The Effect of TiCN Coating on The Hardness of 630 Stainless Steel

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Abstract— In this study, TiCN coating was deposited on 630 stainless steel samples using the magnetron sputtering method at selected parameters. The distance between the target and the coating was kept constant during the operation. Heat treatment was applied to increase the hardness of the material before coating. The hardness values of the hardened and hardened + coated samples were compared. The coating hardness values, elasticity modulus and indenter depths were determined in the measurements made using the nanohardness device. It has been found that the TiCN coating improves the hardness of the 630 stainless steel material.

Keywords— Magnetron sputtering method, TiCN, 630 steel, hardness

I. Introduction

630 stainless steels have average corrosion resistance and good mechanical properties [1]. In this way, they are used in many areas of industry. However, the existing features should be improved according to the usage areas. For example; it should be improved surface hardness for springs, ball-bearing chains, valves, gears, and corrosion resistance for areas of use such as surgical and dental instruments, pressure vessels [2,3]. Hard coating applications are preferred to improve the surface properties of steel. Hard coatings are usually based on carbides, nitrides and carbonitrides of transition metal elements such as Ti, Zr, W, etc. [4].

In this study, TiCN coating was made on AISI 630 stainless steel by using the magnetron sputtering method which one of the PVD methods. Heat treatment was applied to the substrate material(AISI 630) before coating to support the hardness of the coating. Surface hardness values measured before and after coating were compared.

The nanohardness values of the TiCN coating obtained depending on the coating process parameters were determined using different loads. Thus, the mechanical properties of the coating, that are important for the performance of the coating, have been revealed. At the same time, the elasticity modules and the indenter depths under load have been determined.

II. Material Method

The chemical composition of AISI 630 stainless steel, which is used as the base material in the coating process, is shown in Table I.

Precipitation hardened AISI 630 (1.4542) is a good stainless steel quality with very high mechanical strength and good corrosion resistance. The representation and dimensions of this quality steel according to different norms are given in Table II. AISI 630 has a wide range of important uses: Such as jet engine parts, turbine blades, oil plant valve parts, molds, dental equipment, ship fasteners, surgical splint etc.

A. Sample Preparation

The samples were prepared by cutting from AISI 630 rolled cylindrical materials at a height of 9 mm using a Metkon brand Metacut sample cutting device. After the samples were cut, they were subjected to heat treatment, grinding and polishing stages. In order to increase the hardness of the samples prepared from AISI 630 material, precipitation hardening heat treatment was performed and their flat surfaces were ground. Grade 630 martensitic precipitation hardening stainless steel has a combination of high hardness and strength after suitable heat treatment.

TABLE I
CHEMICAL COMPOSITION OF STAINLESS STEEL USED IN EXPERIMENTAL STUDY

Elem. (%)	C	Si	Mn	Ni	Cr	Mo	Cu	Nb	S	P	Fe
AISI 630	<0.07	<0.7	<1.5	3-5	15-17	<0.6	3-5	0.15-0.35	0.03	0.04	Rem.

TABLE II
PROPERTIES OF SAMPLES USED IN EXPERIMENTAL STUDY

Item No	Material standard				Sample size	
	AISI	EN	DIN	UNS	Diameter (mm)	h (mm)
1	630	1.4542	X5CrNi-CuNb16-4	S17400	21	9

The precipitation hardening of the samples prepared from AISI 630 material was carried out under the specified processing conditions First of all, the solution treatment was applied at 1040 ^{0}C temperature and then it was subjected to aging heat treatment by keeping it at 500 °C for two hours. It was determined that the hardness value of the samples subjected to heat treatment was 462 HV. The hardness value of AISI 630 stainless steel before heat treatment was measured as 343 HV. The hardness of the samples before and after hardening is given in Table III.

TABLE IIII
THE HARDNESS OF SAMPLES BEFORE AND AFTER HARDENING

Item NO	Material	Hardness Before HB	Hardness after hardening HV
1	630	343	462

Before starting the coating process, the surface cleanliness of the material to be coated is important. The most important factor in the successful function of metallic coatings is the preparation level of the surface of the metal to be coated. The success of a coating process depends on cleaning the metal before coatingBecause the adhesion ability of the coating material is depended on cleanless. After heat treatment, the oxide layers on the surface must be cleaned.

