p-ISSN: 1410-6957, e-ISSN: 2503-5029
https://jurnal.batan.go.id/index.php/ganendra

THE EFFECT OF THE DC-SPUTTERING PROCESS ON CHANGES IN THE HARDNESS VALUE AND ELEMENTS COMPOSITION OF BIOCOMPATIBLE STAINLESS STEEL 316L MATERIAL

PENGARUH PROSES DC-SPUTTERING PADA PERUBAHAN NILAI KEKERASAN DAN KOMPOSISI UNSUR MATERIAL BIOKOMPATIBEL STAINLESS STEEL 316L

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Received 17 March 2023, revised date 15 May 2023, accepted date 4 July 2023

ABSTRACT

THE EFFECT OF THE DC-SPUTTERING PROCESS ON CHANGES IN THE HARDNESS VALUE AND ELEMENTS COMPOSITION OF BIOCOMPATIBLE 316L STAINLESS STEEL MATERIAL. *Titanium Dioxide (TiO_2) thin films have intriguing optical, photocatalytic, and electrical properties and have been investigated for various applications, including solar cells, biomaterials, corrosion-resistant materials, and gas sensor. In this study, TiO_2 thin films were deposited on the surface of 316L Stainless Steel to improve its mechanical properties as an implant material. The deposition method used was DC sputtering with variations in deposition times of 30, 60, 90, 120, and 150 minutes. Vickers hardness test and SEM-EDX characterization were carried out to determine the hardness value, elemental composition, and thickness of the TiO_2 thin film formed. Based on these tests, it was discovered that the optimal hardness value of 316L stainless Steel material was attained at a deposition period of 90 minutes with a hardness value of 170.10 VHN, and the average thickness of the layer formed was ± 119.02 µm.*

Keywords*: 316L stainless Steel, DC sputtering, titanium dioxide*

ABSTRAK

PENGARUH PROSES DC-SPUTTERING PADA PERUBAHAN STRUKTUR MIKRO DAN KOMPOSISI UNSUR MATERIAL BIOKOMPATIBEL STAINLESS STEEL 316L. *Titanium Dioksida (TiO_2) dalam bentuk film tipis memiliki sifat optic, fotokatalik, dan elektronik yang menarik dan telah dipelajari untuk banyak aplikasi, termasuk sel surya, biomaterial, material tahan korosi, dan sensor gas. Pada penelitian ini, film tipis TiO_2 dideposisikan pada permukaan 316L stainless steel dengan tujuan untuk meningkatkan sifat mekanik dari material 316L stainless steel sebagai material implan. Metode deposisi yang digunakan adalah DC sputtering dengan variasi waktu deposisi 30, 60, 90, 120 dan 150 menit. Dilakukan pengujian kekerasan vickers dan SEM-EDX untuk mengetahui nilai kekerasan, komposisi unsur dan ketebalan film tipis TiO_2 yang terbentuk. Berdasarkan pengujian tersebut didapatkan bahwa nilai kekerasan optimum material 316L stainless steel diperoleh pada lama waktu deposisi 90 menit dengan nilai kekerasan 170,10 VHN dan ketebalan rata-rata lapisan yang terbentuk yaitu sebesar ± 119,02 µm.*

Kata kunci*: 316L stainless steel , DC sputtering, titanium dioksida*

INTRODUCTION

The development of science and technology related to materials occurs very rapidly, and one of its applications is biomaterial. Biomaterials are synthetic or natural substances suitable for direct interaction with biological systems [1]. In medical settings, biomaterials are used in various ways, including medical implants as a substitute for tissue damaged by disease or trauma, as a filler, and to support the healing phase. Under ideal conditions, biomaterials have good biocompatibility, mechanical properties, and easy manufacturing processes [2], [3]. Biocompatibility is a system including physical, chemical, biological, medical, and design aspects and can be defined as an acceptable material function without causing unwanted reactions at the tissue level or the immune system. Some mechanical properties required in biomaterials are ductility, toughness, creep, and wear resistance that imitates the mechanical behavior of the biological tissue that it replaces [4].

