



An Investigation of TiAlCrCN Nanoparticles Coating Deposited by Physical Vapor on Cold Work Tool Steel

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Abstract

In this work, multi-component TiAlCrCN coatings are deposited through physical vapor deposition using arc cathodic PVD on cold work steel tools substrates (Spk 1/2080). It attempts to investigate the effect of silicon in the structure and mechanical characteristics of TiAlCrCN coatings. In this study, energy-dispersive X-ray (EDX), X-ray diffraction (XRD), field emission scanning electron microscope (FESEM), and atomic electron microscope (AFM) has been applied to characterize coating in terms of chemical compound, structure, morphology of surface and cross section, respectively. Nanoidentation, micro hardness and pin on disk test have also been used to analyze mechanical characteristics of coating.

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

Introduction

Many parts of modern technologies need materials with improved surfaces. Surface engineering provide conditions that adjust equipments and tools with their working environment by improving and manipulating their surface characteristics¹. On one hand, it provides some specific efficiency which can include high resistance for new applications. In spite of the recent advancements in engineering, science and developing needs of multi-function films (layers) and coatings, micro-nano mechanical characteristics of thin films` structure and coatings have widely been considered during the recent years². Nanostructure materials are appropriate to be used for erosion and corrosion resistant coatings due to atypical physical, chemical and mechanical characteristics³. There are various methods to produce nanostructure including physical and chemical vapor accumulation, spraying processes, gel-cell process, electrochemical deposition, intense plastic deformation, and etc.⁴.

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 elements⁵. 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 product⁶. However, secondary products also are produced which are exited through gas⁷. 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 nitride⁸. 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 components⁹.

Many researchers have studies hard coatings on cutting tools. Hofmann et al used binary elements like CrN and TiN as hard coatings to increase the efficiency of cutting tools and corrosion as well as erosion resistant pieces in their studies¹⁰. On one hand, in the recent decade, ternary elements of Cr-X-N which can be alloys such as Ti, Al, Si, B, C, and Ni have been used a another method to improve surface characteristics of such pieces¹¹. For example, Canon et al (2004) based on their study reported that CrAlN coating compared with CrN coating showed higher hardness (about 20-32 Gpa) and better resistance against oxidation¹². Based on the studies done by Hofmann et al and also the studies conducted by Ragend et al, it was revealed that using quaternary elements in spiral drilling construction in extrusion frames construction led to a considerable improvement in surface characteristics of such pieces¹³. Bai et al also stated that coatings containing quaternary elements compared to ternary coatings showed higher hardness and resistance against corrosion although friction coefficient of such coatings has been reported to be very high¹⁴.

Atmospheric pressure CVD (APCVD): APCVD is a depositional method done in atmospheric pressure. Atmospheric pressure increase deposition film speed up to 600 to 1000 nm/min. homogeneous nucleation occurs due to rapid condensation of gas molecules, and the films produced from this method show low impressionability and also a considerable coating speed of 1 μm in minute¹⁵.

Atomic layer CVD (ALCVD): In this process, two primary materials, for example water and $\text{Al}(\text{CH}_3)_3$ are entered

alternatively into the reaction tank¹⁶. One of these materials is attracted on the surface of a wafer in the absence of another element and it does not change chemically¹⁷. It is continued to attract the substance. Then, the second substance is added into the environment and the interaction is done. The thickness of film is controlled with the quantity of substance entering into the reaction tank. In this method, the thickness of film can be exactly controlled¹⁸.

Low pressure CVD (LPCVD): This process is done in a pressure lower than atmospheric pressure. In low pressures, the interactions of gas phase are decreased unintentionally and cause to improve evenness of film on wafer. Another method done in vacuum and has less application is ultra high vacuum chemical vapor deposition (UHVCVD)¹⁹.

Metal organic CVD (MOCVD): In this process, metal organic ingredients like tantalumethoxide, TDMA are used to produce coatings made of TiO₂ and TiN. TiO₂ sets on the surface of metal and TiN is applied for tools' harness. When this method is done in very high vacuum, it is called metal organic molecular beam epitaxy (MOMBE)²⁰.

Microwave power CVD (MPCVD): In MPCVD method, the raw material is placed in plasma created by microwaves and gas mixture (usually the mixture of methane and hydrogen)²¹. This interaction induces heterogeneous nucleation process. At the next stages, the nucleuses are adhered together and create an even film²². Orientation of primary nucleuses depends on applied DC flow and the impacts of plasma. The morphology created through this method depends on several parameters such as pressure, gas combination, and silicon context²³.

