RESEARCH PAPER

Experimental Investigation of Single Shot Peening on TiN Coated Surfaces

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Abstract: *Improvement of the mechanical properties of the coated surfaces was the subject of significant research for a long time. A lot of physical and chemical operations were applied and examined on the coating surfaces successfully, but the effect of the mechanical treatments was not widely investigated. In this paper, the effect of a surface mechanical treatment on the micro coating layer has been studied and investigated. For this purpose, the necessary conditions have been created for implying impact on the coating surface. A setup of a gas gun facility with well-designed and prepared projectiles is used to strike the sample surface at different speeds. A significant number of impacts have been inflicted on the test sample of the spade drill insert cutting tool. The consequence of this process is the change in crystal structure of the coating layer, which shows that under the created conditions, the crystal structure was not destroyed, but instead compacted so that the size of the crystal grains has been reduced and considerably refined. Subsequent studies using electron microscopy have led to the measurement of the average size of the crystalline grains before and after impacts. Obviously, a significant effect has been observed, and a meaningful trend has been seen for this change in the form of a linear relationship. The main result is that about 4% reduction in the average grain size happens when the impact speed changes by 10 m/s. In this way, the principal basis for the use of this surface treatment in improving the surface properties of the micro coating layers is provided. This leads to the application of such treatments as an industry process for the improvement of thin coating mechanical properties.*

Keywords: *Surface mechanical treatment, Titanium-based coatings, Grain refinement, Improving coating surface properties, Mechanical impact treatments, Crystal grain size.*

1. INTRODUCTION

Surface treatments for increasing the surface hardness of cutting tools and thus increasing the capabilities of wear resistance and self-lubrication performance have been recently considered. The shot peening method is one of the most effective methods for increasing the strength of surfaces and is widely used for this purpose in various industries [1]. In this process, small metal balls with high hardness and a speed of 20 to 100 m/s are shot at the surface, creating a plastic deformation zone below the surface. Consequently, a compressive stress field is created, which greatly contributes to the anti-fatigue, anti-abrasion, and load-carrying abilities [2]. The shot peening process can be considered as a surface treatment with the ability to create plastic deformations to refine the grains of material in a thin layer of the surface in the range of 40 to 100 nm and thus increase the surface hardness [3].

The shot peening process has always been used to improve the fatigue properties of material surfaces. This is achieved by preventing cracks from initiation or reducing their propagation rate. There has been a lot of research on the shot peening process on pure materials (bulk), including titanium contents. For example, Kato et al. (1999) investigated the effect of this process on titanium-based alloys. For this purpose, several standard fatigue test samples made of Ti-6Al-4V have been prepared at 20 to 450 degrees Celsius and tested in a bending fatigue test device (rotary). The results showed an increase in fatigue resistance at high temperatures for shot-peened surfaces [4].

Because of the special importance of the titanium alloys, the same investigation has been continued by other researchers. Ludian et al. (2008) have compared the effect of shot peening and burnish methods for two types of bulk titanium alloys: the Ti-2.5Cu class and the low-stability Beta C class [5]. They observed that increase in the hardness of the surface layer is caused by the formation of high-density dislocations [6]. In the work of Bae et al. (2008) [7] a comparison of behavior was made for two types of titanium alloys, and in the

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research of Mawad et al. (2008), the same procedure was repeated for a wide variety of materials. The results show that fatigue resistance with high cycles is affected by residual tensile stresses, and especially the amount of mean stress and environmental conditions have more importance. The resulting stress gradient at the surface improved the fatigue performance of the high-cycle by reducing the critical stress in the subsurface material and preventing crack formation [8].

The same methodology has been followed in other similar works. karaoglanli (2004) [9] exposed grade 2 commercially pure titanium to air flow, shot peening, and investigated its effect on abrasion resistance. The samples were exposed to plastic deformations with different time rates caused by severe conditions of shot peening. Consequently, nanoindentation testing was performed to identify the distribution and type of residual stresses, with which the coarse-grained crystal structure in the transition zone can be identified. Optical microscopy and electron scanning microscopy (SEM) have been used to analyze the wear effects and identify the heavily deformed layer. As an important result, it can be said that surface hardness and resistance against abrasion have increased when more severe impacts are implied.

The obvious result of reviewing the process of previous research on shot peening is that performing mechanical surface treatments, especially the most used shot peening on surfaces before or after coating, has rarely been investigated and studied. Therefore, performing any of the surface treatments on the coated objects at a high rate can be the main approach in the present study.