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Not all available metals can be used as implant materials because they do not meet biocompatible requirements. Stainless steel, cobalt alloys, and titanium alloys are some of the most commonly used metals due to their biocompatible properties [5]. Stainless steel 316L is one of the most commonly used stainless steel as an implant material. The 316L stainless steel material has several advantages, including high strength, flexibility, toughness, and corrosion resistance. Therefore, 316L stainless steel can be used in various applications, such as implant materials, biomedical and petrochemical, automotive, and chemical engineering [6]. Besides that, the availability of 316L stainless steel material is easy to obtain and has low production cost [7].

Wear resistance is another important requirement for biomaterials. Because of the complex and corrosive environment of the human body, the 316L stainless steel material is not resistant to wear and tear when used for an extended period. Wearing materials causes metal ions to be released into the body, causing toxicity. To overcome these problems, surface treatment is required by depositing a strong layer on the surface of the biomaterial 316L stainless steel [8]. Other than high wear resistance, surface treatment is also generally applied to specific conditions where good toughness is required. These two properties are opposites because ductility and toughness will decrease if hardness and strength increase. As an implant material, the material is made with a hard outside and the inside remains ductile [9].

Titanium dioxide (TiO_2) combines excellent physical, chemical, mechanical, and biomedical properties. TiO_2 materials in thin films has good biocompatibility and the potential to reduce friction and wear resistance, making it suitable for human implants and medical devices [10]. A study in 2017 done by Yuzhen Wang and his colleagues showed that a 0.2 mm thick TiO_2 thin film deposited on the surface of 316L stainless steel did not crack or degrade after passing corrosion tests [11]. The growth of thin films with surface treatment effectively increases the mechanical strength of 316L stainless steel material [12].

There are several methods of depositing a thin layer on the surface of the material, including chemical vapor deposition (CVD), physical vapor deposition (PVD), chemical deposition, electrochemical deposition, laser deposition, thermal spraying, and sol-gel methods, etc. Sputtering is the most commonly used method in the PVD process, and one of the sputtering methods is Direct Current (DC) sputtering [13]. This method is good for synthesizing thin films on a substrate by breaking the bonds between atoms on the target so that the atoms will arrange on the substrate and form a new thin layer [14]. DC sputtering is suitable for conductive targets [15]. The advantage of sputtering over other methods is that the process is faster and cleaner as performed in a vacuum. Thin layers of materials with high melting points can be produced; almost all solid materials, such as semiconductors, metals, alloys, and ceramics can be deposited, and have stronger adhesion to extend the life of the manufactured parts [16]. In addition, it is easy to control the deposition parameters and reproduce the deposition conditions [17].

In this study, surface treatment will be carried out by depositing a thin layer of TiO_2 film on the surface of 316L stainless steel material using the DC sputtering method to improve the mechanical properties, such as the hardness value of 316L stainless steel material. SEM characterization and Vickers hardness test will then be carried out to confirm the formation of a thin layer on the surface of 316L stainless steel material and determine its hardness value after the surface treatment.

METHODOLOGY

Materials

The materials used in this study were 316L stainless steel substrates, sandpaper with sizes 100, 200, 400, 800, 1000, 1500 and 2000 mesh, autosol, velvet cloth, alcohol, nitrogen (N_2) and argon (Ar) gases, titanium target, detergents, plastics, and tissues.

Tools

The equipment used in this research was a grinding and polishing machine, an ultrasonic cleaner machine, an hairdryer, a Vickers hardness tester, a microscope, a DC sputtering machine, and a SEM-EDX test kit.