For this purpose the heat treated specimens were first sanded with different abrasives from 360 to 2000 before coating, and then polished using an Al_2O_3 solution. Fig. 1 shows the photos of the samples.

Fig. 1a) After heat treatment of AISI 630 Stainless Steel Samples b) Surface photograph after Sanding and Polishing Process

The cleaning provides a good bond between the coating metal and the surface and increases the protection efficiency of the coating. The cleaning of the surfaces to be coated is effective on the coating quality.

If effective surface cleaning cannot be achieved, spallation occurs after the coating process.

Therefore before the coating process, the samples to be coated were cleaned by immersing in aseton and ethyl alcohol for 10 minutes in an ultrasonic bath respectively, and then dried (Fig. 2).

Fig. 2 Cleaning the Samples to be Coated in an Ultrasonic Bath Before Starting the Surface Coating Process

B. Coating Method

TiCN coating was applied on the prepared samples by using the PVD - Magnetron sputtering system.

The coating process was carried out in the following steps;

The samples were placed in the vacuum chamber and a high vacuum level was achieved by means of vacuum pumps (Fig. 3). After reaching the desired high vacuum value, substrate material surfaces were subjected to ion cleaning process in the vacuum environment. Then Ti interlayer and TiCN coating were applied to the samples in the coating process, respectively .

In order to reduce residual stresses at the interface of the coating and underlay and to provide better adhesion on the base material, a titanium interlayer is coated [5]. Ti coating was applied at a target power of 4000 W and 15 sccm argon flows for 30 seconds.

The TiCN coating process was carried out using a target power of 5000 W,-80 V bias voltage in argon + acetylene atmosphere in the coating process. TiCN coatings were applied for 3 hours during the coating experiment. When the coating period is over, the current and voltage are cut off and the samples are allowed to cool [6, 7].

Fig. 3 The Samples placed in the surface coating vacuum chamber and the size of the Samples

The average coating thickness for TiCN coating was found to be 2 µm by Calotest method.

C. Hardness Values

The hardness and elasticity modulus values were measured from the samples surfaces coated in the experimental environment with the CSEM nanohardness tester shown in Fig. 4.

Fig. 4 CSEM nanohardness tester

Instrument which provides the ability to measure the indenter depth under the applied force, throughout the testing cycle. It is capable of measuring both the plastic and elastic deformation of the material under test.

Applied force and depth are measured dynamically during a load-unload cycle. Hardness and Elastic Modulus are calculated directly from the resultant force-displacement curve.

In Fig. 5 shows the load-depth graph of sample obtained from nanohardness tests of TiCN coatings.

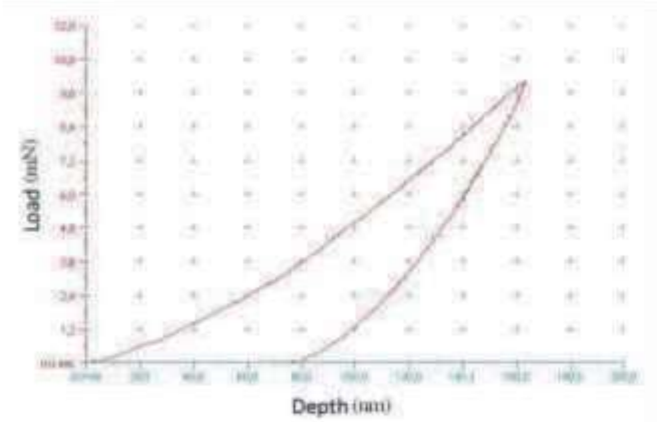

Fig. 5 Load-depth graphs obtained from nanohardness tests of TiCN coatings: (Maximum load 10 mN, loading rate 20 mN / min, unloading rate 20 mN / min).

In Fig. 6, the average hardness values of the samples measured before heat treatment, after heat treatment, and after TiCN coating are compared.