Plasma enhanced CVD(PECVD): In PECVD method, plasma is used to increase the speed of chemical reaction. Plasma vapor's method causes to produce deposit in low temperature which is necessary in producing semi-conductors due to creating the appropriate product. VVD resulted of plasma can be done in the temperatures lower than thermal CVD²⁴.

Rapid thermal CVD (RTCVD): Thermal lamps or other methods are used to heat wafer rapidly in RTCVD. Heating wafer causes interaction in the surface of wafer and decreases its quantity in gas phase. In this case, less suspended nanoparticles are produced²⁵.

Remote plasma CVD (RPCVD): RPCVD is a method similar to PECVD and the only difference between them is in the position of wafer relative to entering plasma. It can be explained in this way that wafer is not directly placed in the area of plasma discharge. Removing wafer from plasma area causes the process is done in room temperature²⁶.

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. 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. It is due to hard solving of MC, M₆ C, M₂₃C₆, M₇C₃ carbides which are present in anneal state. Solving these carbides depends on the used anneal, autinitizing temperature and the time of its maintenance.

Quench: In steel tools, austenite is transformed into Martensite through quenching steel from austenite temperature. Some austenite is remained with Martensite. This transformation takes place when cooling rate is very high or alloy elements of steels postpone austenite transformation into carbide and ferrite. 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 tool²⁷.

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 steels²⁸. Usually, steel should be annealed after quenching and before using. Annealing is to heat hardened steel up to temperature below A_{e1}, 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 properties²⁹. 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 extent³⁰.

Methodology

Quantometre: Quantometre is a device through which the presence of various elements can 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 can 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 sparked³¹.

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.

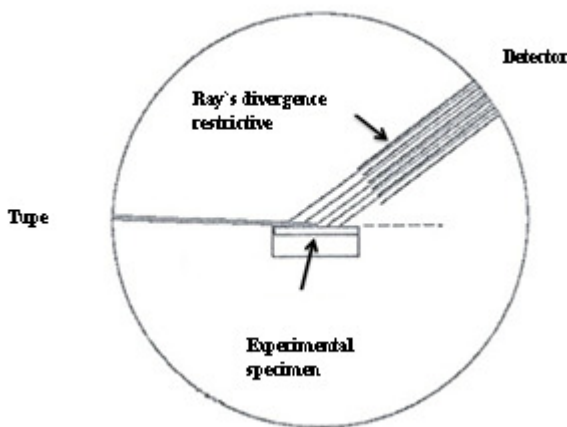


Figure-1

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 cm², only 2μm 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 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.

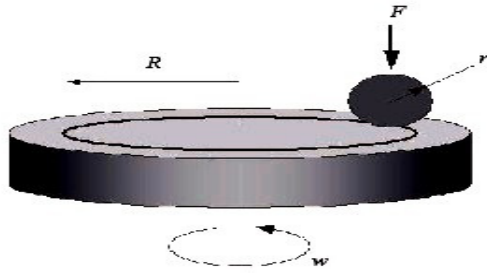


Figure-2
An Image of Pin on Disk Test

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 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.

The way of TiAlCrSiCN coating: 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 TiAlSiN. The point is that voltage of connecting to piece is 400V (DC) during all coating stages. Entering gas and evaporating metal particles are ionized 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 (filter arc) 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-minute pause is considered to quench the system.

The way of coating TiAlCrCN: After placing the specimens into the device, vacuum is made to reach the pressure of 10^{-7} mbar. Then, argon gas is entered into the tank to gain the pressure of 10^{-3} mbar. Bias voltage of 2/5 KV is connected to the pieces while the pieces have rotating movement. In such conditions, argon gas is ionized and positive ions of argon are bombed on the surface of the piece leading to complete clearance of piece surface through positive micro arcs of the surface and stimulation of surface atoms as the result of encountering negative ions of argon. In other words, they reach to a higher balance and are prepared to bond with coating. After preparing the surface of specimen for bonding, argon gas is disrupted to recreate a vacuum of 10^{-7} . 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 TiAlSiN. The point is that voltage of connecting to piece is 400V (DC) during all coating stages. Entering gas and evaporating metal particles are ionized 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 TiAlN 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 (filter arc) is on and subsequently, two targets of AlCr and TiAl 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-minute pause is considered to quench the system. Figure-3 depicts TiAlCrCN coating process.