Experiment

The material used in this study was 316L stainless steel with a diameter of 14 mm and a thickness of 5 mm. Before the deposition and testing process, the specimen must first go through a grinding and polishing process to produce a smooth, flat, clean material surface free of dirt and grease. Before beginning the grinding process, the sandpaper is placed on the grinding machine disc. The grinding process is then initiated using 100 mesh sandpaper while being cooled with running water. After reaching the 2000 mesh sandpaper, the polishing process is continued by rubbing the sample's surface with an auto sol-treated velvet cloth until the sample is smooth and flat. Then the substrate is washed using an ultrasonic cleaner machine with detergent water, distilled water, and finally using,

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alcohol. The substrate is then dried using a hairdryer. The next procedure is the sputtering process at the PRTA-BRIN Yogyakarta particle physics laboratory. The schematic of the DC sputtering device is shown in Figure 1.

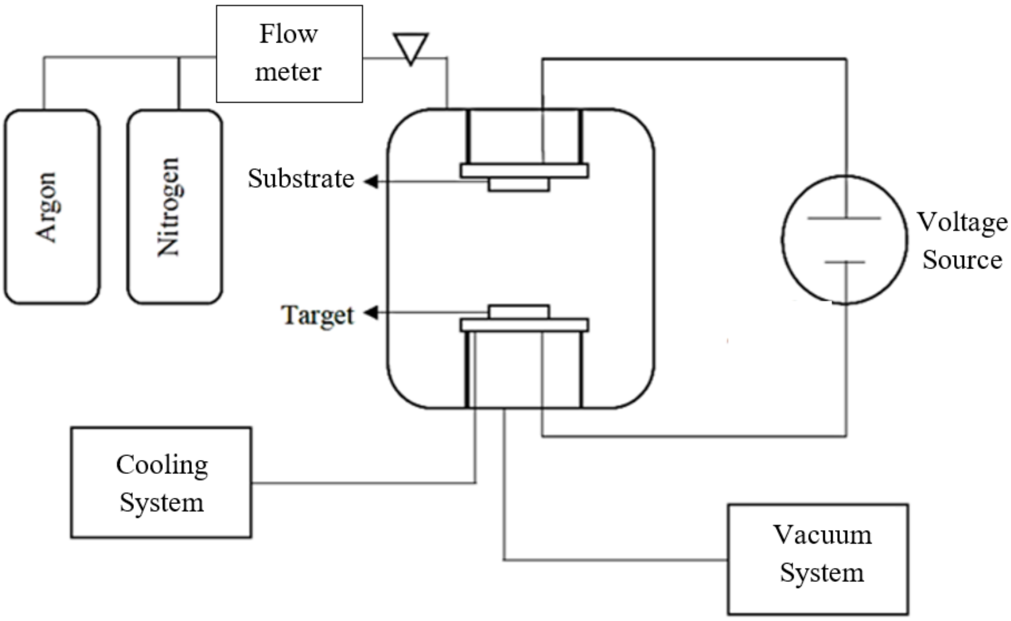


Figure 1. Schematic of DC Sputtering Device [18]

The substrate to be coated with a thin film is placed on the anode, and the target or Ti material to be deposited is placed on the cathode in a vacuum chamber. Low-pressure argon (Ar) gas is put into a vacuum chamber, and a voltage is applied to the two electrodes. Positively charged Ar ions bombard the Ti target at the cathode, which has a negative potential so that the material atoms will then be scattered from the target. These atoms will then stick to the surface of the substrate and form a thin film [19]. During the sputtering process, the parameters are adjusted according to the research conducted by Andriyanti et al. [20], the parameters kept constant were the pressure of the order 10^{-2} Torr, the voltage of 3 kV, the current of 10 mA, and the ratio of the gas mixture was 70 Ar: 30 N to obtain the optimal hardness value of TiO_2 thin film. The deposition times are varied to 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes. The deposited material is then ready for hardness testing and SEM-EDX characterization.

