International Journal of Engineering Science Invention (IJESI)
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 7 Issue 12 Ver. III || Dec 2018 || PP 07-20

Investigation of 2550 Cold Work Tool Steel Coated with TiN and CrN by PVD Method by Scratch Method

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Abstract: *Rapidly developing PVD (Physical Vapor Deposition) coating process is applied in many industrial applications. Coating surfaces are important in many cases. Coating may increase functionality of bottom layer. In many cases, depending on adhesion of material and coating, base material makes important contribution to mechanical properties of materials. In PVD process, coating process is performed at low temperature and a homogeneous structure is obtained. Bonding forces of base material and coating material play an important role in maintenance of coating without deformation. Scratch test is performed to evaluate hardness and adhesion properties of thin films. DIN 1.2550 cold work tool steels, which are used in cutting, pattern, mint molds and scissor blades, and are subjected to wear, are generally used by coating with PVD method in usage areas. In this study, it was aimed to determine the effect of coating on hardness and adhesion ability of DIN 1.2550 material with coated TiN and CrN by PVD technique. Also, sections of test specimens were examined by scanning electron microscope (SEM) and thickness of coating was measured in the study. Surface roughness and hardness measurements were performed after coating process. A nanoindenter and a scratch tester were used to evaluate hardness and adhesion properties of thin films. For this purpose, samples were covered with TiN and CrN thin film by using cathodic arc PVD technique, and results were evaluated by performing hardness and scratch tests.*

Key words: *Scratch, PVD, Coating, Cold work Steel.*

Date of Submission: 10-12-2018 Date of acceptance: 25-12-2018

I. Introduction

One of the biggest obstacles to the production of fast and high-quality materials seen in all areas of manufacturing industry is wear problems that are no doubt encountered. Wear is an undesirable situation in production tools, which are encountered in various stages of production, both in machining and in other manufacturing methods. When production in company is the fastest, removing, replacing and adjusting of mold in case of wear on work piece, on the other hand, in the preparation of the existing mold for re-production by regrinding, polishing or similar processes; it is possible to deal with problems such as loss of time, loss of time and failure to grow crops on time.

Wear is defined as loss of material due to contact between at least two surfaces. If wear exceeds an acceptable limit, wearing part may not perform expected performance from itself. This will cause the material to malfunction [1].

There has been an increase in the number of studies to minimize problems caused by wear on machine parts, in recent years. Therefore, requirement for control of wear has become compulsory for reliable technology in the future [2,3].

Dynamic loads and forces occurring in both cutting tools and work pieces and extrusion pieces can be caused by very different wear behavior on materials. Therefore, different behavior and characteristics are observed in boundary, surface and inner parts of the materials. It is sufficient for surface part to be martensitic in speed steels and cold and hot work steels for many purposes. However, in cases where corrosive and abrasive effects are expected, it is also necessary to reach a surface structure in order to reduce friction coefficients in high temperature environments where parts are exposed to extreme heat effects [4]).

It can be observed that the systems that are exposed to friction and thus wear out and loss productivity, are coated using PVD, CVD, DVD, Plasma or Thermal Spray Methods to improve their working condition in the industrial sector. PVD technique is widely used because of its easy application. It does not cause any damage to microstructure of bottom layer in material and does not require a change in dimensional tolerance. Types of PVD coatings preferred and applied according to the conditions of use are TiN, AlTiN, TiAlN, CrN, TiAlCrN, TiCN and etc.

Besides fine ceramic film coatings with PVD technique are the most preferred coatings because of their adherence to the bottom layer to which they are applied due to incorporation of carbides and nitrides and because of superior properties of wear and abrasion as well as other many advantages [5], nitride based hard ceramic coatings, which are applied by using PVD method, having two or more components, high hardness, better adhesion and tribological properties, are also produced as an alternative coating type and offered to the service of industry. The purpose of the production of these alternative coatings having different compositions and structures is to eliminate weaknesses of previously produced coatings and to have new coatings that exhibit better properties [6].

One of the most important functional requirements in the manufacturing process is the adhesive strength of the coating with the substrate because the performance and life of the coating are limited by the adhesion strength [7]. One of methods used to analyze adhesiveness of coating to the material surface is scratch test. This test consists of production and evaluation of controlled damage on a hard ceramic coating with a single pointing motion (Figure 1). In these tests, the Rockwell C (Rc) diamond pen is used as an indicator. ASTM 1624-05 standard was used in evaluation of these tests [8].

The purpose of this study is to improve mechanical properties of materials coated with TiN and CrN thin film by using cathodic arc PVD technique. Sections of test specimens were examined by scanning electron microscopy (SEM) and thicknesses of coating were measured. Surface roughness and hardness measurements were performed after coating process. A nanoindenter and a scratch tester were used to evaluate hardness and adhesion properties of thin films.

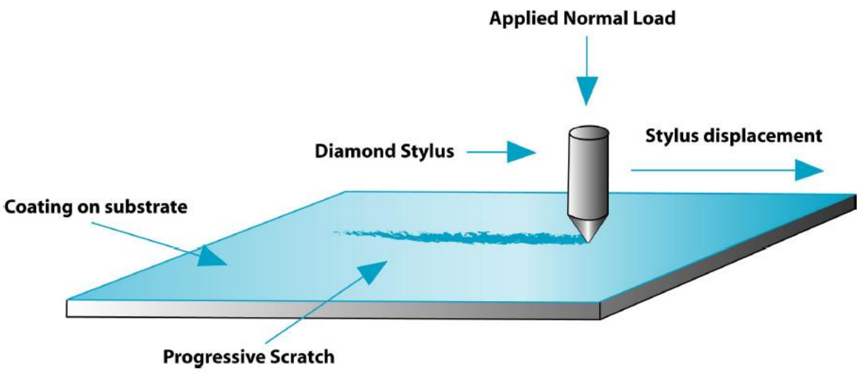

Figure 1. Schematic Test Method [7].

