RESEARCH PAPER

Self-Cleaning Properties of TiN/CrN Nanoscale Multi-layer Deposited on Surgical 420C Stainless Steel

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ABSTRACT

The present paper focuses on the investigation of self-cleaning properties based on studding of water repellency and blood repellency for TiN- and CrN single-layer and TiN/CrN nanoscale multi-layer coatings deposited via Cathodic Arc Evaporation (CAE) method on medical grade 420C stainless steel substrate. X-ray diffraction (XRD) method and Field Emission Scanning Electron Microscope (FESEM) was used to characterize microstructure. Surface roughness parameters were measured by using Taylor-Habsson method. Blood contact angle and water contact angle measurements test were applied to characterize the self-cleaning properties of the specimens. The analysis of sample data shows that coated specimens have more water contact angle values in comparison to bare steel. Among the coated samples, CrN single-layer has the highest water contact angle (80°) due to its lower surface roughness (Ra=0.189µm) among the other samples. Moreover, the findings of the paper prove that samples had inverse behavior against blood and water in the contact angle measurement test. Bare steel has higher blood contact angle (76.1°) and more blood repellency than the coated specimens. It seems that the different behavior of samples against water and blood in contact angle tests is due to the nature of two fluids. TiN/CrN multi-layer coating results had minor differences in water and blood contact angle tests (67.3° and 62.2° respectively).

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INTRODUCTION

Nosocomial infections (NIs) are important in terms of their potential for high mortality, morbidity and elevated hospital costs. The rate of nosocomial infections (NIs) reported in USA is 2.8% and in European countries is 2-5%. NIs caused 5,000 deaths per year in England and cause 17500-70000 annual deaths in the USA. These infections cost the UK's National Health Service (NHS) one billion pounds each year which was the equivalent of 1% of the total national hospital budget and between 17 and 25 billion dollars added to health costs every year in the USA [1-2]. Various studies done in Iran have shown the rate of NIs varying from 8% to 10%. There are over 100,000 hospital beds in 830 hospitals in Iran that approximately 6 million patients admitted annually [3]

Based on the results released by the National Nosocomial Infections Surveillance (NNIS) on 100 hospitals with more than 200 beds each and 6,616,520 studied patients between 2007 and 2010, surgical site infections (26.8%) was one of the main cause of NIs in Iran [4].

Infections related to such biomedical instruments and devices are responsible for at least 1.5–7.2% post-surgery complications depending on the type of surgical procedure [5].

Surgical instruments tend to be contaminated to a certain level during surgeries by micro-organisms that inhabit the skin and organs. Surgical instruments could act as fomites for the pathogens of surgical site infection even if the

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surgical field is not apparently contaminated [6-8].

Proper reprocessing of reusable surgical instruments and medical devices is a critical infection prevention strategy. Cleaning is a key factor to overcoming these infections. An instrument that has not been properly cleaned cannot be effectively disinfected or sterilized and consequently using of the contaminated surgical instruments can result in further severe infection. The level of bacterial contamination is a vital parameter that will determine the time period of the sterilization or the disinfection and efficacy of these processes. The Occupational Safety and Health Administration (OSHA) defines decontamination as, "The use of physical or chemical means to remove, inactivate, or destroy blood-borne pathogens on a surface or item to the point where they are no longer capable of transmitting infectious particles and the surface or item is rendered safe for handling, use, or disposal" [8-11]. In this study the OSHA definition will be used to both cleaning and decontamination. Besides, to keeping (to keep) instruments free of contamination during surgeries, cleaning of instruments should occur as soon as possible after they are used [10]. Results of some studies have been proven that the cleaning of stainless steel surgical instruments during the first 6h after the surgery is essential in order to ensure effective disinfection and sterilization [11,12].

The process that should be followed for sterilization of reusable medical instruments is shown in Fig. 1.

The self-cleaning characteristic is related to the surface contact angle. Self-cleaning coatings are broadly categorized into two groups: hydrophilic and hydrophobic. Both of the groups clean themselves by the action of water. In a hydrophilic coating, the water is made to spread over the surface, which carries away the dirt, blood and other contaminating particles, whereas in the hydrophobic technique a water droplet contact with such a surface can easily roll across the surface on which it rests, collecting dust or other impurities [13-16].

