Bukti ***corresponding author***

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authors: Ratna Kartikasari dan Marwan Effendy

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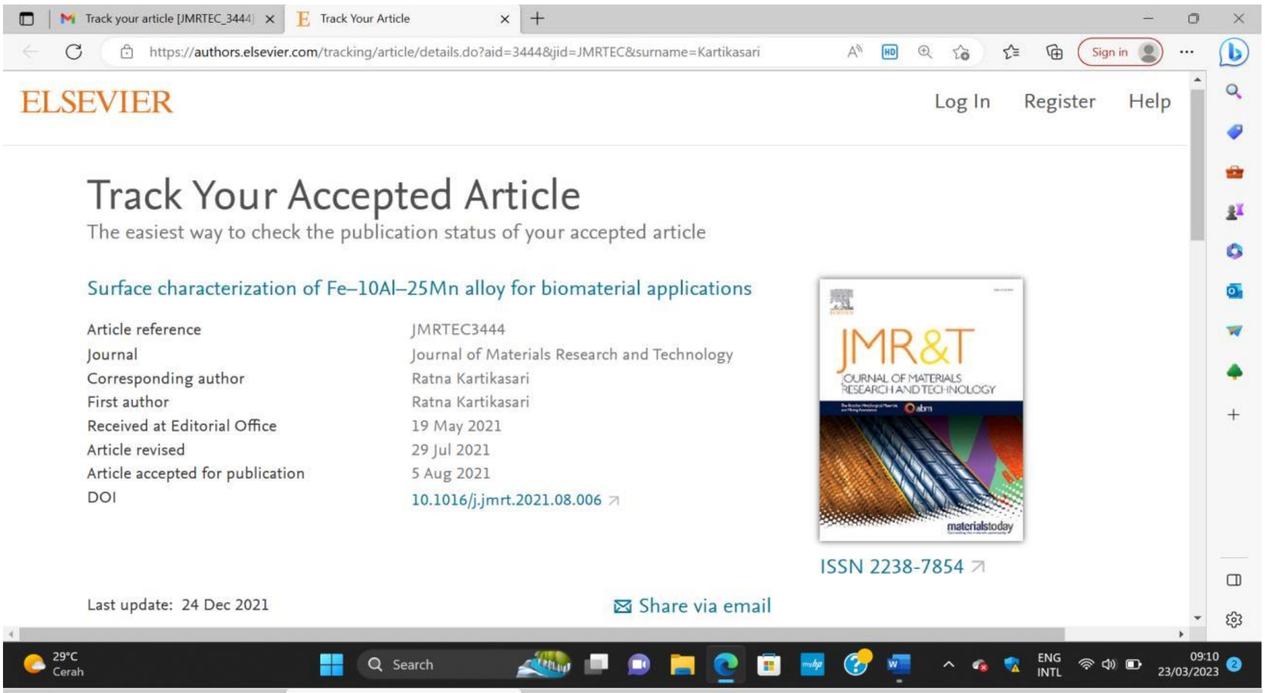

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Surface characterization of Fe–10Al–25Mn alloy for biomaterial applications