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II. Material and Method

Material Used and Heat Treatment

Chemical composition of the cold work tool steel with high hardening depth in oil, medium carbon containing tungsten element, impact and collision resistant, excellent toughness with high hardness, wear resistance and no breakage of the cutting edges are given in Table 1 [9]. Heat Treatment conditions and hardness values of 2550 Cold Work Tool Steel are shown in Table 2. Tempering temperatures are selected in the conditions shown in Figure 2.

Table 1. Chemical composition of 2550 Cold Work Tool Steel used in the experiment

	C	Si	Mn	P	S	Cr	V	W
Minimum %	0,55	0,70	0,15	-	-	0,90	0,10	1,70
Maximum %	0,65	1,00	0,45	0,030	0,030	1,20	0,20	2,20

Table 2. Applied Heat Treatment belong to Cold Work Tool Steel 2550

Annealing for Softening	**710 – 750 °C**			
Hardness After Annealing	Maximum 225 HB			
Annealing for Stress Relieving	Approximately 650 °C			
Hot Forming	1050 – 850 °C			
Hardening	870 – 910 °C			
Hardening Medium	Oil			
Hardness after Hardening	58 – 62 HRC			
Hardness after Tempering	100 °C	200 °C	300 °C	400 °C
	60 HRC	59 HRC	56 HRC	53 HRC

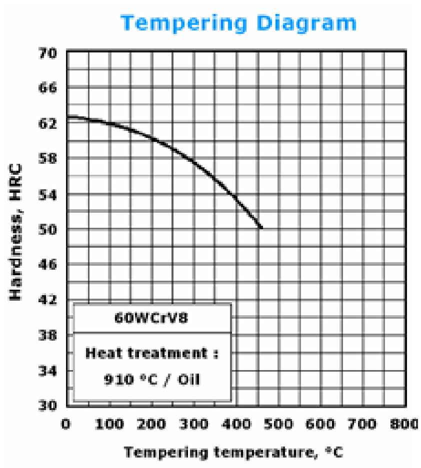


Figure 2. 2550 Tempering Diagram of Cold Work Toll Steel [9].

Coating Process and Coating Parameters

Specimens were first polished and then ultrasonic chemical cleaning with alkaline (basic) detergents were performed. Finally, prior to coating, rinsed with pure water and dried with hot air. Samples placed in the coating cabinet were vacuumed to $4x10^{-5}$ and TiN and CrN coatings were applied by cathodic arc PVD method with the parameters given in table 3. Ionbond TINKAP Surface Technology Industry and Trade Inc. Cooling after coating was performed in a vacuum environment. At the end of the procedure, specimens were removed from cabinet and liquid droplets were cleaned with an AlO2 brush and lubricated with protective oil so that coating process was finished.

Table 3. Parameters of TiN and CrN Coatings.

Material	Coating	Basic Voltage (V)	Cathode Current (A)	Number of Cathode	Nitrogen Pressure (Torr)	Time (min.)	Temperature(°C)	Thickness(μm
1.2550	**TiN**	**-200**	**50**	**3**	**$4x10^{-3}$**	**60**	200-220	3
	CrN	-110	55	3	$6,5x10^{-3}$	70	200-220	3

Measurements of Coating Thickness and Hardness

Hardness measurements were performed from five different points on the specimen by applying 100 grams of load from surface of TiN coated and CrN coated specimens, and it was determined by taking averages of these. Coating parameters were chosen according to base material in order to prevent loss of hardness in the base material.

Scratch Test and Applied Process Parameters

Scratch testing devices are used to determine the mechanical characteristics, such as breakage, damage and adhesion of surfaces of thin films and coatings. This test method can be applied to various hard ceramic coating compositions (carbides, nitrites, oxides, diamond and diamond-like carbon), physical vapor deposition, chemical vapor deposition and direct oxidation to metal and ceramic surfaces [8]. The scratch tests were made on a scratch testing device of brand name CSM. Practical scratch adhesion value of coating is defined as the lowest critical load at which a coating fails. It is an important parameter related to coating-substrate adhesion that could be used for comparative evaluation of coatings. The working principle of the device is based on making a controlled scratch on the material with a stylus. The surface of the coating is scratched with a stylus material (generally diamond or hard metal (Wolfram Carbide)) under a fixed or increasing load. Breakage of the coating starts to happen at the critical load value. The critical load value and the scratch (friction) force can precisely be detected with a sound sensor (MST & RST) mounted on the loading arm. At the same time the breakage can be observed by an optical microscope. In the experiments, the calculation of the critical load (Lc_1) at which the first crack occurred and the critical load (Lc_2) at which the coating was broken were made using Eq.1 according to ASTM C1624-05 (10). Besides, the loading – acoustical emission charts and the parameters given in Table 4 were used in calculations and evaluations.

$$L_{CN} = \left\lfloor L_{rate}\left(l_N / X_{rate}\right)\right\rfloor + L_{start} \text{ (N)} \qquad \text{(Eq. 1)}$$

In the equation;

L_{CN} : Critical scratch load (N=load sequence) (N),

L_{rate} : Loading rate (N/min),

l_N : The distance between the start of scratch and the start of damage (mm),

X_{rate}: Scanning rate (mm/min),

L_{start}: Pre-load value (N).

Table 4. Parameters used in scratch tests.

