An Analysis of TiAlCrSiCN Nanoparticles coating via PVD on Cold Work Tool Steel

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Abstract

The present paper has applied energy-dispersive X-ray (EDX), X-ray diffraction (XRD), field emission scanning electron microscope (FESEM), and atomic electron microscope (AFM) to characterize coating in terms of chemical compound, structure, morphology of surface and cross section, respectively. Nanoidentation, microhardness and pin on disk test have also been used to analyze mechanical characteristics of coating. The findings reveal that using target with chemical compound containing Silicon leads to entering silicon into the structure of coating. Comparing with the results of XRD, it is revealed that adding Silicon to TiAlCrCN coating structure causes TiAlCrCN/a-SiCN nanocomposite structure. The size of crystals existing in coating is in nano scale (about 9/5 nm for TiAlCrSiCN). The data obtained from FESEM and AFM show that during layer growth, some particles with low adherence are formed in the above level atmosphere of coating and are separated from coating surface as preceding the process leading to forming some holes. Further, adding Silicon to coating structure decreases roughness as well as RMS of coating from 9/7 to 4/6. Friction coefficient of coating is decreased by adding Silicon and the degree of hardness is also increased from 27/5 to 38/5 GPa indicating the increase of mechanical characteristics and friction resistance of coating.

Keywords: Physical vapor deposition, reactive cathodic arch, TiAlCrSiCN, nanoparticles, cold work tool steel Spk 1/2080, nano hardness.

Introduction

Cold work tool steels are widely used in industry due to their accessibility, affordability and various applications[1]. In order to increase the capabilities and efficacies of such tools in other scopes, industry needs to study surface engineering and various techniques of sedimentation[2]. Currently, a wide range of studies are conducting on improving surface characteristics of industrial tools and pieces to increase their performance and lifetime through precipitating hard elements on the surface of these pieces[3]. Applying hard multi-components on the surface of such pieces is a main method to protect them against friction[4]. This method is widely used in industry such as cutting tools, medical prostheses, forming and casting tools[5]. Using surface coatings in cutting tools and mechanical components has been changed into a technique in manufacturing industry during the last two decades[6].Recently, some researchers also have studies applying five and six-component coatings on warm and cold work tool steels using various methods that have revealed useful and reasonable results such as the increase of frame`s and tool`s lifetime as well as the increase of cutting speed in cutting tools[7]. In the present study, to create a nano structured coating on cold work tool steel Spk 1/2080, physical vapor deposition with arch has been used in which sedimentation is created through the vapors resulted from electrode[8]. Due to appropriateness and adjustability of sediment`s rate from below nano meter to several micrometers, various applications have been appeared to produce cutting tools coatings, thermal barrier and electrical coatings acquiring the attention of manufactures and researchers to physical vapor deposition method using arch technique. The coatings created by this way are used in case of oxidation, friction and corrosion[9].

Chemical vapor deposition (CVD) is a coating method. Chemical vapor deposition is a chemical process which is used for the thin films deposits of various elements[10]. In a CVD process, a layer of substance is subjected to one or more evaporated substance during which ingredients interact with the mentioned layer and/or are analyzed and create the expected sedimentary product[11]. However, secondary products also are produced which are exited through gas. Chemical vapor deposition are widely applied to produce semi-conductors (as a part of the process of producing semi-conductor nano structures) as well as for various films deposition such as polycrystalline Silicones, Silicon, Amorphous, Germanium, Tungsten, Silicon oxy-nitride, Titanium nitride[12]. CVD process is also used to produce synthesis diamond. Coatings created by this way have a homogenous thickness. CVD method is widely used in industry due to its capability of creating various types of coatings with different compounds and components[13].

As mentioned, there are various methods to produce nano structured coatings which have unique characteristics with respect to the existing needs and features of the produced

coating. Since the coatings produced through physical vapor deposition have many applications in producing nano structured coatings with specific features, coating method is discussed in the following section[14].

Types of PVD methods: Accumulation of physical vapor deposition through physical evaporation method: In accumulation through physical evaporation, coatings are produced on the solid surface through elements` and components` condensation of gas phase. Evaporation in vacuum is the most general method of providing highly pure coatings and under controlled conditions[15]. The principles of this method are based on fully physical effects but accumulation with physical evaporation may be accompanied with chemical interactions in some cases. Accumulation through physical evaporation is classified in different methods such as thermal evaporation, cathodic arch and sputtering methods.