Characterization

Hardness Test. In the Vickers hardness test, a square indenter is used to apply pressure to the sample's surface. Each indenter trace has two diagonal lines (D1 and D2) that later will be processed by the test equipment to determine the hardness value of a material. The indenter load used in this measurement is 5 gf, with an indentation time of 10 seconds and a magnification of 40 times. Tests were carried out at six different points on the sample surface before and after sputtering. The hardness test was conducted to determine the change in hardness of the 316L stainless steel substrate, which had been coated with a TiO_2 thin film with variations in deposition time to obtain the optimum hardness value.

SEM-EDX. TiO_2 thin film samples were characterized using SEM analysis at the Integrated Laboratory, Gadjah Mada University, Yogyakarta. SEM characterization was performed on raw materials and specimens with optimum hardness values. The elemental composition and thickness of the TiO_2 layer deposited on the surface of 316L stainless steel samples were determined using SEM-EDX characterization.

RESULTS AND DISCUSSION

Hardness Test

Hardness characterizes a material's resistance to localized plastic indentation and abrasion, making it a crucial mechanical property in biomaterial selection and clinical use [4]. The hardness test in this study was performed on 316L stainless steel coated with a TiO_2 thin film using the DC sputtering method. Hardness testing was performed to determine the hardness value and level of 316L stainless steel material before and after coating. The method used in this hardness test is the Vickers hardness method with a pyramid-shaped indenter with an angle of 136°. The Vickers hardness is calculated from the length of the diagonal curvature that remains when the

Vickers indenter is pressed against the sample's surface with an applied test force, and then the force is removed [21]. This method was chosen because it has better accuracy and more noticeable indentation results than the Brinnel method.

Hardness testing was conducted at the PRTA-BRIN Yogyakarta particle physics laboratory using a micro-hardness tester of the Matsuzawa MMT-X7 type. This hardness testing machine has a correction factor of 1.3, which is the ratio of the measured value to the standard value. Thus, the hardness measurement value shown on the tool is multiplied by the correction factor to find out the actual hardness value. This machine has an adjustable load from 1 gf to 2000 gf and an adjustable indentation time of 5 seconds.

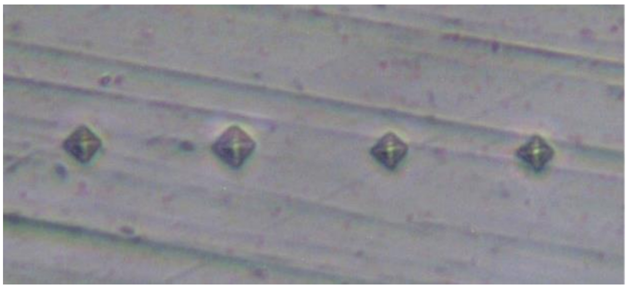


Figure 2. Traces of indents formed on the surface of the material

Vickers hardness testing is carried out on test objects that have previously been prepared, and this is because the test equipment will provide traces of indentation on the surfaces of the material as shown in the figure 2. Therefore, a smooth and flat surface is required to make it easier to read the traces. The test was performed with a 5 gf indentation load and a 10 second indentation time. For each specimen, the hardness value was measured six times. Data on hardness measurement results are shown in Table 1.

Table 1. Data from the sample hardness test results from the DC sputtering process

Sample	Load (kgf)	Indent Time (s)	Average hardness (VHN)	Correction Factor	Actual hardness value (VHN)
*Raw m*aterial			94.48		122.83
30 minutes			95.65		124.34
60 minutes	0.05	10	100.60	1.3	130.78
90 minutes			130.85		170.10
120 minutes			114.18		148.44
150 minutes			83.00		107.90

The data in Table 1 is then presented in graphical form in Figure 3 so that an increase and decrease in the hardness value in the sample can be observed based on the length of time of sputtering.