Parameters	Values Applied
Scratch Type and Form	Linear and progressive
Loading Value at the Start	0,5 N
Loading Value at the End	25 N
Loading Rate	24,5 N/min
Acoustic Sensitivity	8
Scanning Load	0,05 N
Scanning Rate	4 mm/min
Scanning Distance	4 mm
Type of Stylus	Spherical Diamond (Stylus Tip Radius 100 µm)

III. Results and Discussions

Coating Thickness and Hardness

CrN coated samples have an average hardness of 1548 Hv and it is seen that it is the highest hardness sample compared to other samples (Figure 3). At hardness of specimens, applied bias voltage, coating thickness and coating temperature are thought to be effective.

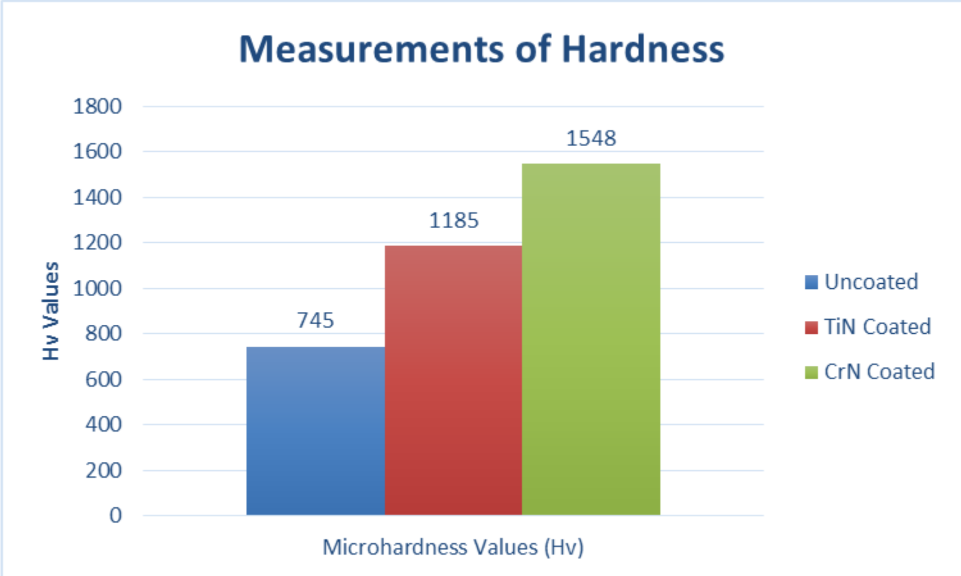


Figure 3. Microhardness values of specimens

In hardness measurement experiments, it was determined that hardness of ZrN coatings was dependent on the bias voltage value and the bias voltage mode applied (10). Surface SEM images, coating thickness measurement SEM images and EDS analyzes of uncoated, CrN and TiN coated samples are shown in Figure 4-13.

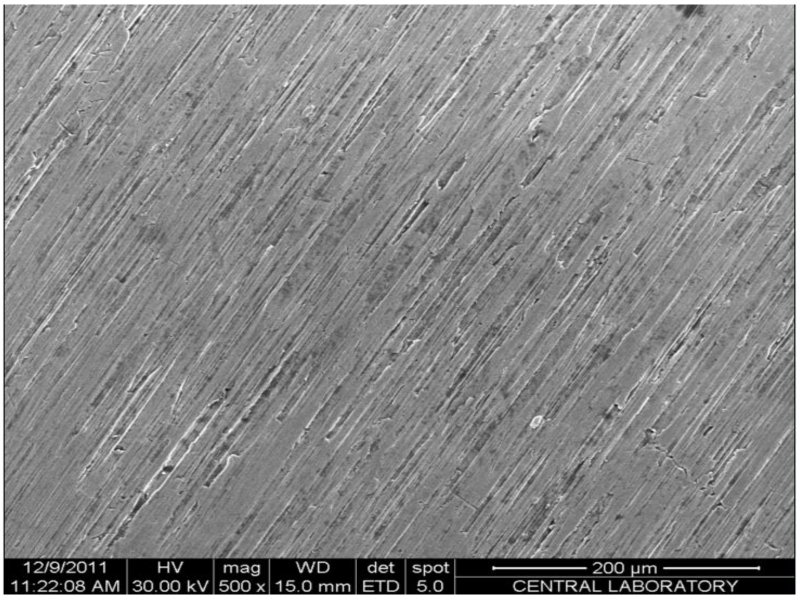
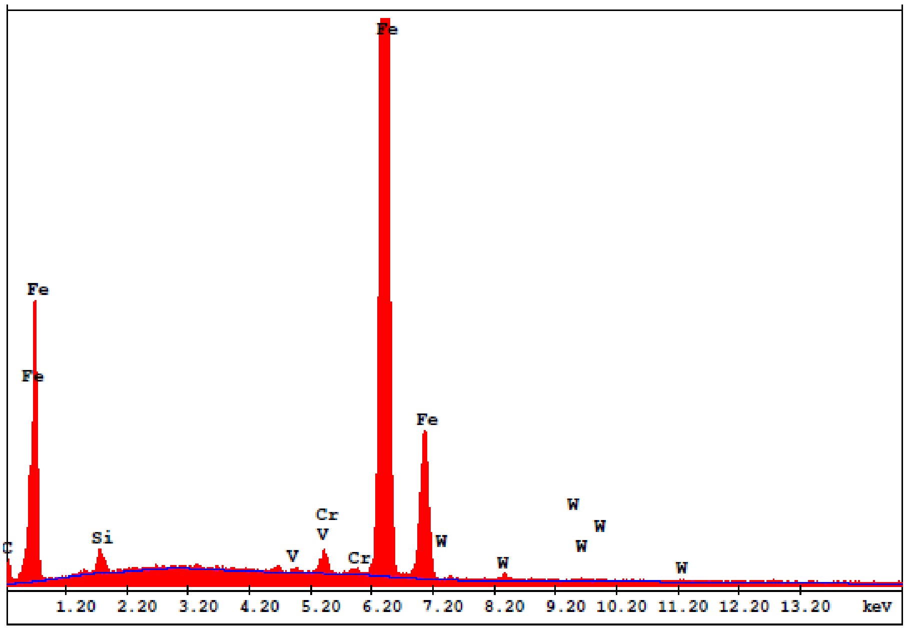