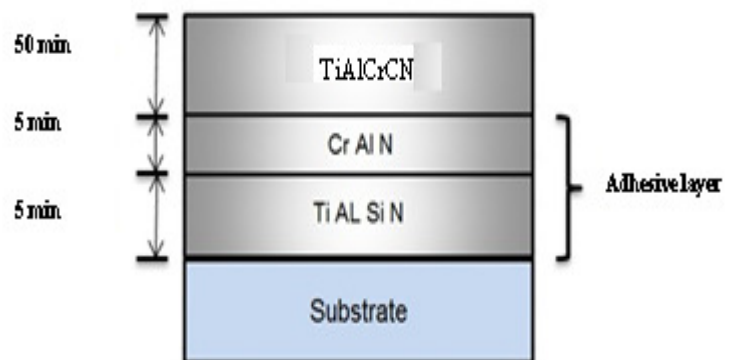


Figure-1
Time and Order of TiAlCrCN

Results and Discussion

To determine chemical compound of coating's structure resulted in the research, *energy dispersive spectroscopy* (EDS) has been used. Figure-2 presents chemical compound pertained to TiAlCrCN coating.

To obtain the chemical compound of the surface, point analysis was done and a square with the dimensions of 10mm. 10mm was considered to obtain the mean value of total chemical compound of the surface. TiAlCrCN coating contains all considered elements such as titanium, aluminum, chrome, carbon, and nitrogen. Table-1 presents the chemical compounds in atomic percentage.

As shown in table-1, titanium (Ti) and aluminum (Al) have the most percentage in the chemical compound of the structure. Although aluminum has the highest ratio in the target used in this stage of the coating process, aluminum has a high fraction

in other three stages. However, the percentage of titanium is more than aluminum in the structure. It can be concluded that in specific conditions of coating applied in the present study, Ti of the coating is more active in the reaction atmosphere and deposited on the surface. Due to weaker chemical bond in methane molecule compared to Nitrogen, methane creates more ionized coating conditions and more active particles. Therefore, with the presence of nitrogen gas in three stages of coating process, the percentage of carbon resulted from methane is the highest in the structure. However, it is expected that a less quantity of chrome exists in the structure of coating due to using a target with low percentage of chrome ($Cr_{30}Al_{70}$).

Table-1
Chemical Compound of TiAlCrCN Coating
(Atomic Percentage)