Accumulation through thermal evaporation: Obviously, atoms and molecules are released from the surface of a substance when it is heated (in solid or liquid phase)[16]. In order that a molecule can leave the surface of a substance, it is needed that vertical component of speed resulted from kinetic energy due to molecules` thermal movement be greater than the attractive force between molecules[17]. Therefore, increasing the temperature leads to the increase of the number of particles satisfying this inequality. After evaporation, the object is reduced for internal energy[18]. To prevent the object reduction, it should be heated. Additionally, during evaporation, as the result of volume expansion occurred in evaporation, some extra works should be done. The number of atoms or molecules evaporated from the surface of liquid or solid depends on the heat intensity[19]. Generally, equilibrium vapor is resulted in a closed thermodynamic system[20]. However, no thermodynamic equilibration is created in evaporation since the surrounding environment of evaporation source acts as a capturing well for vapor. Atoms of evaporation are condensed on all parts with lower temperature than evaporation source[21].

Systematicinvestigation of evaporation rate in vacuumreveals that liquid has a special capability for evaporation which cannot be greater than maximum evaporation rate in a given temperature, even if temperature source is unlimited[22]. Theoretical maximum evaporation rate is obtained only when the number of vapor molecules leaving the surface is in accordance with the number existed to create pressure equalizer on the same surface without returning to the surface[23]. There are various methods for evaporation although, in many cases, there is only one optimum evaporation method[24]. Basically, optimum evaporation depends on accurate selection of evaporation method, evaporation source and evaporation temperature[25]. The method used depends on the purity degree of thin layer; of course practically, evaporation system, pieces and other devices can be influential[21].

Steel preparation for coating: The operations of hard working

steel tools include three stages: Heating steel up to austenitic area to form austenite, decreasing the temperature of steel from austenite temperature to be turned into Martensite, Tempering to omit remained austenite and create carbides in Martensite.

Austenitizing: Austenitizing is one of the most important stages of hard working steel tools operations[26]. The followings are some highly important points about Austenitizing: In this operations, atypical and rapid growth of grains as well as piece deformation should be avoided, Decarburization and oxidation of alloy elements should be considered at piece level and an appropriate atmosphere should be selected with chemical compound of steel, Solving carbides influences chemical compounds and subsequently, harden ability of steel, so autinitizing temperature and the time of its maintenance should be controlled.

Autinitizing temperature of high-alloy steel tools is greater than low-alloy steels[27]. It is due to hard solving of MC, M_6 C, $M_{23}C_6$, M_7C_3 carbides which are present in annealstate[28]. Solving these carbides depends on the used anneal, autinitizing temperature and the time of its maintenance[29].

Quench: In steel tools, austenite is transformed into Marten site through quenching steel from austenite temperature. Some austenite is remained with Martensite[30]. This transformation takes place when cooling rate is very high or alloy elements of steels postpone austenite transformation into carbide and ferrite[31]. Typical quenching environment used for steel include water, saltwater, oil, molten salt, neutral gas, and air that selecting each of them depends on chemical compound and thickness of steel tool.

Annealing: Due to internal tensions created during rapid quenching, almost all hardened pieces are relatively fragile. Therefore, steels are rarely used after quenching and in hardened conditions unless in exceptional cases such as when there is a high need to hardness or for low-carbon steels. Usually, steel should be annealed after quenching and before using. Annealing is to heat hardened steel up to temperature below A_{e_1}, maintaining for a certain time and then slowly quenching up to the room temperature; temperature and the time of heating depend on chemical compounds of steel, dimensions of piece and expected mechanical properties. As a result of annealing, internal tensions are decreased and then, fragility is decreased. In return, hardness and strength of hardened piece will be decreased to some extent.