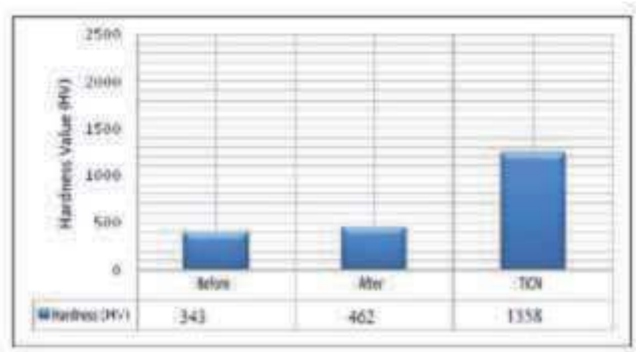

Fig. 6 The average hardness values of the samples measured before-after heat treatment and after TiCN coating.

After the TiCN coating process was applied to the samples, the hardness and elasticity modulus values were measured from the sample surfaces under different application loads and the obtained measurement results are given in Table IV. The CSEM nanohardness tester is a computer-controlled device that allows the hardness and mechanical properties of thin coatings to be measured independently of the substrate material with very low indenter depths (Fig. 4). The device, which has a maximum loading capacity of 300 mN, automatically calculates the hardness value and elasticity modulus according to the contact area of the Vickers tip used by drawing the time-dependent graph of the applied load and the indenter depth on nanometer scale.

TABLE IIIV.

HARDNESS, ELASTICITY MODULE AND TIP INSERTION DEPTH VALUES OF TICN COATED OF AISI 630 STAINLESS STEEL SAMPLES UNDER DIFFERENT APPLICATION LOADS

	Hardness (HV)	Elasticity Module (GPa)	Load applied (mN)	Indenter Depth (nm)
$TiCN^1$	1750.3	156.6	10	162.24
$TiCN^2$	1362.7	150.4	12	211.59
$TiCN^3$	1233.1	146.1	15	247.31
$TiCN^4$	1146.3	146.2	18	281.12
$TiCN^5$	1302.1	157.9	20	280.86

In Fig. 7, the hardness and elastic modulus values of the TiCN coated AISI 630 stainless steel samples under different application loads are shown graphically and the results are compared. Besides, its indenter depth results are also given in Fig. 8.

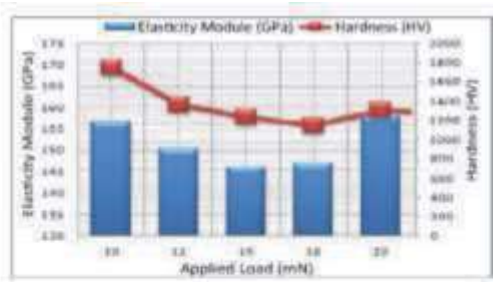

Fig. 7 Hardness and elasticity modulus values of the samples against different application loads.

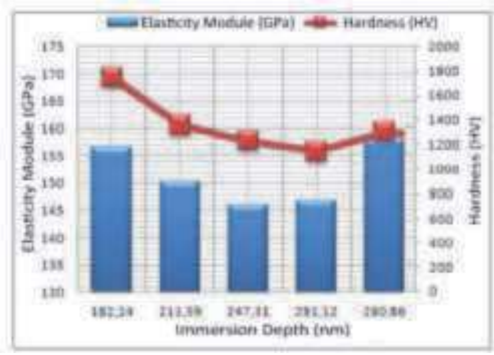

Fig. 8 Hardness and elasticity modulus values of the samples against differen t indenter depths.

III. CONCLUSIONS

Hardness is an important mechanical property for coatings. In order for the coating to fulfill its expected function, the hardness of the coating must also be supported by the base material feature. For this purpose, the samples made of AISI 630 alloy were heat treated and their hardness values were increased. After heat treatment, the sample hardness was obtained at 462 HV.

Afterward, TiCN coating was applied with magnetron sputtering technique and the coating hardness was measured by nanohardness measurement method under different loads and the average hardness value of 1358 HV was determined. It was determined that the coating hardness significantly increased the AISI 630 material hardness.

The hardness, modulus of elasticity and indenter depths of TiCN coated AISI 630 stainless steel samples were investigated under different application loads. The loads used in hardness measurement did not affect the distribution of hardness values, since they are in the area of indenter depth not exceeding one-tenth of the coating thickness. It is seen that as the hardness increases, the indenter depth decreases. The modulus of elasticity is determined for the coating, this value will be of great benefit in future tests for determining wear resistance.

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