Contact angle value which is dependent on surface roughness can determine the surface free energy and also efficiency of the self-cleaning coatings [17-19].

Surface roughness is a 2D parameter of a material surface that affected the self-cleaning process. Irregularities of the material surfaces normally promote blood adhesion and biofilm accumulation. This is due to the increased surface area and depressions in the roughened surfaces [5,7]. There are many different roughness parameters. Ra (arithmetical mean of surface roughness of every measurement within the

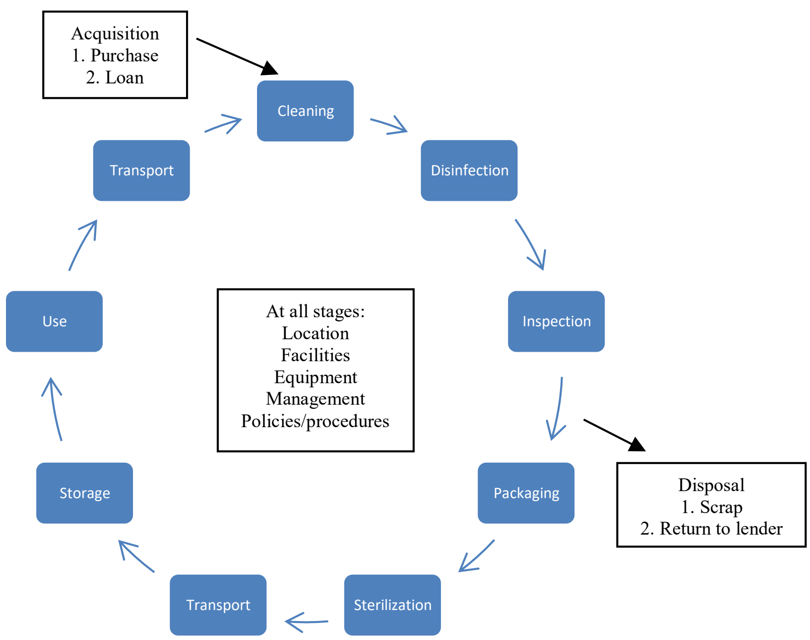


Fig. 1. Standard sterilization protocol for reusable instruments [11]

total distance ½ roughness average) is the most universally used roughness parameter since it is easy to define and to measure providing a good general description of height variations [20,21].

Metals have a high surface energy and are negatively charged and hydrophilic as shown by water contact angles. Near a hydrophobic surface the water is less structured in terms of intermolecular hydrogen bonding between the water molecules, while near a hydrophilic surface water is more structured [7,22,23].

420C stainless steel is one of the most important materials used in the manufacture of general surgical instrument due to superior mechanical strength, ductility, elasticity, corrosion resistance and low cost [20,22,23]. However self-cleaning properties of this alloy is relatively poor [21,23]. As noted, an important way to create a self-cleaning surface is wetting controllability. The ability to control the wetting properties of stainless steel is acutely useful because this alloy has many applications in scientific and industrial fields [17]. Surface modification with advanced materials is an appropriate method to reduce the risk of infections caused by microorganisms and altering the self-cleaning phenomena [24].

Coating is an important area of surface modification. Single-layered and multi-layered coatings deposited by physical methods are increasingly used in all branches of industry practically for surgical instruments [25]. TiN and CrN are two conventional coatings in medical applications that have high hardness, good wear and corrosion resistance. These coatings are physiologically inert, non-toxic, non-carcinogenic and usable in implantable devices approved by the Food and Drug Administration (FDA) of USA [25-28].

Heretofore many researches have been done on fabrication of TiN- and CrN based coatings for enhancement of mechanical, corrosion resistance and antibacterial properties of stainless steel. However few studies have been done on fabrication of nanoscale TiN- and CrN based coatings deposited on 420C stainless steel with studding both blood repellency and water repellency for determination of self-cleaning properties to prevent infection in surgical instruments. Consequently, the main goal of current study is the determination of the effect of the nanoscale TiN- and CrN based coatings deposition on water repellency and blood repellency characteristics of 420C stainless steel with the aim of easier cleaning and thus decreasing the risk of contamination and infection of general surgical instruments.