Methodology

Quantometre: Quantometre is a device through which the presence of various elements cab be measured in such a way that firstly, a piece surface is prepared and grinded to gain a completely even surface free from any contamination and oil; and then, the specimen is placed on the part to be sparked. This test is based on optical emission. The specimen is evaporated

due to electrical discharge, atoms and ions resulted from atomic evaporation are stimulated and radiate; and the radiated light passes through an optical fiber within an optical spectrometer. Considering the range of wave length of each element's light emission, the most appropriate emission line is selected in specimen to measure the concentration of element. The intensity of each element is with its concentration in specimen and the percentage of elements in specimen can be directly computed using standard samples and calibration curves. It should be considered that in some devices, there is no space limit to work on various samples and the specimen can even be investigated without any destruction. But in most devices, specimens of every size cannot be tested due to the limitation of the place of samples positioning and only specific dimensions cab be tested. Of course, it should be noted that the results obtained from these devices are more accurate relative to the devices placing pieces in open space to be sparked.

X-ray diffraction spectrometry with grazing incidence angle: X-ray diffraction experimental method is used to create the pattern of diffracting this ray. In electromagnetic spectrum, X-ray area is placed in the range between γ ray and ultraviolet ray. Using this spectrum area, information of structure, substance type, and material fraction determination can be obtained. Sometimes, it is hard to analyze thin films causing to intensify low diffractions relative to substrate and ground. The compound of these two makes the detection of existing phases, low intensity and back ground diffracted peaks difficult.
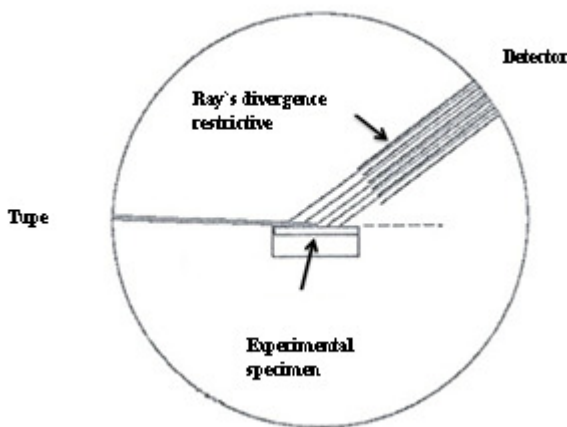

Figure-1
The Cycle of Measuring X-Ray Diffraction Spectrometry with Grazing Incidence Angel

Consequently, a specific technique should be used to analyze thin films. Gazing incidence angel with parallel beam geometry is the most common technique to analyze thin films (100Å). By increasing the path length of encountering X-ray of between-layer can be increased leading to phase detection. Constant ray encountering with the surface of the sample creates very small angel increasing the path length of X-ray within thin film. It helps the increase of diffraction from the surface and the

decrease of diffraction intensity of substrate. During the analysis, only detector turns around the angel, so this encountering angel, the path length and the area of radiated by X remain constant. Figure1 presents the performance of device.

For the layers with thickness of 100 to 200Å, grazing incidence analysis should be used. Of course, this technique has its own limitations. Firstly, this technique acts well only in very even surface and all surfaces cannot practically be provided well even. Secondly, major part of X-ray is wasted in grazing angel. In case of supposing an encountering angel of 0/1 and a surface of 1 cm^2, only $2\mu m$ of X-ray encounters with this surface and the rest encounter to the surrounding or pass through the specimen.

Energy diffraction X-ray (EDX): EDX is an analytical method used to analyze the structure or chemical characteristics of a specimen. It relies on investigation of interaction between an X-ray excitation sources and a specimen. Descriptive capabilities of this\is method are generally based on the principle that each element has a unique atomic structure allowing a unique set of peaks in X-ray spectrum. To excite X-ray emission index of a specimen, a group of high-energy charged particles like protons or electrons, or a group of X-ray are focused. An atom in resting specimen including ground state of electrons (or unexcited) at discontinuous energy levels or electron layers is connected to nucleolus. The applied ray may stimulate an electron in inner shell and remove it from shell while it creates an electrical core in the early place of electron. Then, an electron with the energy higher than an external layer fills the hole and the energy difference between high energy and low energy layers can be released in the form of X-ray. The number and energy of X-rays emitted from a specimen can be measured through an energy diffraction spectrometer. Since X-ray energies indicate energy difference between two layers as well as atomic structure of element emitting the ray, the elements compound can be measured.

Friction coefficient using pin on disk: In friction test, the degree of friction can be obtained from weight reduction or volume reduction. To measure volume reduction or friction depth, specimens should be placed in environment temperature while it is not practical during work due to thermal expansion and contraction and complex reaction of surface layer film or chips.