Figure 4. SEM image taken from lateral surface of non-coated specimen



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
C K	10.63	35.55	0.0216	1.1400	0.1781	1.0006
SiK	1.51	2.16	0.0049	1.0831	0.3018	1.0022
V K	0.16	0.12	0.0018	0.9568	0.9605	1.2162
CrK	0.91	0.71	0.0115	0.9757	0.9792	1.3235
FeK	84.85	61.04	0.8308	0.9800	0.9973	1.0018
W L	1.95	0.43	0.0147	0.8131	0.9282	1.0000
Total	100.00	100.00				

Figure 5. General EDS Analysis taken from lateral surface of non-coated specimen

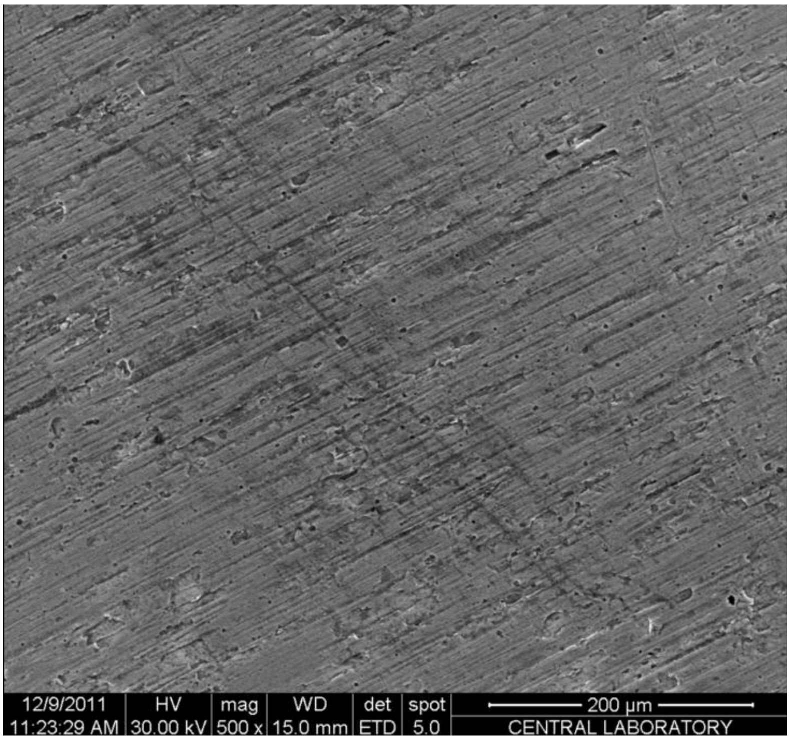
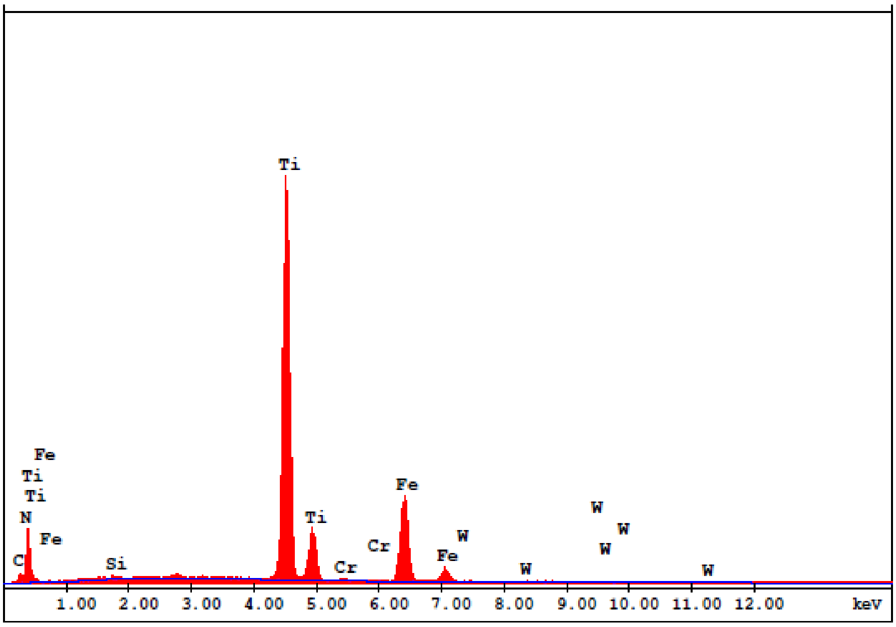


Figure 6. SEM image taken from lateral surface of TiN Coated specimen



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
C K	8.13	17.81	0.0255	1.1036	0.2837	1.0009
N K	25.14	47.22	0.0782	1.0954	0.2838	1.0011
SiK	0.29	0.27	0.0013	1.0493	0.4421	1.0049
TiK	46.14	25.34	0.4460	0.9403	1.0046	1.0231
CrK	0.25	0.13	0.0020	0.9415	0.8174	1.0343
FeK	19.40	9.14	0.1632	0.9455	0.8891	1.0007
W L	0.65	0.09	0.0051	0.7819	1.0055	1.0000
Total	100.00	100.00				