MATERIALS AND METHODS

Substrate preparation

In this study 420C stainless steel with 3mm of thickness was chosen as a base metal. Optical Emission Spectrometer (OES) confirmed the chemical composition of this alloy (Table 1).

At first surface hardening of stainless steel specimen took place in salt furnace at 1037°C and were quenched in warm oil. Then specimen was tempered at 150°C for 20min for the aim of removing residual stresses. Finally samples were cut to a dimension of 40×60mm^2. Samples were subjected to surface sanding, mirror polishing and finally surface passivation with electro polishing method. Hardness of the substrate was measured 980HV.

Deposition process

The samples were ultrasonically cleaned in acetone and ethanol solutions for 30min. This process was repeated thrice for removing all of the probable contamination on the surfaces. Samples then were introduced in the PVD chamber and subjected to etching and preheating in Argon glow discharge plasma for 15min. to clean up more levels of contamination and to enhance the adhesion of the coating on the substrate.

A CAE PVD equipment model YN Saleh (Yarnican Saleh, Tehran, Iran) was used to deposit the coatings. The working pressure in deposition system was kept at 1×10^{-3}torr. Ti and Cr high purity metallic targets (99/99%) were mounted on vertically opposed cathodes. The Nitrogen gas was fed into the chamber while an AC power supply connected to substrate holders was used as bias output fixed at -100V during deposition process. Temperature of the substrates was 200°C and duty cycle of process was 50%. Substrates in the

Table 1. Chemical composition of examined 420C stainless steel

Elements	C	Mn(max)	P(max)	S(max)	Si(max)	Cr	Ni(max)	Fe
Wt.%	0.45	0.31	0.018	0.003	0.61	14.3	0.33	balance

distance of 150mm were rotating in front of the metallic targets. Through hold activating one of the metallic targets and allowing Nitrogen gas to enter into the chamber, single-layered TiN and -CrN coatings were formed. For deposition of multi-layered TiN/CrN, the Ti and Cr targets were covered intermittently with a steel bulkhead. The total deposition time for all specimens was 90min. and the overall thickness also was about 1.5µm. To avoid rapid cooling, coated samples stayed in chamber for 30min. Finally bare and coated samples were cut to a dimension of $10\times10mm^2$ and $20\times20mm^2$ for tests.

Coatings characterization

Structural characterization and microscopy

The phase structure of the samples were characterized by JEOL (model JDX-8030) X-ray diffractometer (XRD) with an accelerating voltage of 30kV, current 20mA and using the CuKα (λ=1.54Å) radiation. Cross-section as well as thickness of coatings was analyzed by a FESEM model TESCAN, Mira3.

Roughness analysis

Surface roughness of the samples was examined by the Taylor-Hobson equipment model Surtranic25 with a resolution of 0.01mm.

Contact angle measurement

Using water and human blood as liquids of the test, water repellency and blood repellency of the samples were measured via contact angle measurement technique by means of Dataphysics system model OCA-15 plus contact angle analyzer. Prior to these measurements, the samples were ultrasonically cleaned in acetone and deionized water and dried. This test is performed with small droplets of liquids (4µl) were placed on five location of the surface at 27°C and pressure of 90kPa. The angles in the form of degrees were then estimated from the photos taken by the video camera using photo analysis software.

RESULTS AND DISCUSSION

Structural characterization and microscopy

Fig. 2 represent XRD pattern of all specimens. For single-layered TiN, -CrN and multi-layered TiN/CrN coatings, the crystal planes of TiN phase based on ICSD 00-006-0642 is consist of (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2), and for CrN phase based on ICSD 01-076-2494 is consist of (1 1 1), (2 0 0) and (3 1 1) that confirmed the creation of TiN and CrN phases in the multi-layered and single-layered