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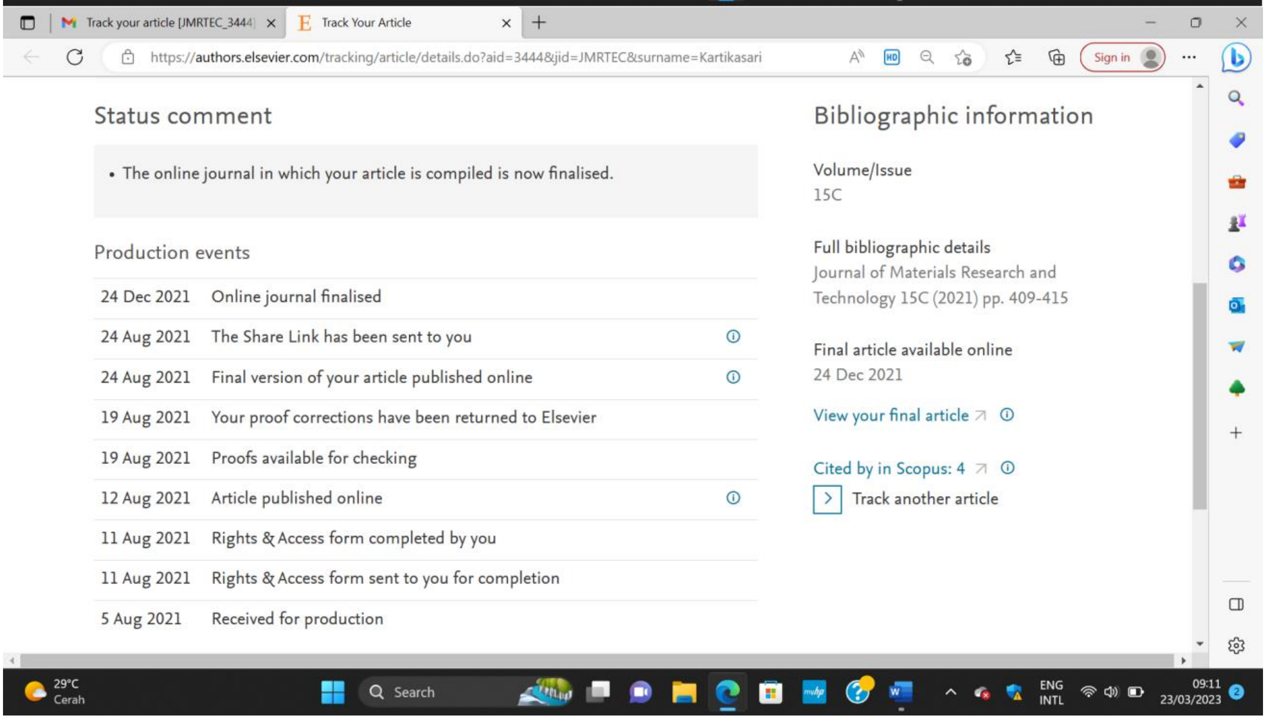

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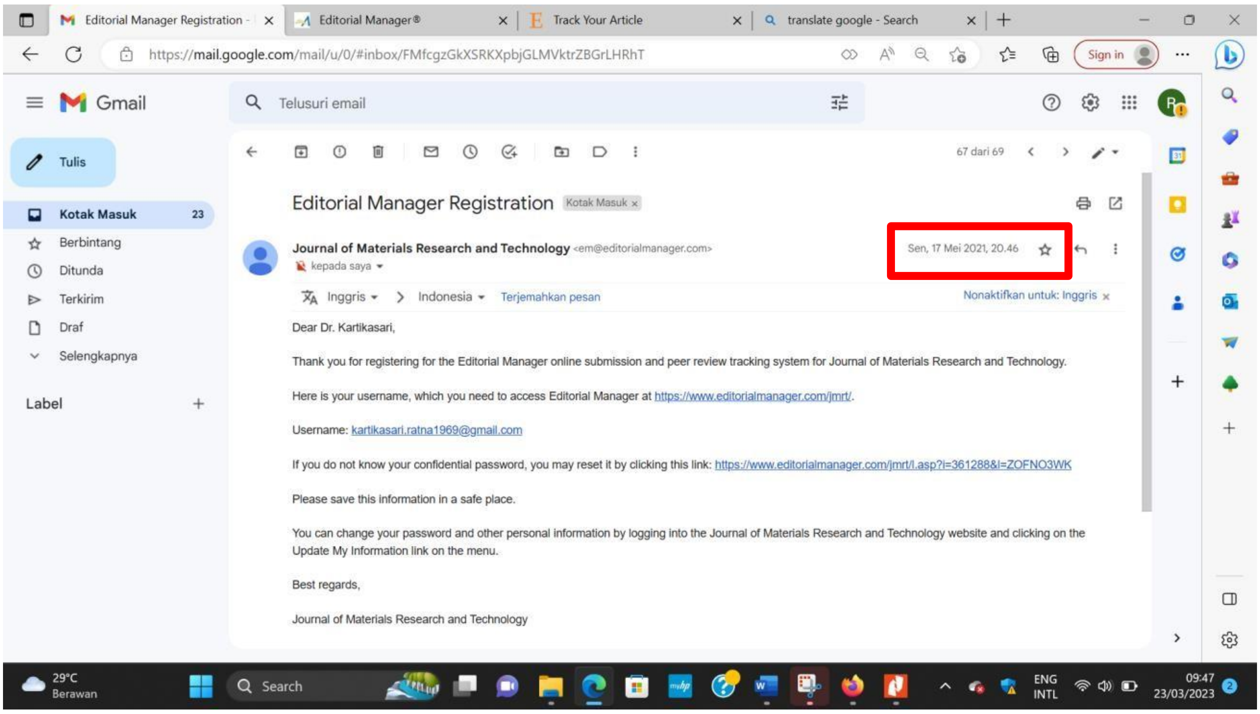

