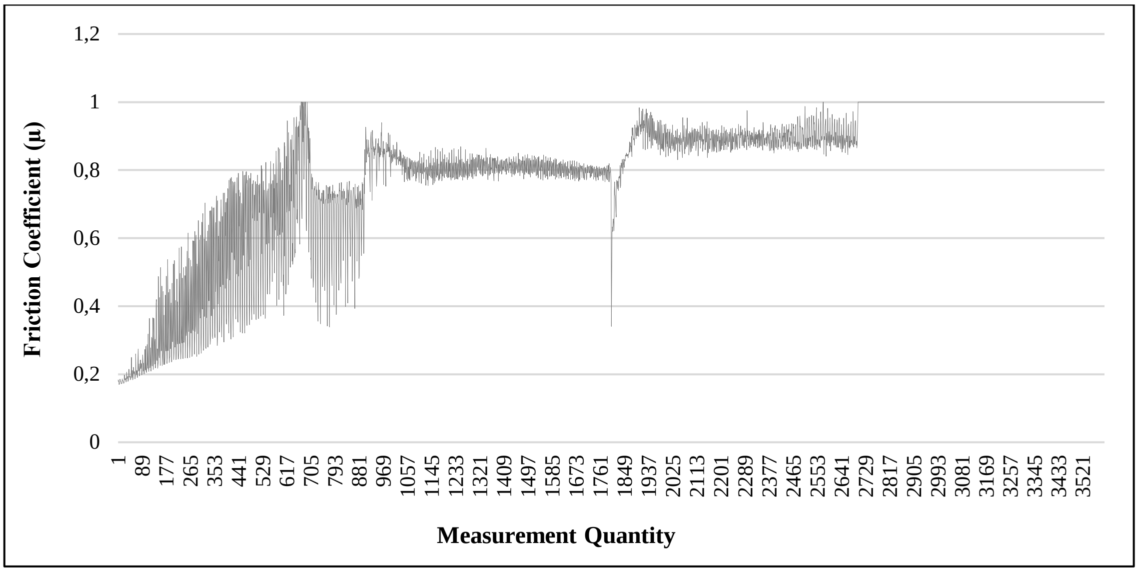


Figure 5:
Friction coefficient variation of unnitrided D6 tool steel

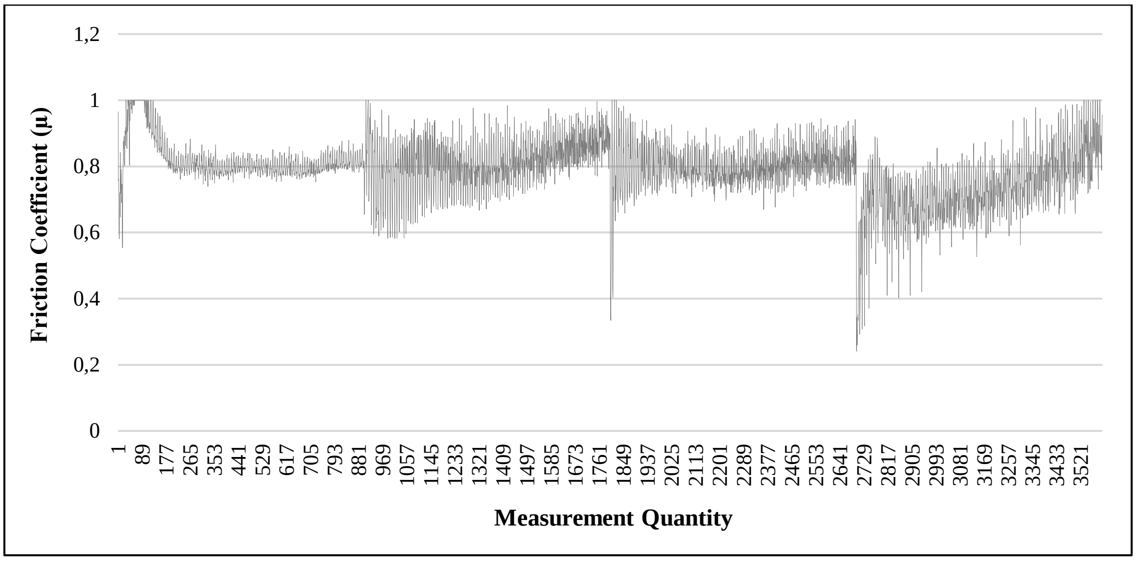
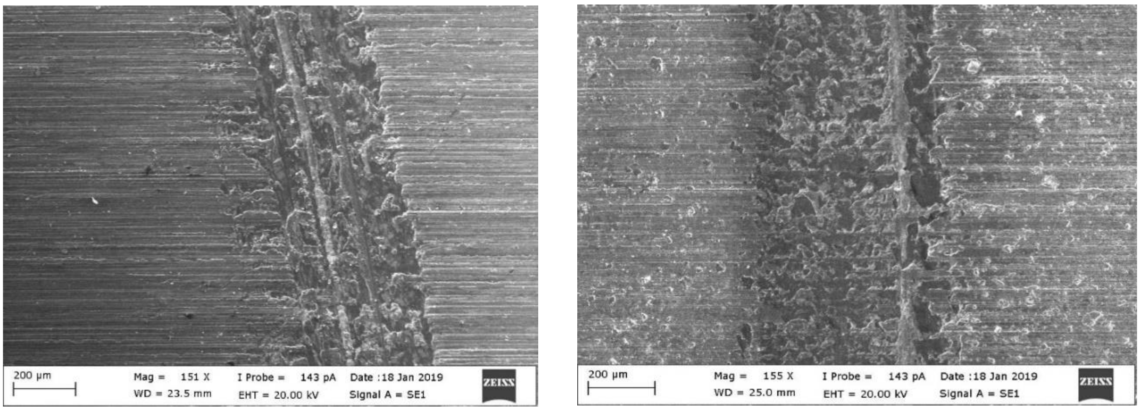