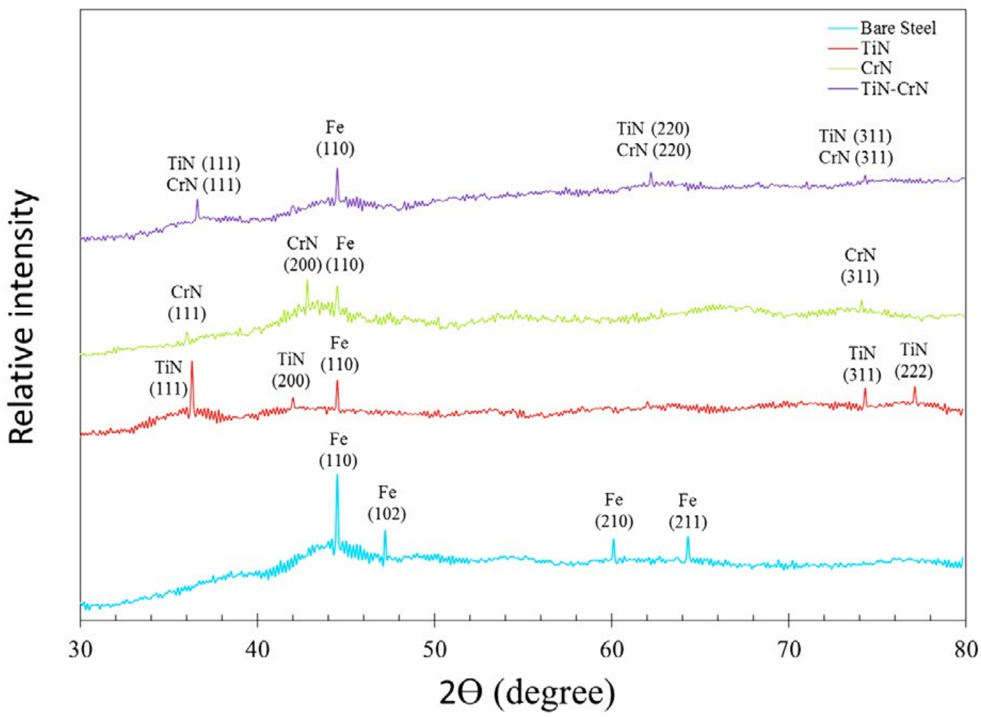


Fig. 2. XRD patterns of the samples

coatings. Peak's weakness is due to low thickness of the coatings.

Surface FESEM images of the samples are shown in Fig. 3. Existence of the pin holes and macro particles in coated samples is due to nature of the CAE method. Macro particles could be due to the rapidly melting of the target material by the arc during CAE process. Melting and boiling points of target materials are important factors in the number and size of macro particles on the surface.

Cross-sectional images of coated samples are shown in Fig. 4. As seen all coatings have the same thickness at about 1.5µm. Also in Fig. 4-c the formation of dense and uniform structure with distinct interface between CrN and TiN layers is confirmed. The bright and dark layers are belonging to CrN and TiN phases respectively. The bilayer period of TiN/CrN multi-layer coating is100nm.

Surface roughness analysis

Surface roughness known as roughness is a component of surface texture. Roughness plays an important role in determining how a real object will interact with its environment. A roughness value can either be calculated on a profile (line) or on a surface (area). The profile roughness parameters are more common. Based on ISO 4287:1997 standard, there are many different profile roughness parameters in use, but Ra is by far the most common, though this is often for historical reasons and not for particular