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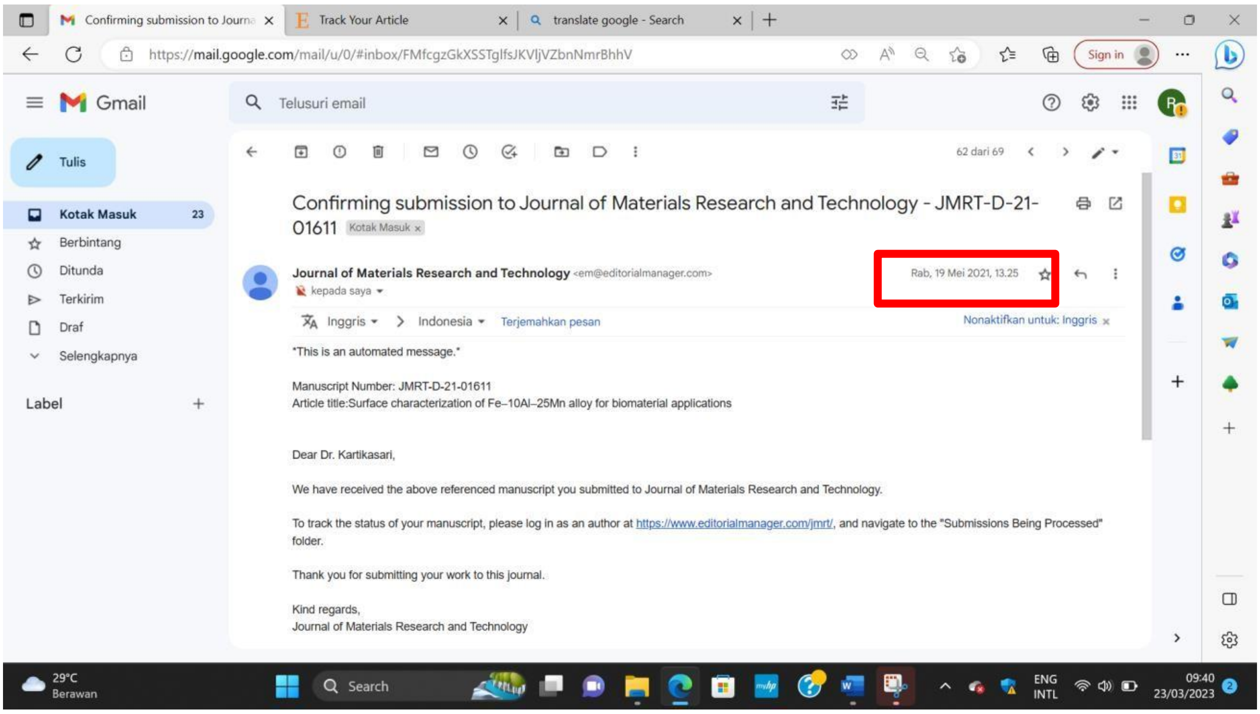