The degree of friction depends on force, distance, environment, the way of conducting test, device, type of coating, and speed. In friction test can be used to obtain friction coefficient using load cell, continuously, friction force or having applied vertical force. There are various methods to measure friction relatively and friction in standards such as pin on disk, reciprocating friction and etc. The appropriate method is selected with respect to working conditions. In pin on disk method, object is tested by a pin under force and after passing a certain distance, its weight reduction (volume reduction) is obtained based on distance and

reported by appropriate curves.
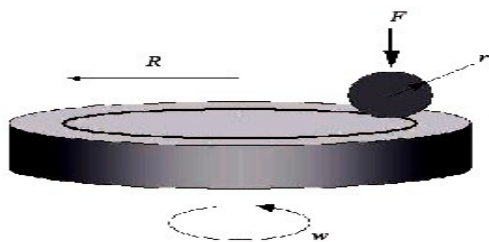
Figure-2
An Image of Pin on Disk Test

Before test and weighting, specimens are cooled using non chloride solutions and the solutions that do not form film. For magnetic object, its magnetic characteristic should be removed. Afterwards, specimens are weighted up to accuracy of 0/0001 or its dimensions are measured up to 2/5 mm. then, appropriate force is applied and the number of necessary cycles is regulated. It should be noted that the test should not be interrupted. After test, the specimen is taken and cleared and color change, form change around friction place, fine cracks, and lump are controlled. Test of the study were performed using pin on disk and the main objective of the test is to examine friction coefficient of specimens. Figure 2 shows the schematic of pin on disk test.

The way of coating TiAlCrSiCN: After the stage of preparing the specimen surface for bond, Argon gas is cut off to make the vacuum of 10^{-7} mbar. Hybrid gas (70% Nitrogen and 30% Argon) is entered into tank to gain the pressure of 10^{-3} mbar and then, the target $Ti_{40}Al_{50}Si_{10}$, with amperage of 150A and the voltage of 40V is on for 5 minutes to coat TiAlSN. The point is that voltage of connecting to piece is 400V (DC) during all coating stages. Entering gas and evaporating metal particles are ionizied through ion source. After the end of the first layer`s time, the target $Cr_{30}Al_{70}$, with amperage of 150A and the voltage of 40V is on for 5 minutes to coat AlCrN in TiAlSiN layer with the same gas condition. Then, hybrid gas (50% Methane and 50% Nitrogen) is entered into tank to gain the pressure of 10^{-3} mbar. In this stage, magnetic diffraction (filtyer arch) is on and subsequently, two targets of AlCr and TiAlSi are on simultaneously and the main coating is created. The whole process is done in 50 minutes for the main coating, every 10 minutes a 10-miniute pause is considered to quench the system. Figure 3 depicts TiAlCrSiCN coating process.

The main coating is done on two primary layers only due to high adherence of this element to the layer and substrate. For example; good adherence is not created when the main coating on the substrate due to the great hardness difference between layer and substrate which leads to high superficial tensions. During quenching, coating causes to separate coating from substrate. To prevent this, the parameters of the devices should be changed causing that created coating precipitates on substrate

in the form of graphite layer and the slightest touch, even hand touch on the surface coating remove the coating from the substrate; and it is because of the presence of a high percentage of elements such as Chromium and Carbon in cold work steel tools.In vapor deposition process, Chromium and Carbon are not so interested to precipitate and bond with their peers in substrate and causes the lack of adherence to the substrate. Due to the mentioned reasons, firstly, the softest layer is coated with this difference that silicon is placed at the first layer and the device can calibrate only two targets, due to the lack of elements like chrome and carbon coating; and finally, the main coating is created with a higher hardness compared to two early coatings.
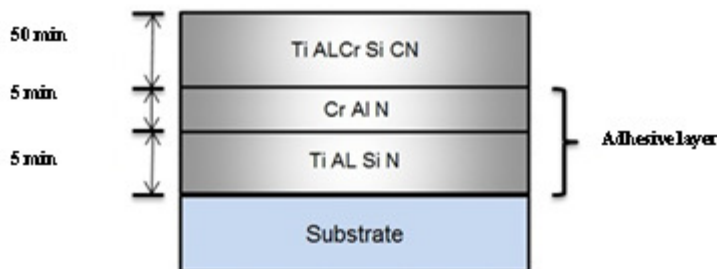
Figure-3
Time and Order of TiAlCrSiCN

Results and Discussion

Figure 4 shows TiAlCrSiCN coating and table-1 presents atomic percentage of its chemical compound. As the results reveal, chemical compound has been considered in an area of surface to maximize the accuracy of obtained data.