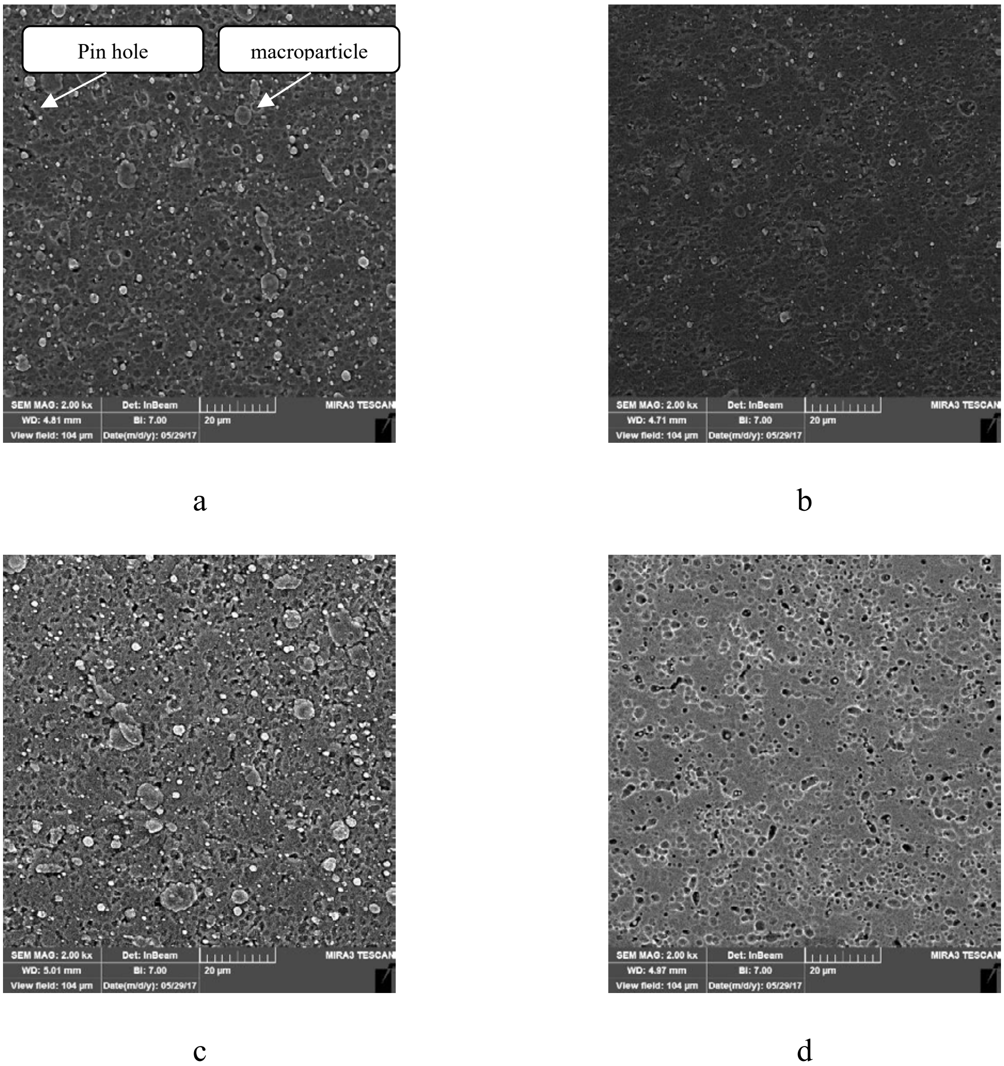


Fig. 3. Surface FESEM images of samples. a)TiN, b)CrN, c)TiN/CrN, d)Bare steel

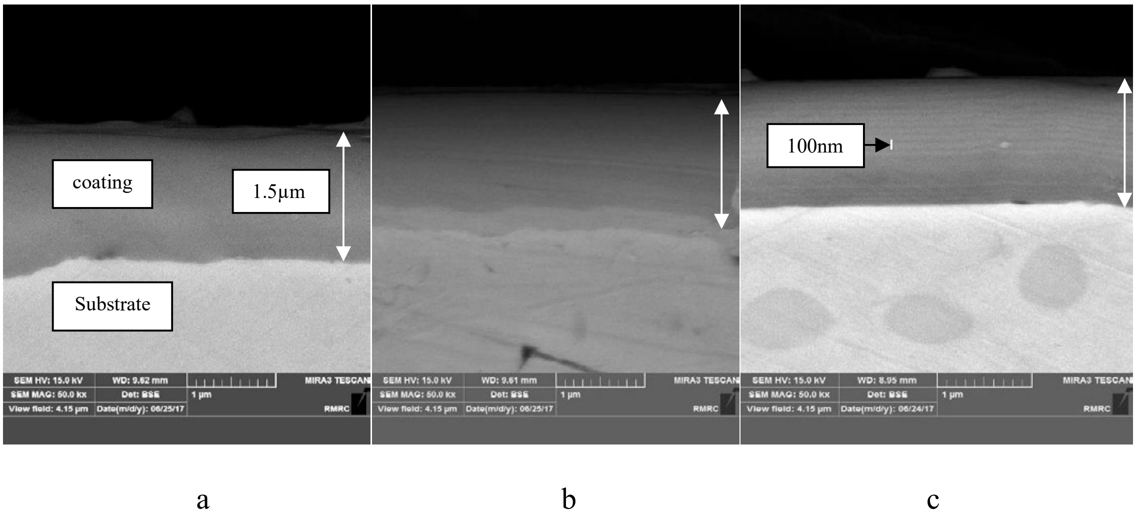


Fig. 4. FESEM cross sectional images of the samples coated in thickness of 1.5µm. a) TiN, b) CrN, c) TiN/CrN