Elements	N	C	Cr	Al	Ti
Atomic percentage (%)	9/2	11/8	4/8	32/6	41/6

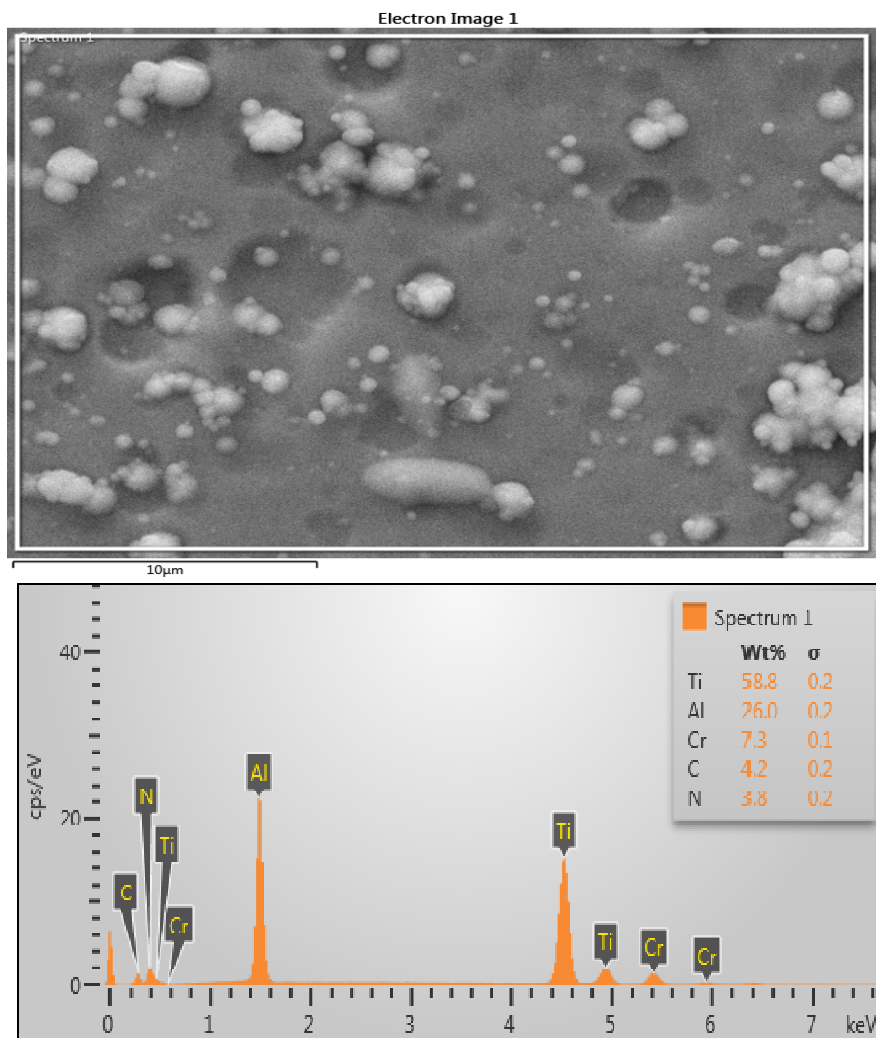


Figure-4

The Results of Chemical Compound Resulted from EDX Pertained to TiAlCrCN Coating (Weight Percentage)

Figure-5 shows morphology of the surface pertained to TiAlCrCN coating with the zoom of 4000. According to the image, it is observed that the surface of coating is even and condense covered by microparticles (0/7 mm). The particles are appeared in the shape of hunk in some areas. Since the particles are circular, it is likely that the particles are formed at the atmosphere above the coating surface (not at the coating surface) and then deposited on the surface. So, the adhesive strength of the particles to the surface is less since very low penetration is occurred between the particle and surface due to low working temperature.

Therefore, the particles are detached from the coating surface by the growth of layer and atoms` encountering as well as their uneven and increasingly growth, and their heavy weight (figure-6). The high points which can be seen in even surface are the results of particles and hunks detachment. Figure 4 depicts the

10000 zoom of the surface of the same coating.

As it is observed, there are some islands on each island leading to uneven growth of islands and subsequently, hunk formation. The presence of hunks in the coating surface and during its growth prevents the regions under these hunks and particles from growing just like the regions exposed to the atmosphere. Figure-5 shows the image of FESEM of TiAlCrCn coating`s cross section. As the former coating type, this coating has also a condensed surface without porosity in micro dimensions. Further, in this case, some hunks are created in the coating surface due to the aforementioned reasons. In this case, the mean of particles` size equals 0/6 mm. As figure-7 shows, higher zoom indicates that the surface in nano scale has also condensation without porosity. The shown particle pertains to the particles created in atmosphere or hunk.

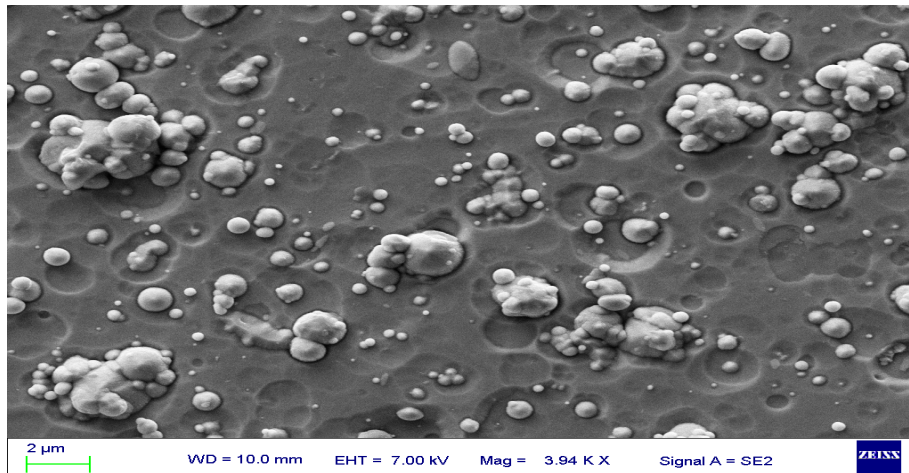


Figure-5

The Morphology of the Surface Pertained to TiAlCrCN Coating with the Zoom of 4000