According to the study of previous research, all of which show that mechanical treatment methods are mainly performed on uncoated materials, and it is generally believed that mechanical treatments, especially impact on coated surfaces, destroy these surfaces. Therefore, very little work has been done regarding surface treatment on coated materials. In the studies conducted by the authors, it has been proven for the first time that hard coatings have the ability to withstand impacts without causing damage to the crystal structure of the coating. Then, a similar idea is presented that applying impact to surface coatings can produce the same desired results of this process for uniform materials (uncoated). To investigate this hypothesis, very intensive experiments have been performed to prove that the crystal of the coated surfaces is not destroyed by impact, and then the impact effect on the crystal structure of the coating surface has been investigated. The results of this research, which are presented and validated for the first time, are introduced in the present paper.

2. EXPERIMENTAL PROCEDURES

2.1. Surface Mechanical Treatments

The main result of reviewing previous research on the mechanical treatment processes, especially shot peening (i.e., impact on the surface) is that such treatments before or after coating have rarely been investigated and discussed, and instead, non-mechanical operations such as heat treatment and changes of alloy material composition have been more investigated. Therefore, performing any of the surface treatments on the coated objects that can be described as a high-rate process can be one of the new approaches in this field.

In this case, some of the works, such as the research of Komarov et al. (2016), can be referred to. In which surface hardening by heat treatment method with carbonation process has been considered. By performing this operation, the properties of microhardness and wear resistance have been significantly improved. For TiAlN and TiN coatings, the hardness can be increased by 7 times, wear resistance by 2.3 times, and toughness by 4.5 times. This increases the amount of toughness and adhesion and creates anti-cracking properties. The fracture mode in the coatings is mainly brittle, which is accompanied by the formation of spiral or circular cracks, while the surface shows more plastic behavior before heat treatment. This leads to the formation of a controlled infiltration layer between the underlying layer and the top [10].

To improve the anti-wear properties, laser surface treatment has also been used on the substrate material. First, the surface of the substrate material is bombarded with a laser beam and then coated. Kedong et al. (2016) [11] bombarded the base material with an Nd:YAG laser beam and then coated it with TiAlN alloy. The results show that the anti-abrasion properties caused by the adhesion between the non-similar materials have made great progress. The speed of laser scanning has a great impact on the creation of adhesion strength. Therefore, the material's potential anti-abrasive properties are improved by doing the same process. The mentioned researchers continued their investigation to improve the lubrication properties of titanium

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aluminum nitride coatings by examining the effect of laser grooving on the surface of the underlying material before the coating process. For this purpose, two types of laser beams at the nano scale and micro scale have been utilized in the previous method. MoS_2 powder is used to polish the surface of the underlying material before being coated with TiAlN. The results of the measurement of lubrication properties showed that creating grooves with a laser beam on the surface of the substrate material and adding MoS_2 have considerably improved this property. Laser grooving at the nano surface improved the abrasion resistance but did not have much effect on the friction coefficient. The addition of molybdenum sulfate powder significantly reduces the coefficient of friction, and micro-scale grooving preserves this material for a longer period of time [11].

2.2. Single Shot Peening

In order to test the effect of mechanical treatment on the surface of coated material, we chose the single-shot process. This type of treatment allows us to evaluate the consequences of the impact without interruption by other factors, such as the interference of multiple impacts. To check the impact of a single shot, it is necessary to apply a full, controlled, and concentrated impact. In order to control the impact point and obtain well-defined conditions, the method of applying impact with a single projectile has been used. This concept (single-shot process) was used for other purposes previously. So, it is discussed and analyzed in detail by the aid of FEM by other researchers [12]. A gas gun facility is used to perform surface operations by applying impact to the surface of a specified object. According to Fig. 1, the device consists of a compressed gas reservoir, a straight pipe, and a velocity measuring instrument. After placing the projectile in the pipe head, the pressure gas valve suddenly opens, and gas under high pressure pushes the projectile through the straight pipe and throws it at high speed from the end of the gun. The laser beams of the velocity measurement section then come into operation as the projectile passes through them and measures the projectile's velocity immediately before hitting the sample surface. The operation basis of the laser velocity measurement section is that two laser beams with a fixed distance are headed towards the optical sensors on the opposite side. The projectile cuts off these laser beams subsequently on its path, and the interruption pulse is sent by the sensors to the data processing instrument. By measuring the time between two pulses, the speed can be obtained by dividing the distance between two sensors by the measured time. This calculation is done by a data acquisition calculator in the measuring device [10].