As it is observed, by adding cathode containing silicon alloys with making other conditions constant, this element is entered into chemical compound of coating structure. As the early state, in this case, titanium has the greatest amount in addition to high quantity of aluminum in the used targets compound. In chemical compound of this coating, silicon has the least quantity. The target used to enter silicon has only 10% silicon, so reaction atmosphere should have the least percentage of silicon. Low quantity of silicon in coating structure is the evident of this claim.

Table-1
The Chemical Compound ofTiAlCrSiCN Coating Based on Atomic Percentage

Elements	N	C	Si	Cr	Al	Ti
Atomic percentage (%)	8/7	13/3	2/5	4/9	28/5	42/1

As it is observed, both states have the same chrome quantity. Considering the fact that the used target and also coating conditions are similar in both states, so there is similar quantity of this element in coating atmosphere.
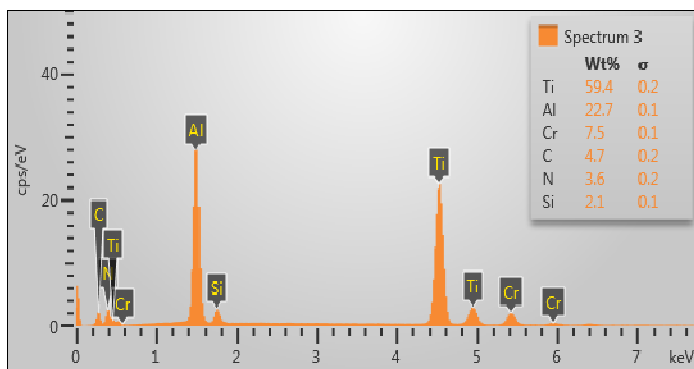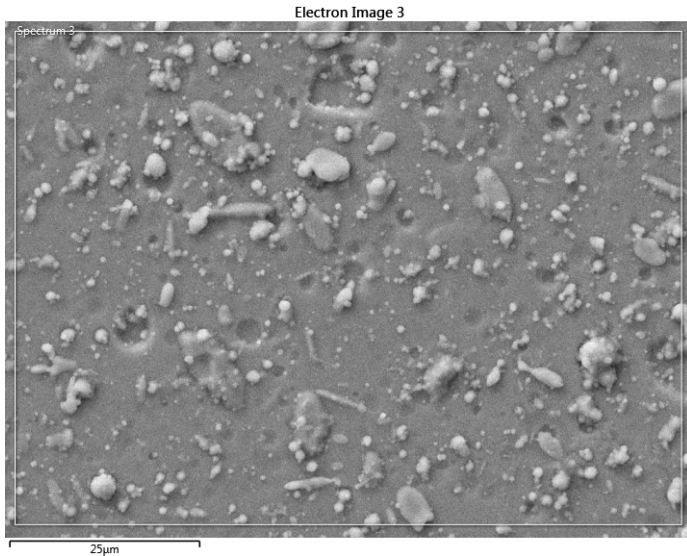
Figure-4
The Results of Chemical Compound Obtained from EDX Method Related to TiAlCrSiCN Coating Based on Weight Percentage

With regard to the point that the target used in the first sate has only a 10 percent difference of aluminum in its chemical compound, the quantity of this element is decreased in chemical compound of coating up to 4%. It can also be considered as the reason of 0/5 increase in titanium quantity since removing aluminum, as a more active element relative to titanium, the reaction atmosphere provides better conditions for the reaction of this element. Entering silicon into the chemical compound of the structure changes the quantity of carbon in the structure. It is likely that this element compared with aluminum has a higher tendency to react with carbon particles in atmosphere leading to the increase of the element in the structure. Adding active elements like silicon, aluminum and titanium increases the presence of carbon in coating structure. Hence, it can be concluded that in coating conditions used in the study, carbide compounds of this element are more stable compared to nitride compounds.