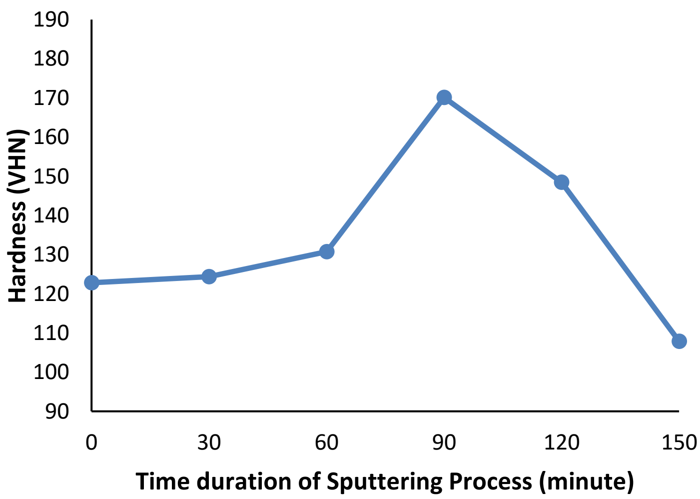


Figure 3. Graph of surface hardness values of samples from the DC sputtering process

https://doi.org/10.55981/gnd.2023.6834

Table 1 and Figure 3 shows that the lowest average hardness value is raw material, which is 122.83 VHN. Because ductility is opposite to the hardness value of the material, 316L stainless steel material has the highest ductility before surface treatment. The sample that obtained the maximum hardness value on the surface was 316L stainless steel after being deposited for 90 minutes with a hardness value of 170.10 VHN, while the ductility below the surface of the thin film material remained constant. Compared to the raw material, there was an increase in the sample hardness value of ~ 48.175% on the surface after being deposited for 90 minutes. Based on these data, it can be concluded that the longer the deposition time, the hardness value of the sample will increase, but only up to the optimum hardness point, namely at 90 minutes. The increase in hardness is due to the longer the deposition time, the greater the number of atoms deposited on the surface of the substrate; thus, the arrangement of the atoms coating the surface of the substrate will be denser. The optimum hardness value is achieved because, most likely, under these conditions, the layer formed on the sample's surface has a perfect arrangement of atoms with a high density so that a material with a high surface hardness value will be produced [22].

Research conducted by Andriyanti et al. [12] namely depositing titanium nitride (TiN) on the surface of 316L stainless steel material using the DC sputtering method, obtained an optimum material hardness value of 145 VHN at a deposition time of 90 minutes with a ratio of argon and nitrogen gas used was 70:30. Research conducted by Ananda Gifari et al. [16] produced Aluminum 5038 material with the highest hardness value of 94.7 VHN from an initial hardness of 52.14 VHN after being coated with TiN using the DC Sputtering method at a deposition time of 120 minutes. Sandeep et al. [23] conducted a study by coating aluminium bronze with nickel material using the electroplating coating method so that the thickness of the nickel layer formed was 200 μm with a hardness value of 806 VHN, with a comparison of the previously modified aluminum bronze hardness value of 204 VHN. Compared with these studies, the surface treatment to improve the mechanical properties, namely the hardness value of the material in this study, has been fulfilled.

The hardness value of the samples decreased at a deposition time of 120 and 150 minutes. This decrease in hardness value is called the supersaturation process [24] or the resputtering process [25]. This study reached the saturation point after the deposition process had passed 90 minutes. After reaching saturation conditions, if the deposition time is increased up to 120 and 150 minutes, the hardness value of the sample decreases. This is because when the deposition time exceeds the optimum hardness time of 90 minutes, the surface layer of the substrate with higher density will cause grain growth accompanied by the formation of vacancies and micropores. This means that the density of the layer formed will be relatively lower when compared to the density in the area around the surface. Therefore, the level of coating hardness obtained after passing through saturated conditions will decrease [26].