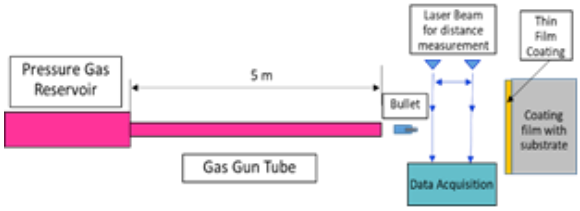

Fig. 1. Schematic view of a gas gun facility used in impact testing

To adjust the projectile speed, the pressure of the actuator gas can be changed. The more precise the pressure adjustment, the more accurately the output speed can be controlled. In the device used in the current experiments, which is locally made in the Impact Laboratory of K.N. Toosi University of Technology, the accuracy of gas pressure control is about 0.01 bar. Since the targeting accuracy by this device is limited, the distance between the opening of the gas gun barrel and the target material should be as small as possible (about 20 cm) to increase the accuracy of hitting the desired point. This has created a serious limitation in the performance of laser speed sensors that are more than 20 centimeters away from each other. To solve this problem, it is necessary to make determination throws to adjust the point of impact on the target as well as to determine the exact speed. Therefore, the test process is set up in such a way that at a certain pressure, multiple impacts are made and a stable velocity is measured, then with the same gas pressure, the sample is impacted. This has led to a significant increase in the number of shots, so that for almost every 10 shots given, there is a single precise and controlled shot at the target.
The dimensions of the projectile have been chosen as small as possible to allow the impact effect to be closer to the small scales (micro or nano). This allows subsequent analytical simulations to be performed using a smaller number of elements. For this purpose, a gas launcher tube with the smallest possible diameter has been used. Here, a pipe with a smallest diameter of 22 mm, which allows a precise alignment of 2.5 meters in length, was selected. According to this diameter, a bullet made of Ertalon is designed to carry a metal

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projectile head. The schematic shape of this bullet and the final projectile form with the necessary dimensions is shown in Fig. 2a. The most important point in achieving the speed and accuracy of the projectile is to strictly keep a close tolerance between the outer diameter of the projectile and the inner diameter of the gas gun barrel, which should lie in the range of 0.3-0.4 mm tolerance.
First, a metal pin was used to apply the impact to the target surface. Although it was initially thought that the use of a metal pin would create a smooth surface that would be more suitable for any kind of measurement, the results have shown the opposite. It has been observed that the end of the pin would not hit the surface in an exact perpendicular direction. Thus, it has been proven that the use of a spherical metal projectile creates a better controlled, concentrated, and precise footprint. Therefore, according to Figs. 2b,c, and d, other types of projectiles have been designed and built. In the second type of bullet, a metal ball is used to apply the impact, and a pin on the opposite side is used to maintain the weight balance. In the third and fourth types of projectiles, the balancing pin has been eliminated due to its effect on deforming the shape of the bullet body, and only the metal ball has been kept. The actual image of this type of projectile is shown in Fig. c. The dimensions and mass of all the projectiles used in the impact test procedure are mentioned in Table 1. It is clear that the specification of all the projectiles is almost the same, except the pin head projectile, which is used only for evaluation and primary check of the procedure. Some of the projectiles with high deviation from the common specification are neglected.

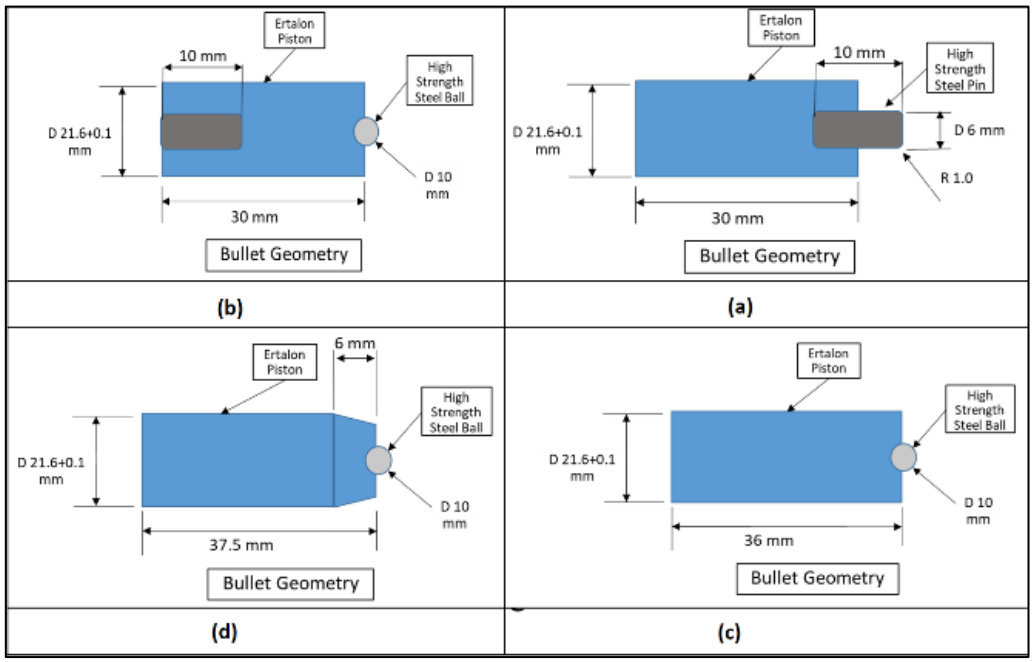

Fig. 2. Drawing of the 4 types of bullets used to hit the target object in the Gas Gan Facility