Figure 6:
Friction coefficient variation of plasma nitrided D6 tool steel



a. *b.*

Figure 7:
*SEM images of **a.** unnitrided D6 tool steel, **b.** plasma nitrided D6 tool steel*

The graph of the friction coefficient of the unnitrided D2 tool steel can be seen in Fig. 8. The initially low friction coefficient increased to about 1 with increasing wear. In general, compared with the unnitrided D6 tool steel, a higher friction coefficient was obtained with this sample. When the wear trace of this sample was examined, wear has been exacerbated locally (Fig. 10-a). In Fig. 10-a, it is also seen carbide-induced marks in this wear zones. This wear track explains the higher friction coefficient. However, it can be said that the wear resistance of this sample is higher than that of the D6 tool steel in this tribosystem (Fig. 7-a and Fig. 10-a). The secondary molybdenum and vanadium carbide precipitates in the microstructure of this sample have increased the wear resistance of the D2 tool steel. On the other hand, the plasma nitriding process has decreased the friction coefficient relatively and increased the wear resistance of D2 tool steel (Fig. 9 and Fig. 10-b).

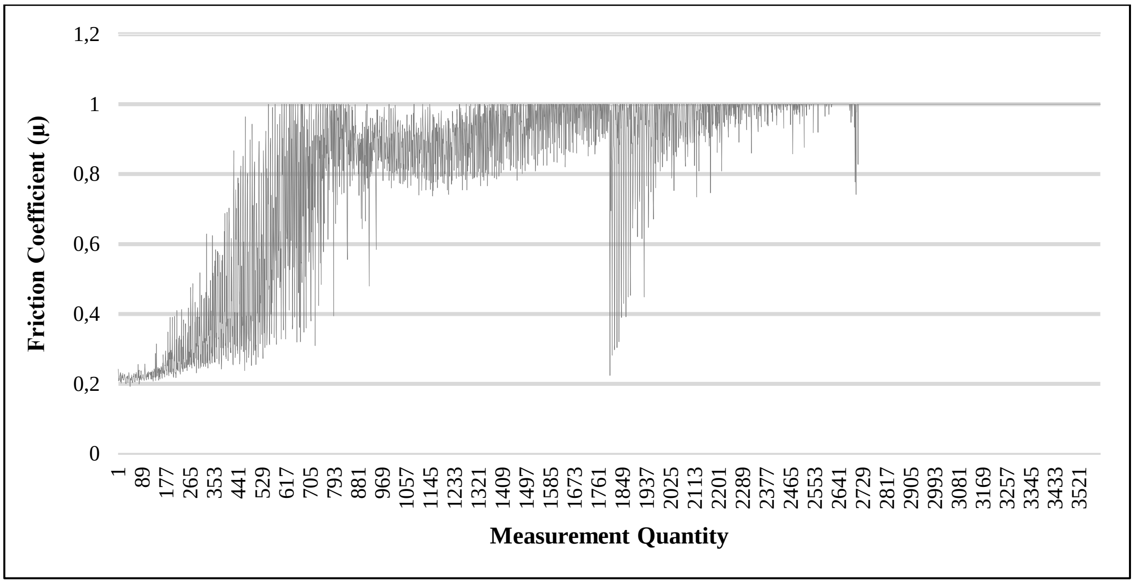


Figure 8:
Friction coefficient variation of unnitrided D2 tool steel

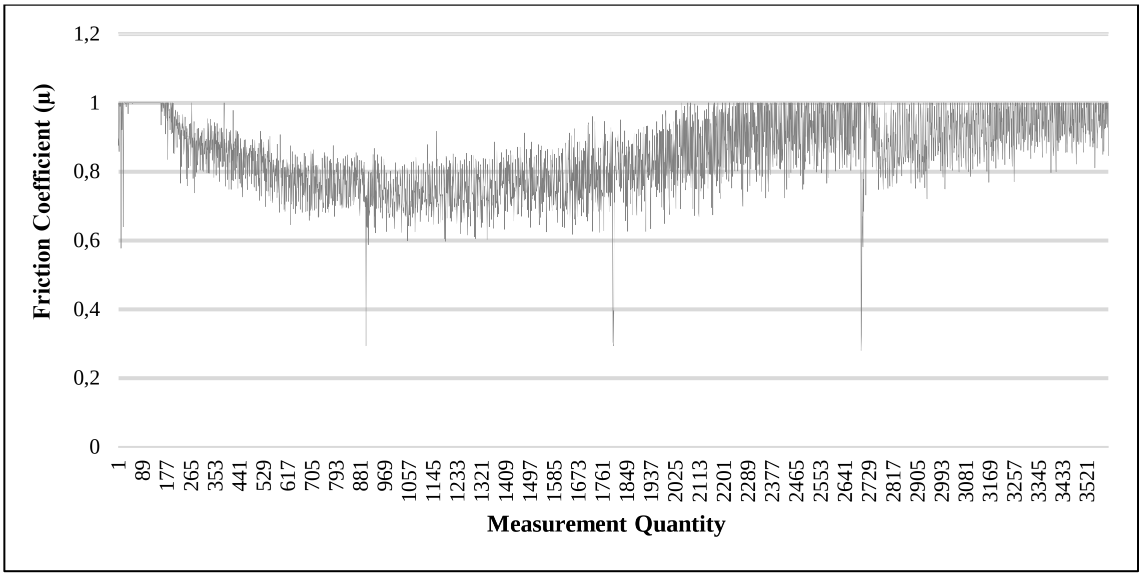
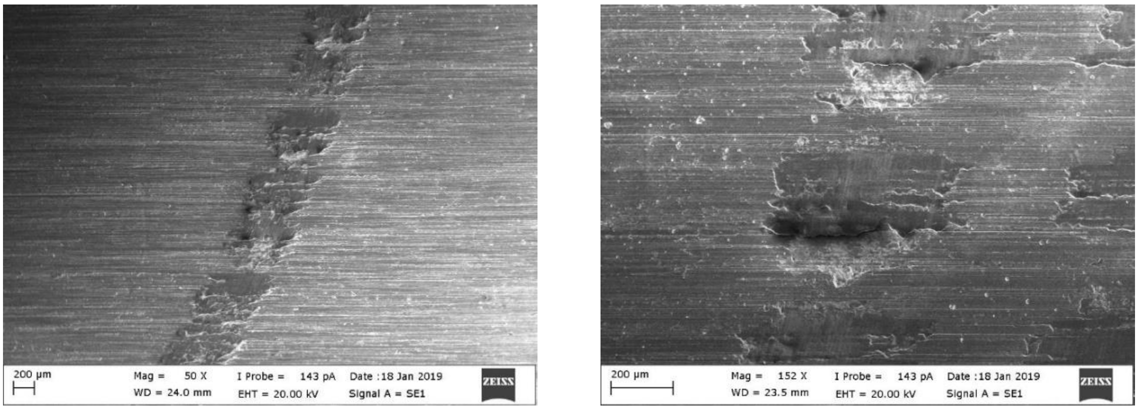


Figure 9:
Friction coefficient variation of plasma nitrided D2 tool steel



a. *b.*

Figure 10:
*SEM images of **a.** unnitrided D2 tool steel, **b.** plasma nitrided D2 tool steel*

Fig. 11 shows that the friction coefficient of the unnitrided Calmax tool steel was about 0.8. The friction coefficient of this steel has reached the regime in a much shorter time. This is related to the finer carbides in the microstructure of this steel unlike the other steels. In this steel, Fig. 12 shows that the friction coefficient after plasma nitriding was generally lower than that of the other steels. However, from Fig. 13, it cannot be said that plasma nitriding process increased the wear resistance of Calmax tool steel in this tribo-system. The wear traces in this sample do not contain brittle exfloation marks. As a matter of fact, surface hardness after plasma nitriding in this steel was relatively lower compared to other samples.