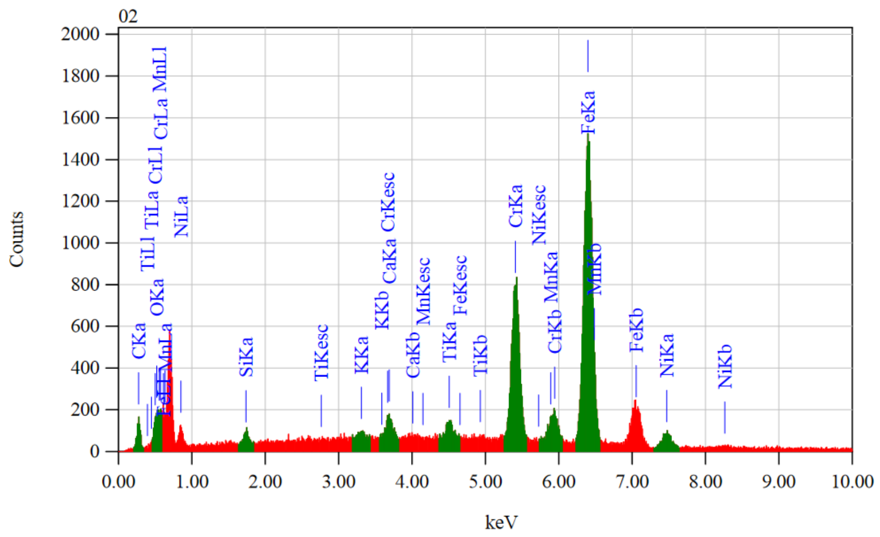
The decrease in hardness may also be caused by an increase in high plasma power resulting in an increase in energy and the number of Ar^+ ions so that more oxygen atoms are released when the ions collide with TiO_2 molecules. The collision can occur on the target, the molecules heading for the substrate, or on a thin layer that has been deposited. This impacts the destruction of the thin layer structure so that the deposited TiO_2 thin layer becomes an amorphous structure. The high plasma power causes the deposited thin layer to undergo a resputtering process so that the film thickness decreases [16].

According to the explanation above, depositing a TiO_2 layer with a high hardness value on the sample surface necessitates the appropriate deposition time. Biomaterials with a high hardness value will exhibit less wear, which can extend the life and safety of biomedical devices [4]. The SEM-EDX test was then performed to determine the thickness of the thin layer formed and the composition of the elements on the sample's surface.

SEM-EDX

Raw Material Characterization. This characterization aims to determine the elemental composition of the 316L stainless steel material used in the study. The EDX test results show the raw material's elemental composition, as shown in Figure 4.

From the results of the composition test, the dominant elements in the material used in this study were Fe, Cr, Ni, C, Mn, Ti, Ca and K, with a mass percentage of 64.58%; 19.20%; 5.62%; 4.78%; 1.50% ; 1.41%; 1.19% and 0.29% respectively. Research conducted by Hafizi et al. [27] stated that the elemental composition of 316L stainless steel is composed of less than 70% Fe; 10.1% Ni; 0.02% C; 17.2% Cr; 2.1% Mo; 0.003% Ti; 0.002% Al; 0.019% Nobidium and several other elements.



ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0140

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	4.78	0.08	18.18				1.4998
O K	0.525	0.99	0.06	2.83				1.2905
Si K	1.739	0.43	0.03	0.70				0.3270
K K	3.312	0.29	0.03	0.34				0.3287
Ca K	3.690	1.19	0.05	1.35				1.4524
Ti K	4.508	1.41	0.07	1.35				1.5260
Cr K	5.411	19.20	0.24	16.85				21.4647
Mn K	5.894	1.50	0.12	1.25				1.5153
Fe K	6.398	64.58	0.52	52.78				65.1082
Ni K	7.471	5.62	0.22	4.37				5.4876
Total		100.00		100.00				

Figure 4. EDX micrograph of 316L stainless steel material

TiO_2 Thin Film Characterization. The SEM-EDX characterization in this study aims to observe and determine the thickness and elemental composition of the TiO_2 layer deposited on the surface of 316L stainless steel material by the DC sputtering method. This test was carried out at the Integrated Laboratory of Gadjah Mada University on raw material test objects and objects coated with TiO_2 with optimum hardness values, with a deposition time of 90 minutes. The EDX test results show the TiO_2 thin film elemental composition, as shown in Figure 5.