Table 1. Specifications of the projectiles and bullet heads used in impact test procedure

Impact No.	Striker Type	Striker Dia. (mm)	Projectile Mass (gr)	Target Material
Test 1	Type 1	6 (pin)	12.3	TiN Disc (Sample 10)
Test 2	Type 2	9.99 (ball)	16.4	TiN Disc (Sample 10)
0	Type 3	9.99 (ball)	16.42	TiN Spade Drill
1	Type 4	9.99 (ball)	16.42	TiN Spade Drill
2	Type 4	9.98 (ball)	16.48	TiN Spade Drill
3	Type 4	9.99 (ball)	16.52	TiN Spade Drill
4	Type 4	10 (ball)	16.5	TiN Spade Drill
5	Type 4	10 (ball)	16.41	TiN Spade Drill
6	Type 4	9.84 (ball)	16.29	TiN Spade Drill
7	Type 4	10.28 (ball)	16.82	TiN Spade Drill

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Fig. 3. Photo of the final shape of the bullet used to hit the target object in the gas gun facility

2.3. Impact Test

Impact testing on different types and aims has been done on a wide variety of materials in the past, but the important point that should be noted is that impact testing and its effect on coated materials do not have a long history. Therefore, in the present article, a special focus has been made on this regard. To do this, a sample of a cutting tool that has a smooth and relatively large surface suitable for applying impact has been used. Similar samples of such tools are widely used in spade drills, such as in Fig. 4. The drill used in the present experiment has a high-speed steel substrate that is coated by a layer of titanium nitride (TiN) deposited by chemical vapor deposition (CVD) method. In order to study the effect of high-velocity impacts on the tool surface, the experiments were designed in such a way that the impact would hit the lateral surface of the spade drill. For this purpose, the sample is installed vertically on the side face of the device fixture in such a way that the footprints of the successive impacts are located sufficiently far from each other.

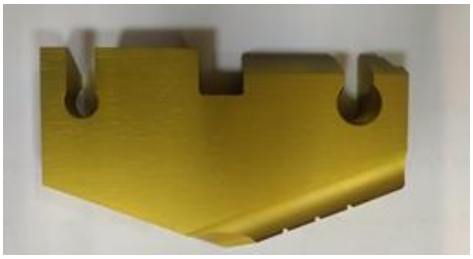

Fig. 4. Spade drill coated by a thin layer of Titanium nitride

In order to adjust the test setup, the location of the projectile hit point on the fixture of the device should first be determined and marked. This is done by shooting the fixture face before installation of the spade drill. The hit point location is marked on the fixture side face in Fig. 5. The spade drill tool sample is then fixed and stuck to the side face of the fixture after adjusting the specified hitting position (Fig. 6).

Fig. 5. View of the holding fixture in the impact test facility and how to mark the hit point before installation of the sample

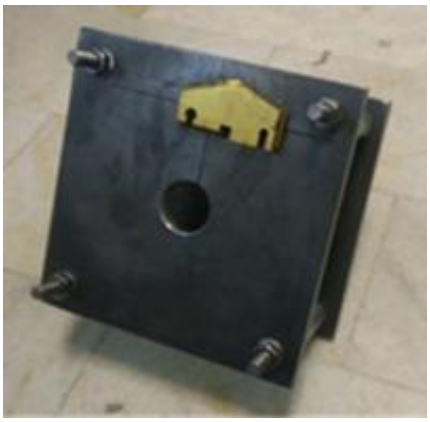

Fig. 6. View of the holding fixture of the sample in the impact test facility, and how to fix the sample piece on the fixture

After all the above preparations, the testing facility is ready for implementing the speed determination impacts. The result is the determination of the projectile velocity in a constant gas pressure. Firstly, to evaluate the impacted surface quality and impact accuracy, a sample coated with the physical vapor deposition method (PVD base sample) was used (Fig. 7 left).