merit, as the early roughness meters could only measure Ra. Other common parameters include Rz and Rq. Ra (average roughness) is mean value of roughness irregularities measured from a mean line within the sampling length (L). Rq is the root mean square (RMS) average of the profile heights over the evaluation length. Ten point heights of irregularities, Rz (ISO), is the average of the absolute values of the heights of five highest profile peaks and the depths of five deepest valleys within the evaluation length (Fig. 5). Equations (1), (2) and (3) show calculating formula of the values of roughness parameters [29].

$$Ra = \frac{1}{L}\int_0^L |Y(x)|\,dx \tag{1}$$

$$Rq(RMS) = \left(\sqrt{\frac{1}{L}\int_0^L Y(x)^2\,dx}\right)^{\frac{1}{2}} \tag{2}$$

$$Rz(ISO) = \frac{1}{n}\left(\sum_{i=1}^{n} pi - \sum_{i=1}^{n} vi\right) \tag{3}$$

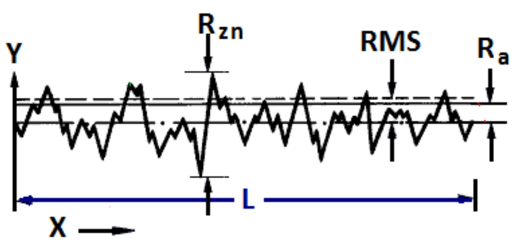


Fig. 5. Ra, Rq (RMS) and Rz parameters of roughness

Ra, Rq and Rz parameters obtained from the analysis of samples in the present paper had shown in Table 1 and their diagram was presented in Fig. 6. Ra values obtained from different surfaces of samples were in the range 0.189 to 0.423, indicating differences in the 2D surface topography. Rq increased with increasing roughness Ra in all in the range of 1.841 to 7.995. Rz values followed like all measured topographical parameters the variation in Ra in the range of 0.249 to 0.645. Based on the results can be said that coated samples have less value of surface roughness in comparison to bare steel. CrN single-layer and TiN/CrN nanoscale multi-layer have respectively the lowest and the highest value of surface roughness among the coated samples.

Contact angle measurement

Fig. 7 show water and blood contact angle images. The contact angle values are listed in Table 3. Also the comparison graph of the contact angle values for water and blood is shown in Fig. 8. Based on results, coated specimens have more water contact angle values in comparison

Table 2. Parameters of roughness for the examined sample.

Sample	Parameters of roughness[µm]		
	Ra	Rq	Rz(ISO)
TiN	0.223	0.279	1.841
CrN	0.182	0.249	2.538
TiN/CrN	0.359	0.476	3.484
Bare steel	0.423	0.645	7.995

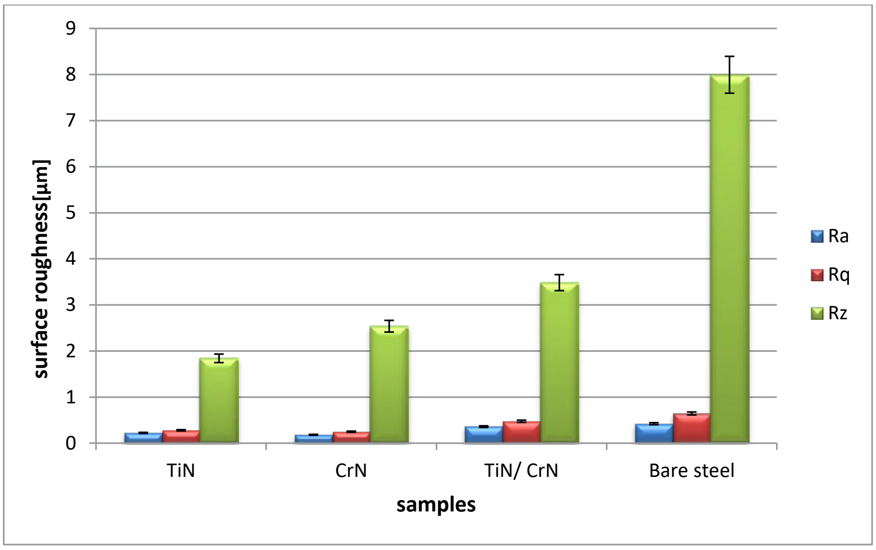


Fig. 6. Ra, Rq and Rz values for the examined samples. Error bars are standard deviations