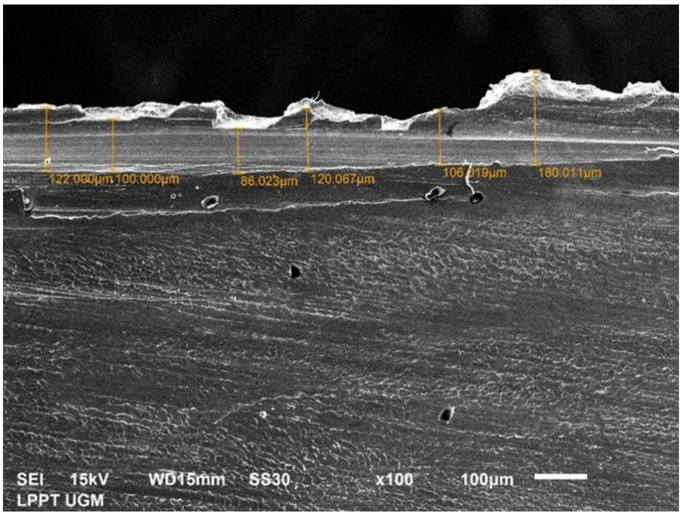


Figure 5. The cross-section morphology of the TiO_2 thin film

Figure 5 shows the results of the SEM test showing the cross-section of the test material. It can be observed that there is a TiO_2 layer formed on the surface of the 316L stainless steel material with an average layer thickness of ± 119.02 µm. This is indicated by the difference in color on the sample's surface. The color which tends to be lighter is assumed to be the TiO_2 coating, and the color which tends to be darker is assumed to be 316L stainless steel material. There is a difference in the thickness of the TiO_2 layer in the sample, which is probably due to the inhomogeneity of the substrate surface during the preparation process [14] and the influence of parameters

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such as temperature differences at each substrate surface point. Since DC sputtering machines cannot control temperature, the temperature at any point on the substrate surface cannot be known. The higher the temperature of the substrate, the wider the distance between atoms, making it easier for TiO_2 atoms or compounds to diffuse deeper so that a thin layer will be produced. Conversely, when the substrate temperature gets lower, the distance between atoms gets smaller, and diffusion becomes more difficult so that the TiO_2 compound that is formed accumulates on the surface of the substrate and produces a thicker layer [20].

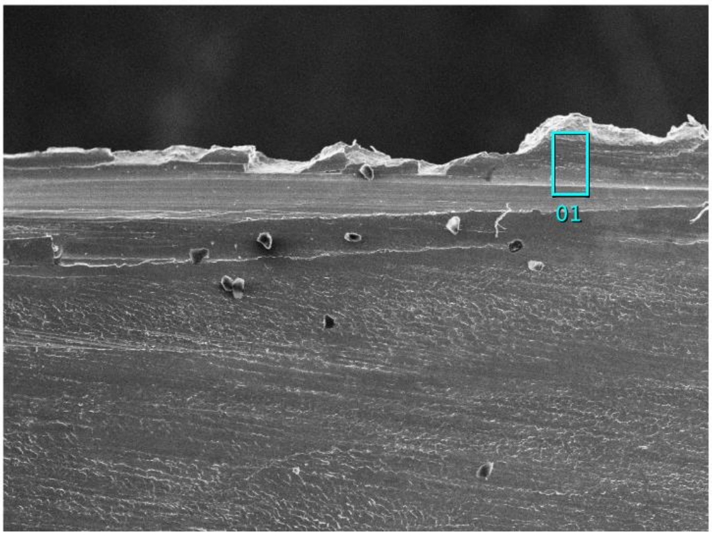
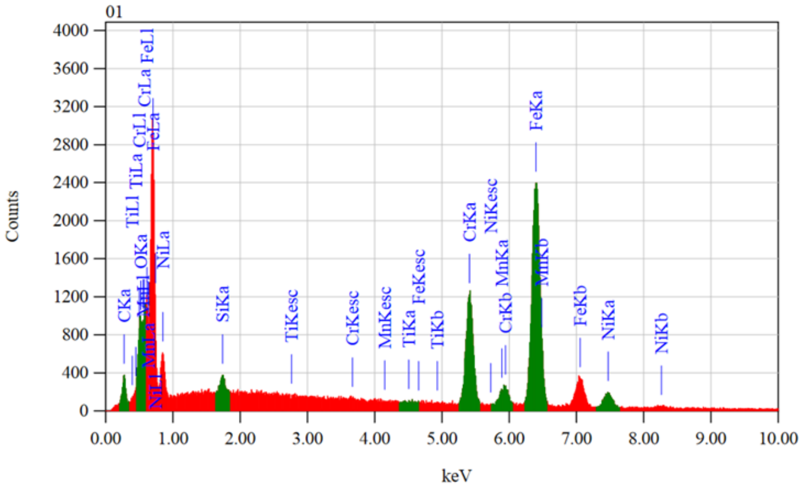