To start the test, the projectile of the first type and the part of the base sample have been selected according to Fig. 7. The result of the impact test indicates that due to the microstructural effect of the impact, the refinement of the steel pin surface and orthogonality on the target object have a great effect on the quality of the surface after impact. Evaluation of the pin surface and impact point by using Optic microscope is shown in Fig. 8. The most important conclusion is that no matter how much careful you are in making the pin, eventually due to the deviation of the bullet while exiting the

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gas gun barrel, the orthogonality with respect to the target surface will be lost, and the impact will occur through one corner of the steel bullet. Fig. 8 clearly illustrates this situation. Also, in this figure, the roughness of the surface resulting from the grinding lines of the pin head is also well observed on the surface of the target.

Fig. 7. Type I projectile with pin-shaped steel bullet (right) and TiN-coated steel specimen (left)

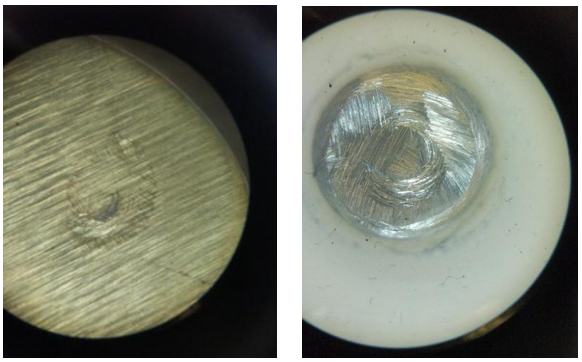

Fig. 8. Optic Microscopic image of projectile pin surface (right) and projectile pin impact point on target specimen surface (left)

3. RESULTS AND DISCUSSION

During the test program, a total of 120 shots were performed, of which the selected 8 shots conformed to the sample necessary conditions, so that the results were acceptable for further conclusions. The specifications of the aforementioned shots are listed in Table 2. Three types of bullets were used to inflict these impact shots. The difference between the bullets with each other has generally been in the mechanical properties of the metal balls, including strength and hardening treatments. The material of the steel balls is selected from the steel type 100Cr6, which is suitable for use in bearing balls. The material composition is according to the ISO 683-17 standard [13]. So, the hardness of the balls, which is important in the evaluation of the impact effect, was 60HRC. To achieve uniform test results, the balls are selected with the same characteristics. The Ertalon 4.6 used for the projectile body has proper flexibility and weaker mechanical properties than metal materials.

The deformation of the steel balls was highly variable in proportion to the hardness and speed of the impact. In Strike No. 2, in which a soft ball is used, the deformation of the ball surface was very high and even led to the destruction of the ball. Therefore, in order to prevent the effect of damage at the impact point, it was decided that this type of soft ball should not be used. Using semi-hard balls, it has been observed that the pellet has been deformed to a very large amount but has had little effect on the surface of the sample, the rest of the bullets have been made with hard balls.

One of the important measures to evaluate the impact is the momentum of the impact. It can be measured by $\vec{p} = mv$ which is the multiplication of the mass by the speed of the projectile. The mass includes all the elements of the projectile, including the pellet and the bullet. The results are mentioned in Table 2.

Table 2. Specifications of impacts applied to coated sample with various types of striker (projectile) and gas pressure

Impact No.	Striker Type	Gas pressure (bar)	Impact speed (m/s)	Impact energy (J)
Test 1	Type 1	0.8	30	0.369
Test 2	Type 2	0.8	30	0.492
0	Type 3	1	40	0.6568
1	Type 4	2	78	1.28076
2	Type 4	2.5	88.7	1.461776
3	Type 4	2	78	1.28856
4	Type 4	3	98	1.617
5	Type 4	3	98	1.60818
6	Type 4	4	117	1.90593
7	Type 4	3.5	106	1.78292

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In order to investigate the structure of surface metallography before and after impact, the SEM scanning microscope model AIS-2100 SERON Technology located in the electron microscopy laboratory of Amirkabir University of Technology was used. High precision and clear photography were the main advantages of this facility. The maximum magnification of this microscope is maximum 25,000 times, which is completely suitable for studying the crystal structures.

In the first practical impact, which is marked by the number zero in Table 2, the resulting impact effect was very small, so that it was almost imperceptible even by high magnification of optical microscopic examination. This matter happened due to the fact that the impact velocity was about 40 m/s. In Fig. 9a, the impact surface image of the coated steel specimen (Base sample of disc type Fig. 7) is illustrated with an optical microscope. From this case, it can be concluded that due to the very high level of coating surface hardness, much higher speed impacts are required. Therefore, in the subsequent impacts, the velocity has been significantly increased to the range of 78-117 m/s. In Figs. 9b,c, and d, the effects of successive impacts with higher speeds on the spade drill coating surface can be seen. With these speed levels, it can be said that the collision can be considered as a high-rate impact. After cleaning the spade drill sample, it should be stuck and fixed on the holder of the device to be ready for scanning with electron beams. The pressure of the electron microscope medium should be reduced to 4-10 Torr to create a relatively high vacuum environment. The voltage difference of the device should be such that it does not cause electrons to penetrate deep into the part material, and can demonstrate the surface information in a better way. For this purpose, the device voltage is adjusted in the range of 15-20 kV. The result of the electron scanning of the surface of the initial sample before impact is shown in Figs. 10 & 11 with different magnifications.