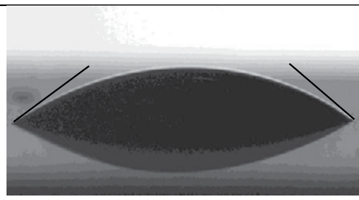
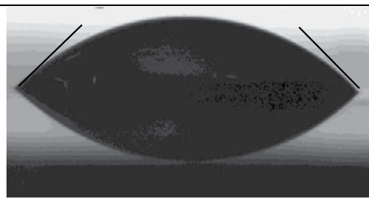
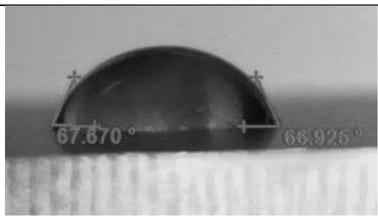
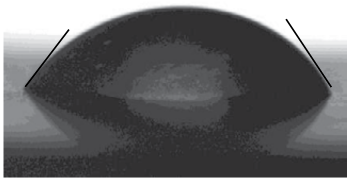
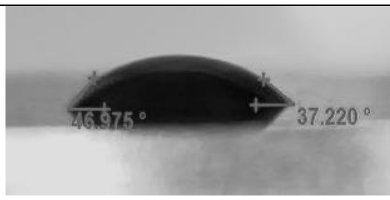
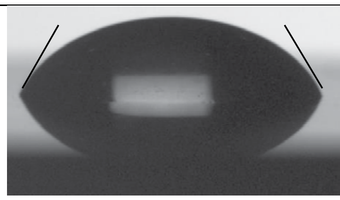
sample	water contact angle	blood contact angle
TiN		
CrN		
TiN/CrN		
Bare steel		

Fig. 7. Water contact angle and blood contact angle measurement images of all samples