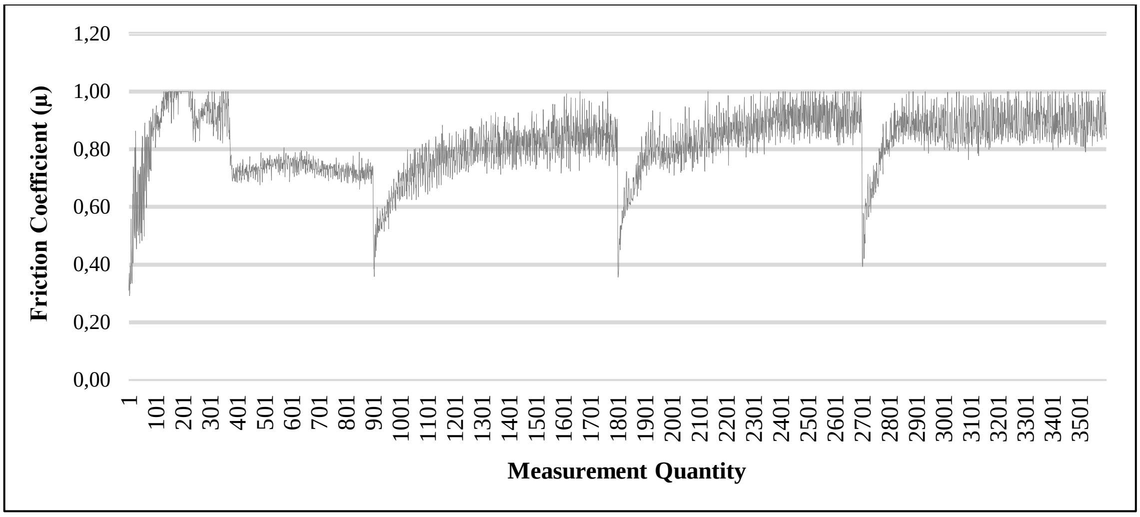


Figure 11:
Friction coefficient variation of unnitrided Calmax tool steel

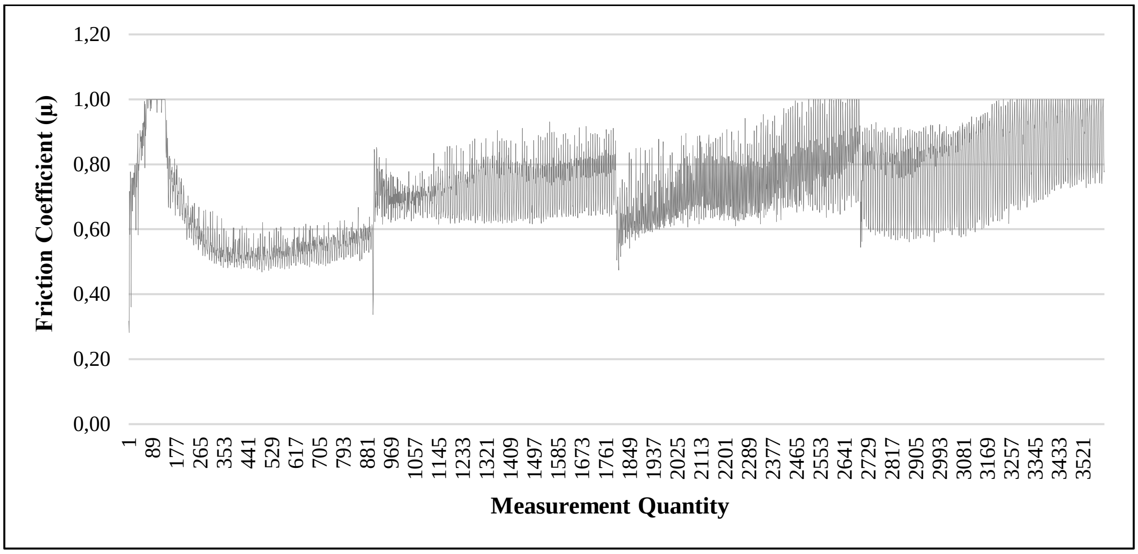
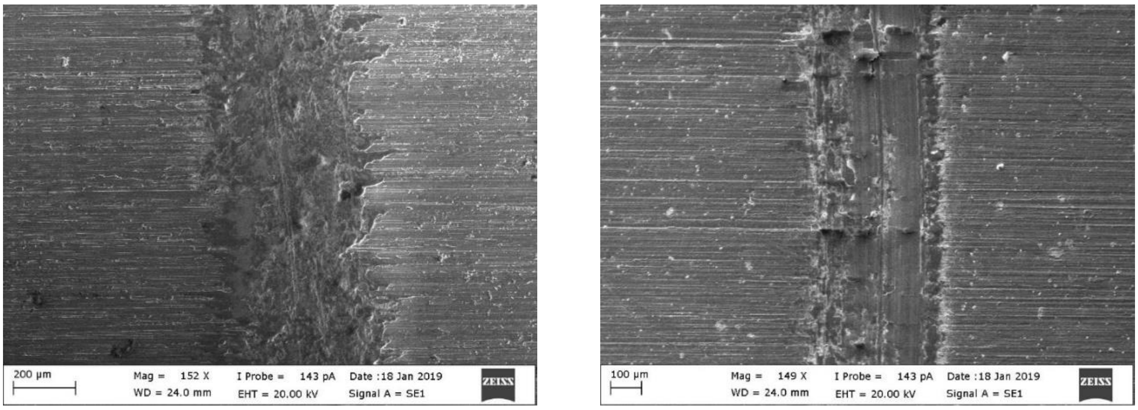


Figure 12:
Friction coefficient variation of plasma nitrided Calmax tool steel



a. ***Figure 13:*** *b.*
SEM images of ***a.*** *unnitrided Calmax tool steel,* ***b.*** *plasma nitrided Calmax tool steel*

D2 cold work tool steel had the best wear resistance within plasma nitrided cold work tool steels in this study. This is related to the fact that this steel contains medium level carbon and has nitride forming alloying elements such as Mo, V. On the other hand, plasma nitrided Calmax cold work tool steel exhibited relatively lower friction coefficient than the other plasma nitrided tool steels. When the graphs showing the variation of the friction coefficient are evaluated together, it can be concluded that the initial surface roughness of the plasma nitrided tool steels on the existing tribosystem has almost no effect on the friction coefficient. In the ball-on-disc tribosystem, the point stress that the ball touches on the sample reaches very high values, which renders the surface roughness ineffective. On the other hand, brittle nitride layer fractures increased the friction coefficient of the plasma nitrided tool steels.

4. CONCLUSIONS

The wear-friction characteristics of plasma nitrided cold work tool steels (D2, D6 and Calmax steels) were experimentally investigated in this study. From this investigation, the following major conclusions can be derived:

- Plasma nitriding process increased the surface roughness of the cold work tool steels. However, this effect on the surface roughness through the plasma nitriding process was found to be decreased as the surface roughness of the base metal increased.
- Fe_4N (γ'), Fe_3N (ε) and CrN phases were generally obtained on the surfaces of all plasma nitrided cold work tool steels.
- Higher surface hardness was obtained in plasma nitrided D2 and D6 cold work tool steels containing higher carbon content and alloying elements. However, Calmax cold work tool steel containing lower carbon content and alloying elements had a higher hardening depth than the other tool steels.
- In the ball on-disc tribosystem, when the plasma nitrided or unnitrided tool steel was used as a disc, no significant mass loss was observed in the tests to characterize the wear of the test samples.
- Plasma nitriding process improved the wear resistance and decreased the friction coefficient of the cold work tool steels. D2 cold work tool steel containing molybdenum and vanadium alloying elements showed the highest wear resistance after plasma nitriding process, whereas plasma nitrided Calmax cold work tool steel exhibited the lowest wear resistance. However, plasma nitrided Calmax tool steel had relatively lower friction coefficient than the other plasma nitrided cold work tool steels.

- The initial surface roughness values of the plasma nitrided tool steels had almost no effect on the friction coefficient in the tribosystem in this study. Brittle nitride layer fractures have determined the main effect on the friction coefficient.

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