In the continuation of the sample testing of the microscopic material structure, scanning and imaging of the surface of the impact-hitting location caused by subsequent impacts have been performed. The results are shown in Figs. 12-16 for consequent impacts footprints. As can be seen from the study of the images in the locations where different impacts are implied, the severe impacts on the surface of the coated part at relatively higher speeds do not cause any structural destruction, such as cracks, delamination, and surface defects. Thus, the primary hypothesis that impacting or hammering can be used as a surface treatment on the coated materials is supported. Thus, the authors paved the way for deeper research and concluded that further tests can be conducted to prove this idea. The next step is to prove that the impact treatment can be used for the improvement of surface characteristics.

Investigation of crystal structure images of the coating surface clearly shows that the impact has a considerable effect on the size of the grains, so that the size of the grains has been significantly reduced, and thus a more compact crystal structure has been created.

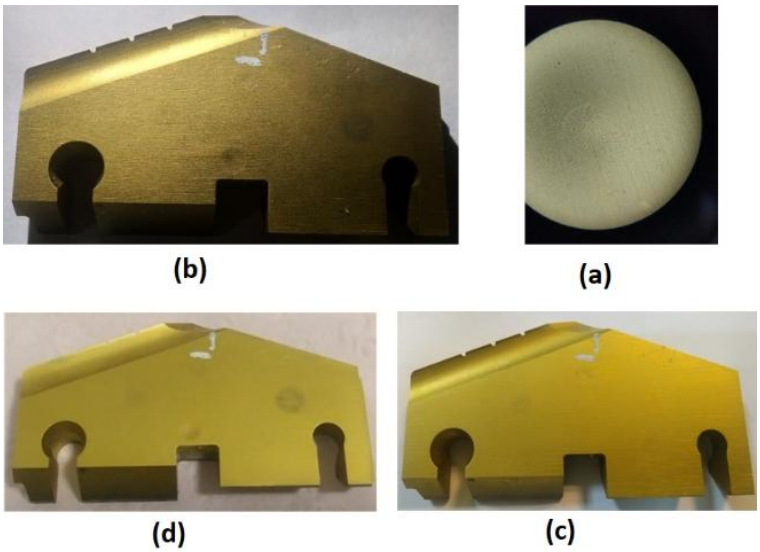

Fig. 9. Microscopic image of impact effect on coating surface in different speeds: a) at 40 m/s on steel coated specimen (disc type), b) at 106 m/s, c) at 98 m/s and d) at 117 m/s on spade drill

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Generally, size reduction means some effects that cause compacting and splitting of the crystalline grains. But this does not necessarily mean damage in the crystalline structure, but may mean splitting some microstructures from the bigger grains, which makes the grain smaller. One of the main aims of SEM microscopy was to show the effect of the impact on the surface coating structure. The images don't show any sign of delamination but show strong evidence of size reduction. This result was supported by comparing the microscopic photos before and after the impacts.

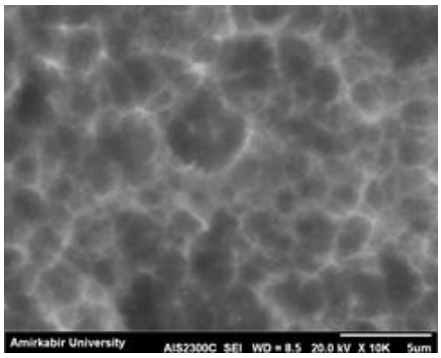

Fig. 10. SEM Image of TiN Coated Surface Spade Drill Before Impact with 10000 magnification

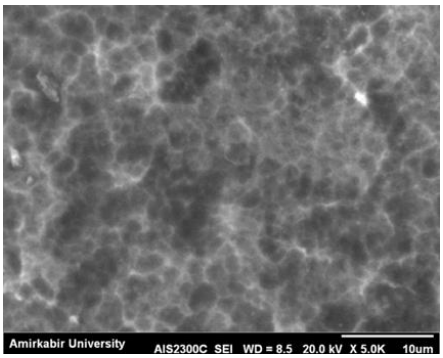

Fig. 11. SEM Image of TiN Coated Surface Spade Drill Before Impact with 5000 magnification