Considering the results obtained from the chemical compounds indicates the presence of silicon in TiAlCrSiCN coating structure and also with respect to the fact that there is no peak in the results of X-ray diffraction related to nitride and carbide silicon compounds, it can be concluded that TiAlCrSiCN coating is in the form a nano-composite coating in which TiAlCr(C,N) crystals are scattered in Amorphous ground of Si(C.N) since the nitride if steels containing Si are considered as nano-composite. Figure 5 presents the schematic of microscopic structure of coatings. Also, with respect to the peak existing in angle 41°, it can be expected that the presence of silicon in the primary layer decreases TiAlN phase or decrease crystallite since in sectional image obtained from FESEM of the specimen, the thickness of layer is decreased and its related columns present a border with less clarity since elements like silicon tends to amorphous structure in coating conditions used in the study.
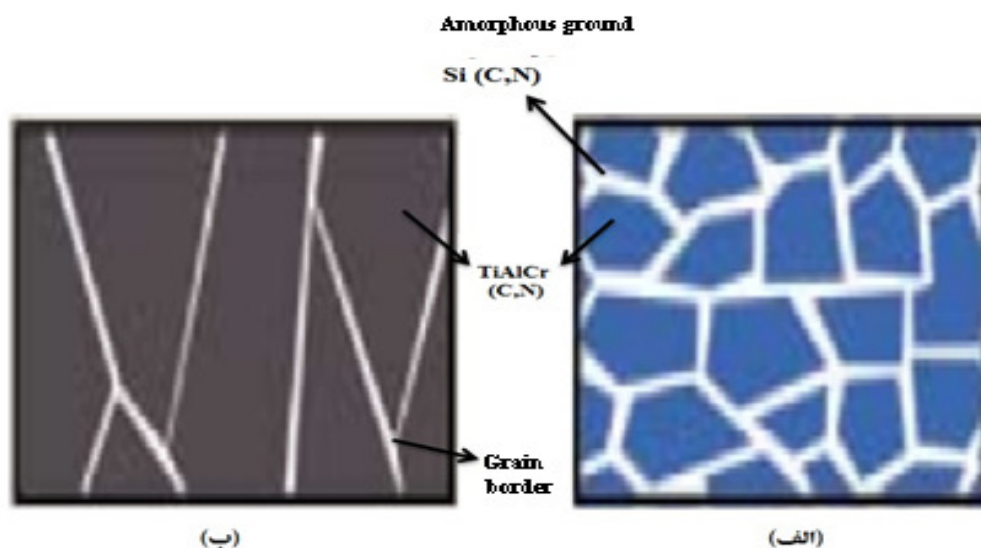

Figure-5
The Schematic of Microscopic Structure of Coatings a) TiAlCrSiCn and b) TiAlCrCn

Table-2 presents the results of computations of grain size in each coating using the results of X-ray diffraction and Scherer`s relation. Changing the place of related peaks in the two coating indicates the change of distance of their pertained crystalline plates which can be related to the remained tensions in the structure. Since adding silicon to the coating compound and creating amorphous ground increase strain and subsequently, tension in the structure, pertained peaks of TiAlCrSiCN compounds are transferred to smaller angels; so it can be claimed that the presence of silicon and amorphous phase formation cause tensile stresses in crystals.

The results of EDS test (figure-6) reveal that these particles are different in chemical compounds. For example, following figure shows the analysis of chemical compounds of one of the particles existing in the surface of TiAlCrSiCN. Comparing the obtained results with the results pertained to the coating (table-

2), the decrease of some elements such as aluminum and chrome can be observed. It can be due to the fact that such elements penetrate in surface more and penetrate to the substrate by being separated from the target and then reacted. Or it can be stated that other elements such as carbon, nitrogen and silicon show less penetration in the comparison and thus, have more participation in the atmosphere. Therefore, the particles formed in this area contain a higher percentage of the mentioned elements while the particles are made of the coating materials.