Figure 6. EDX testing at point 01 with 100x magnification

As shown in Figure 6, using the EDX technique, the elemental composition of the TiO_2 layer was determined at point 01 on the surface of 316L stainless steel material resulting from the DC sputtering process. EDX is a technique for analyzing the elemental components of materials included with the SEM tool. This method detects X-rays produced by the interaction of an electron beam and a sample. This method can be obtained by mapping the distribution of chemical elements in the material [14]. Figure 7 shows the SEM-EDX micrograph on a cross-section of 316L stainless steel material after the DC sputtering process with a deposition time of 90 minutes.



ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.0117

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K	0.277	6.35	0.07	22.46				1.9617
O K	0.525	2.62	0.07	6.95				3.6329
Si K	1.739	0.75	0.04	1.13				0.5683
Ti K*	4.508	0.07	0.03	0.07				0.0812
Cr K	5.411	17.94	0.18	14.66				20.3961
Mn K	5.894	1.28	0.08	0.99				1.3009
Fe K	6.398	63.76	0.41	48.51				64.9531
Ni K	7.471	7.23	0.19	5.23				7.1056
Total		100.00		100.00				

Figure 7. EDX micrograph of 316L stainless steel material after the DC sputtering process

Using the EDX technique, the elemental composition of the TiO_2 coating on the surface of 316L stainless steel material resulting from the DC sputtering process was determined. Figure 7 shows that a thin layer of TiO_2 has been deposited on the surface of the 316L stainless steel material. The content of Ti and O deposited elements are 0.07% and 2.62% by weight respectively, which comes from the DC sputtering process. In the spectrum, other elements, such as C, Si, Cr, Fe, and Ni, are the constituent elements of 316L stainless steel [14]. The C content has increased compared to before the DC sputtering process, possibly due to the vacuum environment during the deposition process [28]. The increase in O content is possible due to corrosion that arises because the substrate is not stored in a vacuum [20].

CONCLUSION

The study showed that samples that underwent the deposition process for 90 minutes using the DC sputtering method had an optimum hardness value of 170,01 VHN. The SEM characterization results of this sample showed that a TiO_2 thin film had formed on the surface of 316L stainless steel with a thickness of $\pm$ 119.02 μm. Based on the EDX test it was found that the contents of the elements Ti and O were 0.07% and 2.62% by weight, which came from the DC sputtering process.

ACKNOWLEDMENTS

The author would like to thank the Particle Physics laboratory facilities at PRTA-BRIN Yogyakarta, as well as Bu Wiwien Andriyanti and Prof. Heri Sutanto for their guidance and assistance in this research process.

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