Figure 7. General EDS Analysis taken from lateral surface of TiN coated specimen

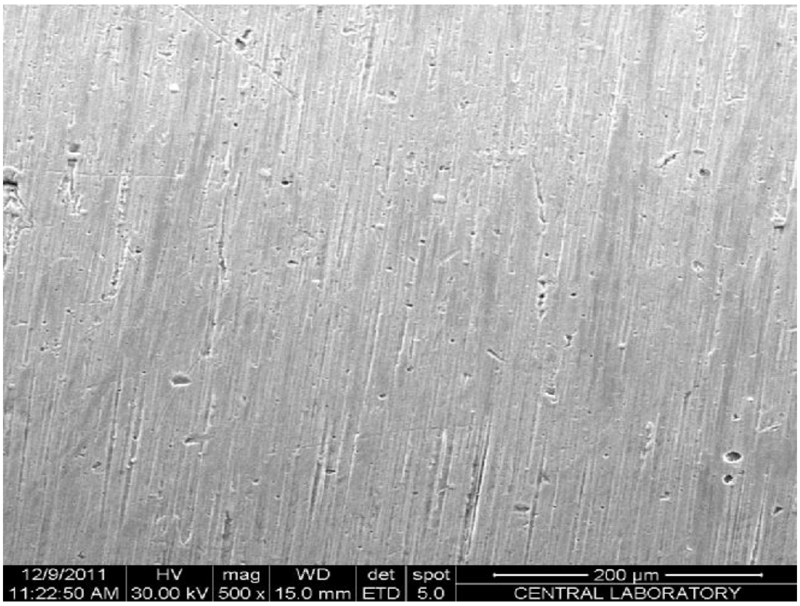


Figure 8. SEM image taken from lateral surface of CrN Coated specimen

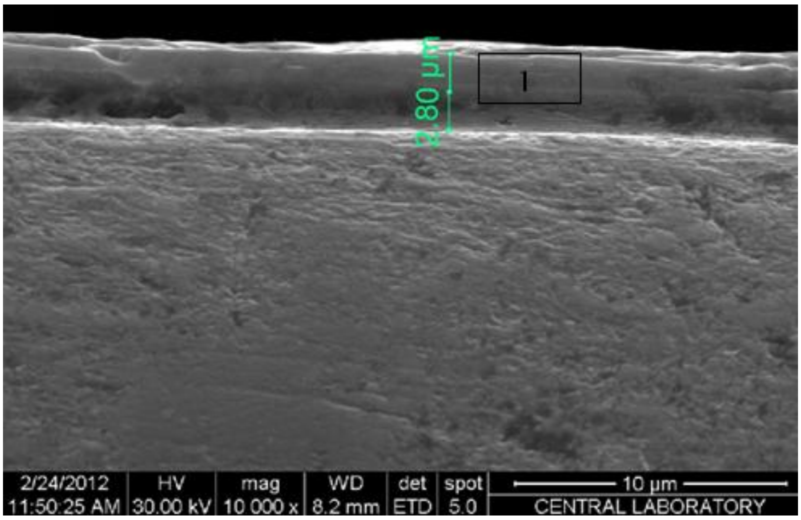
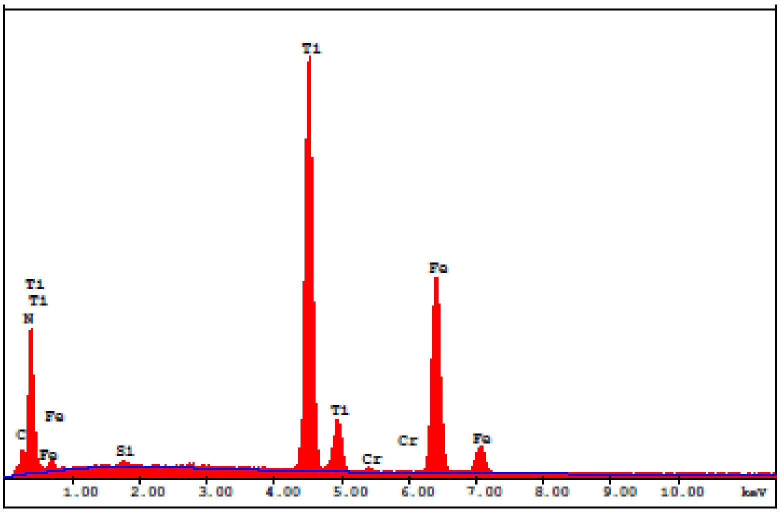


Figure 9. SEM image taken from section of TiN Coated specimen



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
C K	11.35	20.16	0.0373	1.0746	0.3059	1.0010
N K	38.69	58.95	0.1103	1.0666	0.2672	1.0006
SiK	0.38	0.29	0.0018	1.0220	0.4560	1.0033
TiK	26.03	11.60	0.2512	0.9136	1.0186	1.0371
CrK	0.33	0.14	0.0029	0.9154	0.9014	1.0576
FeK	23.22	8.87	0.2024	0.9192	0.9482	1.0000
Total	100.00	100.00				

Figure 10. EDS Analysis taken from coating surface in section of TiN coated specimen