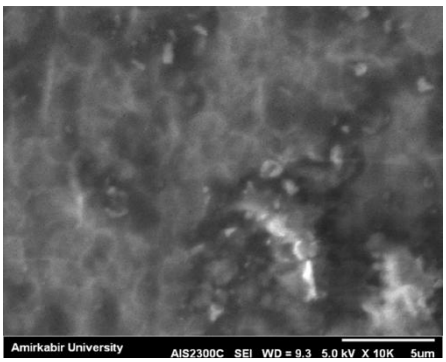

Fig. 12. SEM image of TiN-coated Spade Drill surface for impact No. 1 with magnification of 10000

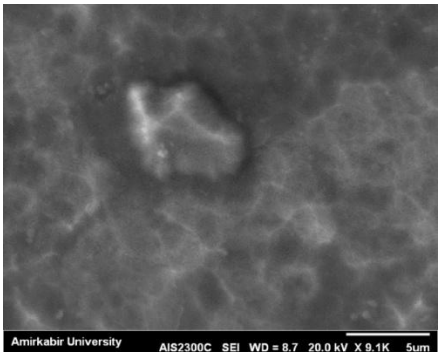

Fig. 13. SEM image of TiN-coated Spade Drill surface for impact No. 2 with magnification of 9100

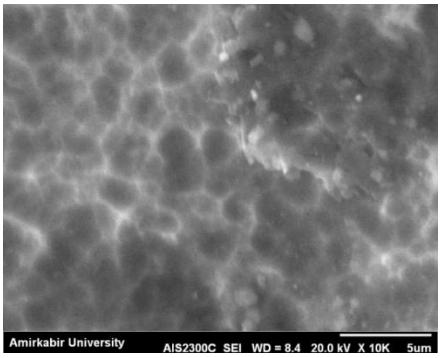

Fig. 14. SEM image of TiN-coated Spade Drill surface for impact No. 5 with magnification of 10000

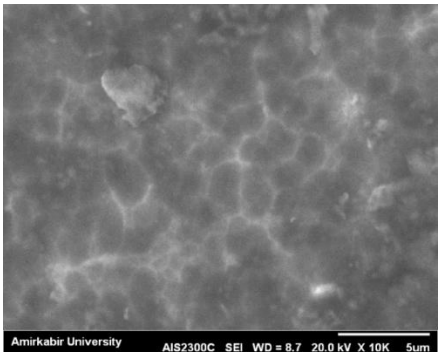

Fig. 15. SEM image of TiN-coated Spade Drill surface for impact No. 6 with magnification of 10000

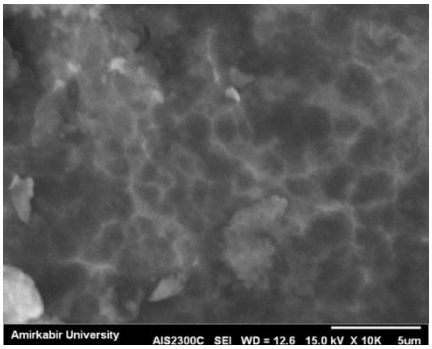

Fig. 16. SEM image of TiN coated Spade Drill surface for impact No. 7 with magnification of 10000

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To allow a quantitative study on this result, the Heyn method can be used to measure the size of the crystal grains by using a microscopic image. This method is recommended for materials that do not have uniform and unique dimensions for the crystal grains. In this method, which is also known as the intersection method (or intercept method), the intersection of lines with a certain length to the boundary of the grains is counted, and finally yields the average size of the crystal grains [14].
In Fig. 17, an example of measuring the average size of the crystalline grain of the coated surface before impact is shown. Considering that the counting of the intersection points of grains with straight lines should be done in at least 3 places, here it has been done in 6 places to achieve higher accuracy. In the next step, the Heyn method was applied for the measurement of the average grain size in the footprint of the impacts at all speeds. In the diagram of Fig. 18a, the results of measuring the average grain size are plotted in terms of impact speeds. Also, this diagram can be drawn for the impact momentum to take in consideration both the speed and mass effects at the same time. It is foreseen that more accurate results can be concluded. The results are mentioned in the diagram of Fig. 18b. A similar trend of variations is observed compared with Fig. 18a.
Evaluation of the trend of these diagrams shows that there is a direct relationship between the impact speed rate and the refinement of the crystal grains. The higher the impact speed rate, the higher the refinement effect on the grains, which has a direct effect on the increase in the hardness of the sample surface. This can lead to an improvement in the mechanical properties of the material. Unal et al., through a detailed study of titanium samples, proved that surface treatment can lead to an observable increase in the modulus of elasticity [15]. Also, Hall and Screw proved the relationship between yield stress and average size of crystalline grains at ambient temperature, and also described the relationship between fracture due to rupture and this parameter [16].