Table-2
Chemical Compounds of Particles existing in the Surface of TiAlCrSiCN (atomic percentage)

Elements	N	C	Si	Cr	Al	Ti
Coating	8/7	13/3	2/5	4/9	28/5	42/1
Particle	11/9	22/3	5/2	2/1	16/1	42/4
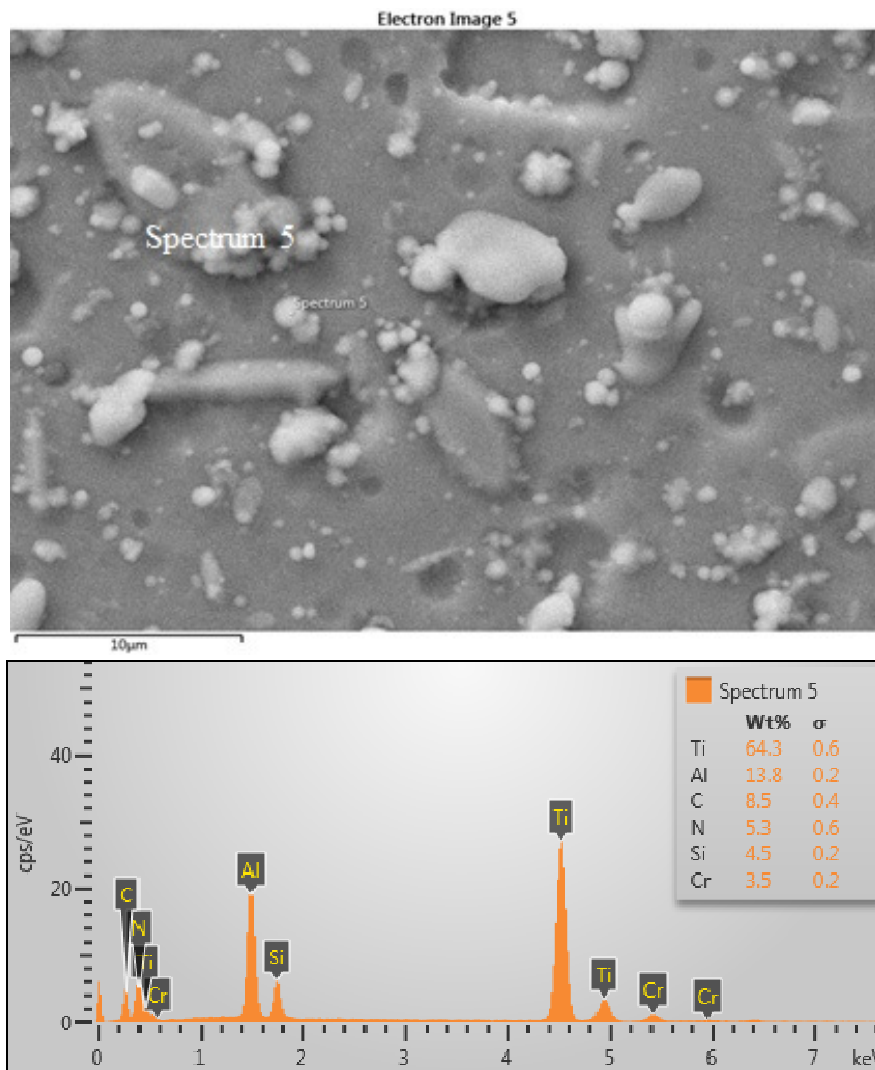
Figure-6
The Results of Chemical Compounds Obtained from EDX Method Pertained to the Particles Existing in the Surface of TiAlCrSiCN

Conclusion

Based on the findings, adding silicon to chemical compound of the coating causes to create nano-composite structure in the form of TiAlCrCN crystals in SiCN amorphous ground. Also, the coating surface always is full of circular particles formed in the reaction atmosphere and deposited on the coating surface. The circular particles increase roughness of the surface and subsequently, the quantity of coating`s RMS. The circular particles also locally increase the friction coefficient of coating surface.

Recommendation: Some recommendations obtained from the present work are presented as follows: Investigation and comparison of TiAlCrCN and TiAlCrSiCN coatings` corrosion. Investigation and comparison of erosion and corrosion characteristics of TiAlCrCN and TiAlCrSiCN coatings. Investigation of the effect of silicon quantity in mechanical and structural characteristics of TiAlCrSiCN coatings. Investigation of TiAlCrSiCN coatings` adherence and the effect of silicon in its intensity. Using TEM test to investigate structural coating and the effect of silicon in the structure. Conducting actual industrial tests on tools coated with TiAlCr(Si)CN and investigating the effect of its presence to increase the tools` capability.

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