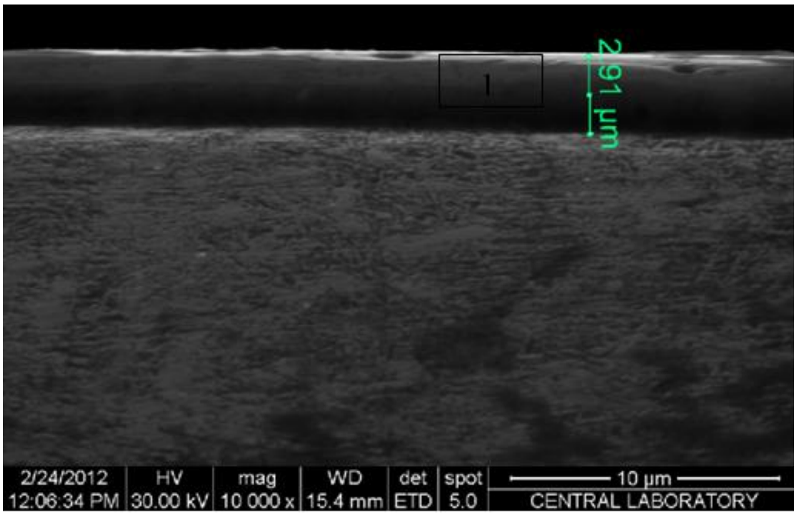
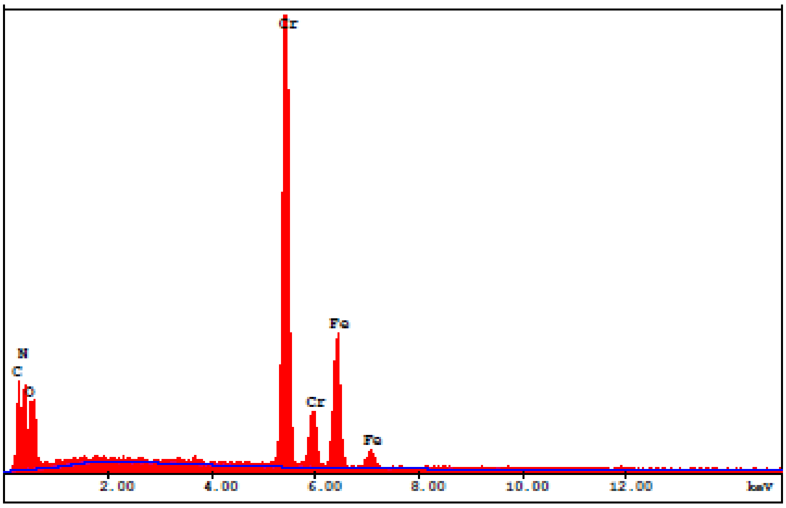


Figure 11. SEM image taken from section of CrN Coated specimen

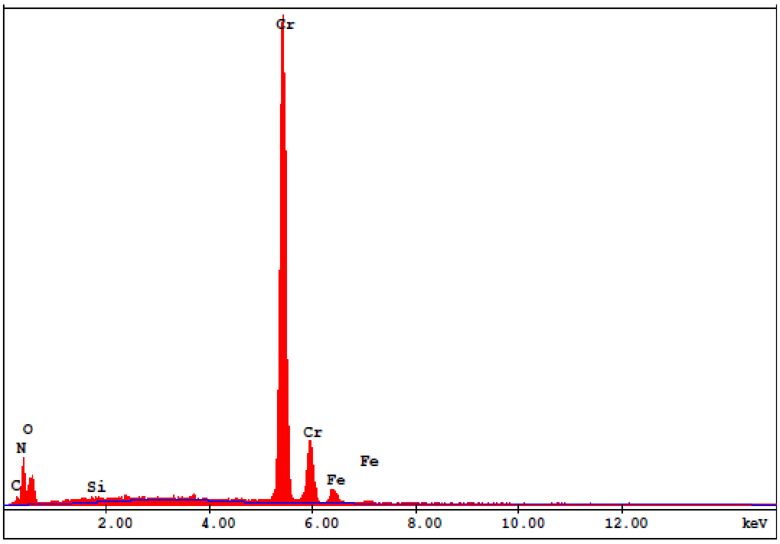


EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
C K	22.60	35.14	0.0794	1.0553	0.3327	1.0007
N K	27.57	36.76	0.0570	1.0474	0.1973	1.0007
O K	12.97	15.14	0.0204	1.0404	0.1509	1.0010
CrK	25.81	9.27	0.2489	0.8980	1.0366	1.0359
FeK	11.06	3.70	0.0938	0.9018	0.9406	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
C K	94.05	1.30	2.11	72.31
N K	96.36	1.87	2.09	51.54
O K	79.78	2.40	2.32	33.27
CrK	1079.58	12.52	0.62	86.24
FeK	328.23	10.36	1.15	31.67

Figure 12. EDS Analysis taken from coating surface in section of CrN coated specimen



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
C K	6.12	13.67	0.0174	1.1018	0.2575	1.0009
N K	20.14	38.54	0.0640	1.0935	0.2902	1.0014
O K	8.36	14.00	0.0191	1.0861	0.2097	1.0028
SiK	0.44	0.42	0.0019	1.0475	0.4077	1.0035
CrK	62.01	31.97	0.5977	0.9401	1.0200	1.0053
FeK	2.94	1.41	0.0226	0.9441	0.8139	1.0000
Total	100.00	100.00				

Figure 13. General EDS Analysis taken from lateral surface of CrN coated specimen

Scratch Tests

This test is designed to evaluate mechanical integrity, deterioration modes and practical adhesion strength of a particular hard ceramic coating on a given metal or ceramic substrate. The test method does not measure basic 'bond strength' of bond between coating and substrate [8].

In the experiments, the critical load (Lc_1) at which the first crack occurred, and the critical load (Lc_2) at which the coating broke were calculated. The scratch images obtained with optical microscope, were evaluated together with acoustical emission graphics. The numerical data obtained from the experiments are given in Table 5.