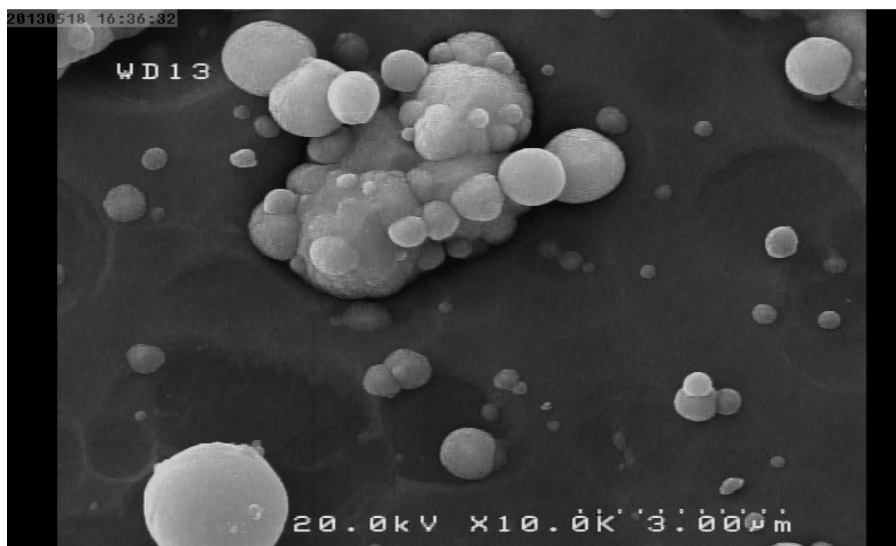


Figure-6

The Morphology of the Surface Pertained to TiAlCrCN Coating with the Zoom of 10000

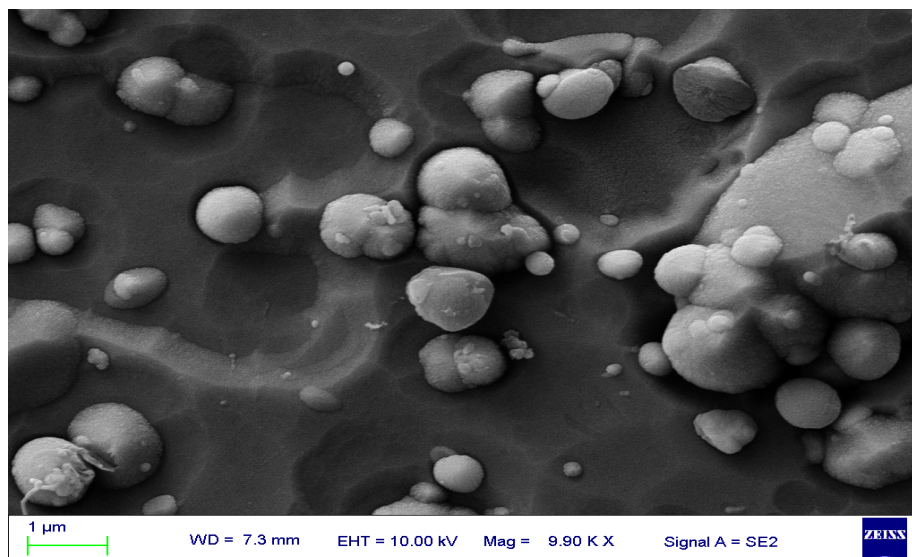


Figure-7
FESEM Image Pertained to TiAlCrCN Coating with the Zoom of 10000

Conclusion

Based on the findings of the present study, it can be concluded that:

Adding silicon to the structure of TiAlCrCN coating decreases the roughness of the coating surface. Adding silicon to the structure of TiAlCrCN coating increases the hardness of the coating surface. Adding ceramic and hard coatings increases the hardness of substrate surface by creating sandwich composite. Composite becomes harder by adding harder layer (silicon coating). TiAlCrSiCN coating compared with TiAlCrCN coating is more resistant to plastic deformation. Adding ceramic surface coatings intensely decrease the friction coefficient of the substrate surface, so the tribological characteristics will be decreased to a large extent. 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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