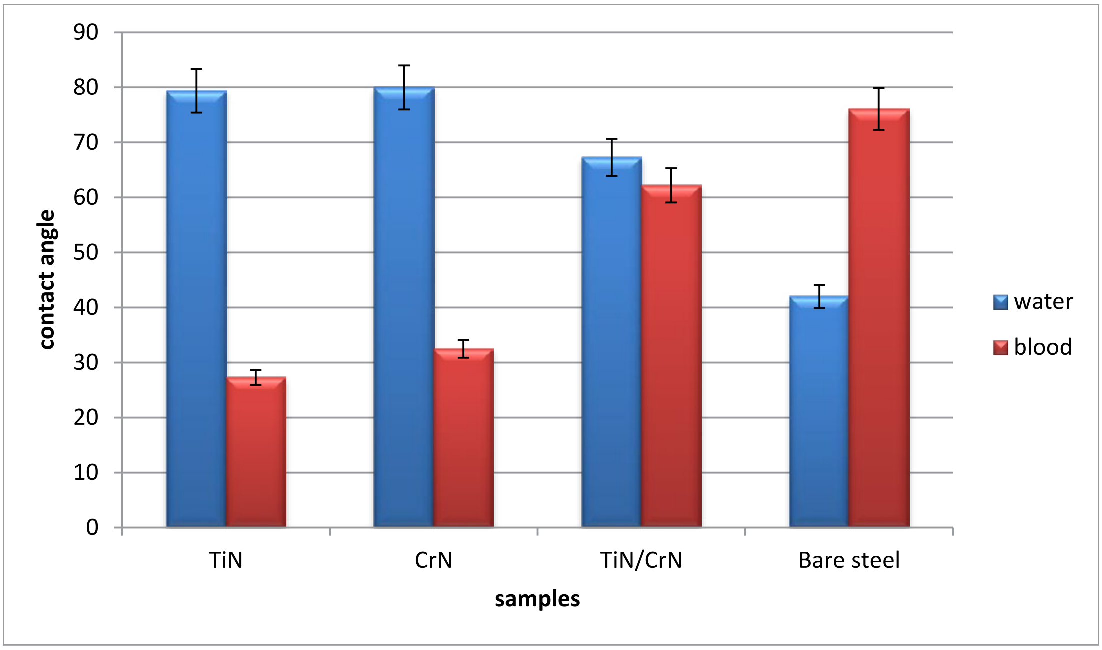


Fig. 8. Diagram of contact angle values for the examined samples

Table 3. Contact angle values for the samples

Sample	Water contact angle	Blood contact angle
TiN	79.4	27.3
CrN	80.0	32.5
TiN/CrN	67.3	62.2
Bare steel	42.0	76.1

to bare steel. Among the coated samples, CrN single-layer has the highest (80°) and TiN/CrN multi-layer has the lowest (67.3°) value of water contact angle respectively. These results are due to surface roughness of samples. With decreasing of the surface roughness, the water contact angle also increased. It should also be noted that the reduction of surface energy due to coating has taken place. Increasing the water contact angle can lead to better self-cleaning properties of the coated specimens compared to the bare steel.

On the other hand the results of contact angles of the examined samples for blood are on the contrary of water. Although coated samples have higher water contact angle values, but bare steel has higher blood contact angle value (76.1°). Among the coated samples, TiN/CrN multi-layer has higher blood contact angle value (62.2°). Blood contact angle values for TiN- and CrN single-layer coated samples are 27.3° and 32.5° respectively.

The difference in the contact angle results with water and blood is due to the nature of two fluids. Blood is a non-Newtonian fluid. A non-Newtonian fluid is a fluid that does not follow Newton's law of viscosity. Whereas water is a Newtonian fluid. On the other hand, the driving forces behind the adsorption of proteins of blood on surfaces are very complex [30]. Noncovalent forces, including hydrophobic interactions, electrostatic forces, hydrogen bonding, and van der Waals forces are effective. [31,32].

Smooth surfaces seem to experience less protein absorption than rough surfaces. This feature can be due to local accumulation of protein molecules inside the pores of the surface, which increases the adsorption of protein of blood in rough surfaces. It is generally accepted that the presence of nanoscale roughness limits the level of contact that occurs between the protein of blood and the surface [33,34].

More hydrophobic surfaces are thought to be appropriate candidates for the production of anti-biofouling materials). Nevertheless in some cases more hydrophobic surfaces can have an opposite effect on blood adhesion [35,36]. Nature of blood and its interactions with more hydrophobic surfaces is complex. The dynamic nature of both the blood cells themselves and the wetting processes can lead to a wide range of observed effects that vary with conditions. Therefore it cannot be stated firmly that more hydrophobic surfaces are useful or harmful to blood cells in general.

CONCLUSIONS

In this article water repellency and blood repellency for TiN- and CrN based nanoscale coatings deposited via CAE method on 420C stainless steel substrate were studied. The following conclusions can be drawn from this research:

1- Coated specimens have more water contact angle values in comparison to bare steel. Among the coated samples CrN single-layer has the highest water contact angle (80°). The results of this test were consistent with the results of the surface roughness test.

2- Samples had inverse behavior against blood and water in the contact angle measurement test.

3- Bare steel has higher blood contact angle (76.1°) than the coated specimens. It seems that different behavior of samples against water and blood in contact angle tests is due to the nature of two fluids. Blood is a non-Newtonian fluid whereas water is a Newtonian fluid. Also the driving forces behind the adsorption of proteins of blood on surfaces are very complex.

4- TiN/CrN multi-layer coating results had minor differences in water and blood contact angle tests (67.3° and 62.2° respectively).

Finally it was found that to get access to self-cleaning properties, material surface should have both features of water and blood repellency. This effect is a useful property to use with general surgical instruments in which the adhesion of blood should be avoided and easy cleaning with water should be done. With this view, TiN/CrN multi-layer coating is an appropriate candidate for general surgical instruments applications.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript

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