Table 5. Coating Thickness, Hardness and Scratch Test Measurement Data.

Material	**Surface**	**Coating Thickness Mean (µm)**	**HVIT Mean Vickers Hardness (HV-10mN)**	**Scratch Testing**			
				Load (N)	**Critical Load (Lc_1)**	**Critical Load (Lc_2)**	**Failure Type**
1.2550	Uncoated	-	745	-	-	-	-
	TiN	2,8	1185	25	2,95	**5,7**	Chipping
	CrN	2,91	1548	25	3,75	**7,90**	recovery spallation

Graphs of friction, friction coefficient, acoustic emission and penetration depth obtained from scratch test applied to coated samples are given in Figure 14-15.

Indentation and Scratch test methods are widely used in adhesion studies of thin / thick films. All these methods describe manner in which coating film from base material is removed in a sense and resistance shown against it. Scratch test method is a widely used mechanical test method [7]. Critical loads (small (Lc_1), big

(Lc_2)) calculated via using Eq. 6.4 by the help of normal and friction force changes, acoustic emission graph and microscopic investigations (determination of breakpoints) were given in Table 5.

When structure and shape of scratches obtained in experiments according to the scratch catalog given in ASTM C1624-05 is investigated, it is seen that scratch surfaces of CrN coating are smoother and scratch is continuous and torsion type recovery spallation is formed in coating. In the TiN coating type, it is seen that there are regional discontinuities on scratched surfaces, and rupture in chipping style edge regions with breakage of the coating. Critical breaking load of CrN coating is higher than the TiN coatings. Because of type of refraction and symmetrical fracture structure, it can be concluded that coating is homogeneous. Break formed as spallation and chipping indicates that base material is not very well diffused. TiN coating was not immediately damaged, but it is at lower levels in small critical loads (Lc_1) where the first damage occurred. Microhardness of the CrN coating is higher than the TiN coating. Same situation is similar to critical load (Lc_2). In addition, comments obtained from scratch type surveys and SEM images taken from section support each other.

Critical load (Lc_2) in CrN coatings is approximately 38.5% higher than the TiN coating. The results are consistent with the hardness values obtained after coating [8].

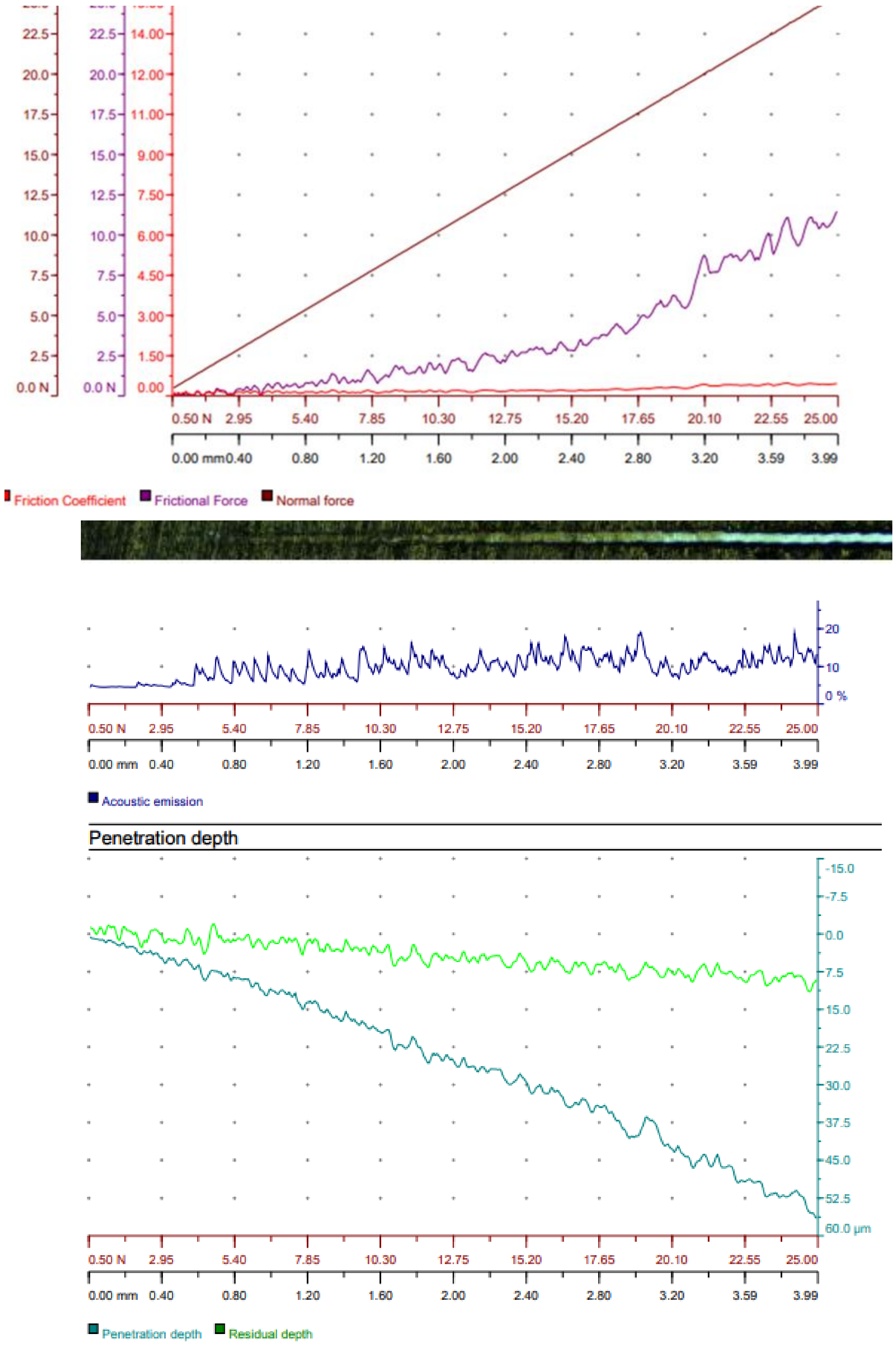


Figure 14. Scratch Test Results applied to TiN Coated specimens (friction graph, microscopic image of scratch, and acoustic emission graph)