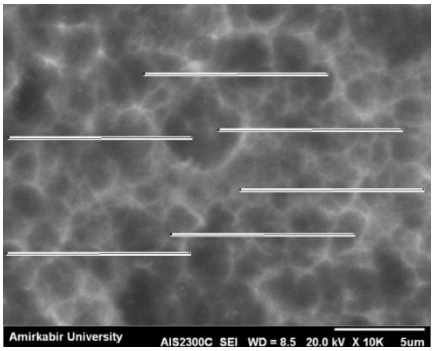

Fig. 17. Using the Heyn Method to calculate the average grain size of the crystal structure of the TiN coating before impact

The study of the diagram in Fig. 18 shows that the results are significantly interrelated to each other, and even by using a linear equation, it is possible to provide a specific formula to describe them. The following equation can be presented for this purpose:

$GS = Av + B$

where GS denotes the size of the crystalline grain and v demonstrates the velocity of the impact. Here, the coefficients A and B depend on the type of bullet, the diameter of the bullet, the hardness of the bullet, and other sample test conditions. In the present specific case, based on the conditions of the test and specimen, these constants have the following values:

$A = -4.7 \times 10^{-4}$

$B = 1.9157$

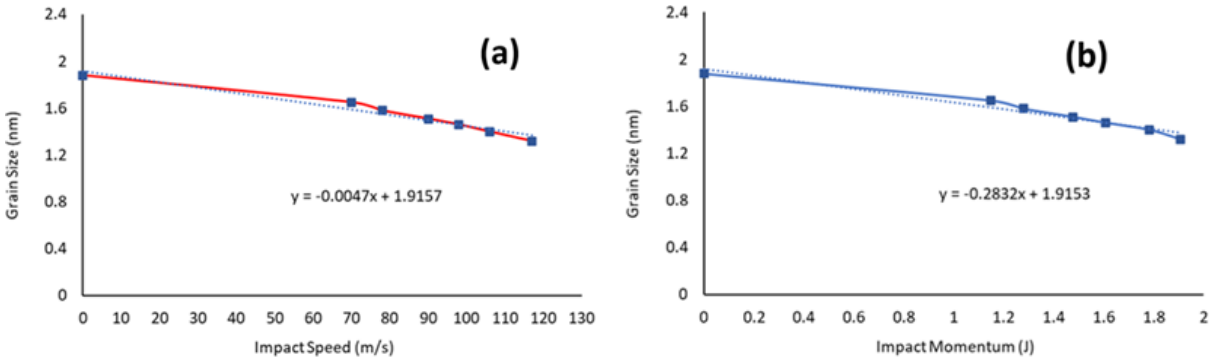

Fig. 18. Diagram of the crystalline grains' average size changes of the coating surface in terms of a) impact speed and b) momentum of the impact for TiN coated surface

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The trend of changes in the average size of the crystalline grain indicates that each increase in the impact velocity by 10 m/s, cause decrease of the average size of the crystal grains by approximately 4%. Definitely, it should be noted that the changes in grain size start from a minimum limit speed and continue to increase until the maximum limit speed.

4. CONCLUSIONS

Contrary to the popular belief that impact on the surface of the micron coatings causes destruction and even its removal, here it is possible to inflict impact without causing damage to the crystal structure. This is proved by microscopic investigations using a SEM (scanning electron microscope) in controlled conditions. For this purpose, a spade drill cutting tool with a suitable coated surface and proper material has been used. After preparing the necessary conditions for applying precise impacts to the sample surface, a significant number of impacts were exerted on the surface of a sample coated with titanium nitride. Consequently, the necessary measurements were performed with the aid of stochastic approximations. The results indicate that the average size of the crystalline grains decreased due to impact. The measurements showed that by increasing the impact velocity in mechanical treatments, the average grain size decreased, which follows an inverse and almost linear relationship. The trend of changes is such that by increasing the velocity of the projectile by 10 m/s, the average crystal grain size decreases by 4%, which is a significant increase. Taking into account the relationship between the average grain size and modulus of elasticity, yield stress, and rupture criterion, this change in the characteristics of the coating layer can be considered very important.
As a future vision of the present research, it can be mentioned that further investigation is necessary to customize this method to make it useful for industrial applications. Since the impact made by the ball striker can only make local and non-uniform footprints, a method of impacting should be developed to make uniform refinement rather than local refinement in the grain size on the entire sample surface.

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