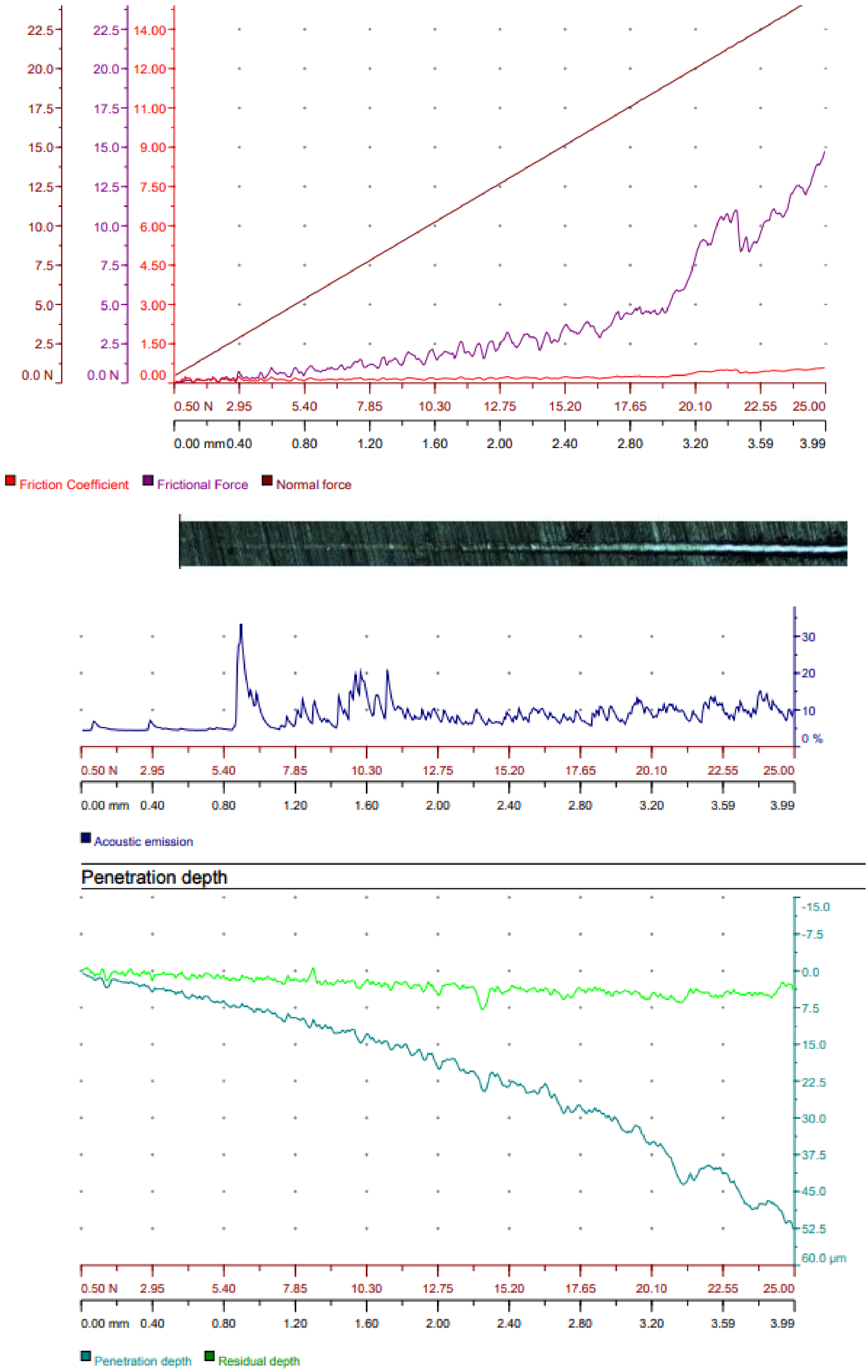


Figure 15. Scratch Test Results applied to CrN Coated specimens (friction graph, microscopic image of scratch, and acoustic emission graph)

IV. Results

1. Hardness values of coated samples increase 1,59 times compared to uncoated samples. The microhardness ratio of CrN coatings was 30% higher than TiN coatings.

2. The critical loads (Lc2) for CrN coatings are approximately 38,5 % higher compared to TiN coatings for the same base materials. The results are compatible with the hardness values obtained after the coatings. Critical load (Lc_2) shows an increase with increasing coating thickness and hardness.

3. It is seen that scratched surfaces of CrN coating are smoother and scratch is continuous, torsion-type (recovery spallation) clamping damage occurs in coating.

4. TiN coating type also appears to have rupture in the edge regions of chipping style with the breakage of the coating, where there are regional discontinuities on the scratch surfaces.

5. Coating conditions, coating material and coating thickness are effective criteria in determining adhesion properties of coatings to base material.

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Ayhan Aytaç" Impact evaluation of Some Parameters Affecting Surface Qualities by Taguchi Experimental Design Method: An Experimental Study on Heat Treated Low Carbon Wheel Rim" International Journal of Engineering Science Invention (IJESI), vol. 07, no. 12, 2018, pp 07-15