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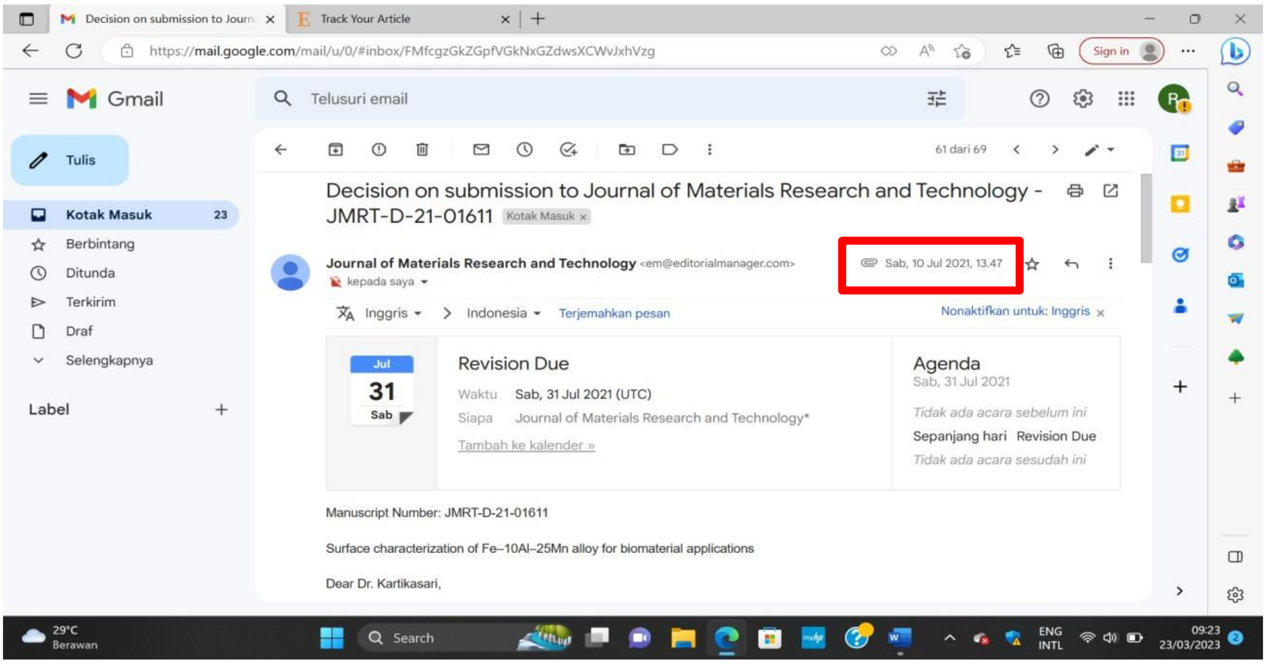

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Surface characterization of Fe–10Al–25Mn alloy for biomaterial applications

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Journal of Materials Research and Technology

Editor and Reviewer comments:

Reviewer #1: The manuscript reported a FeAlMn alloy for biomedical applications. Major revision is suggested before considering for publish.

1. In introduction, authors claimed that the selection of materials should not cause harmful effects, but FeNiCr alloys are known to cause biosafety problems. Please rearranging this part.
2. Al is a well-known neurotoxic element and has been reported to cause hepatoxicity and allergic problems, how do authors consider the biocompatibility and biosafety of the Fe-Al-Si alloy.
3. The third paragraph regarding the corrosion environment is encouraged to delete, since this part is irrelevant with the discussion of introduction.
4. ASTM norm should be specified in the manuscript when mentioned.
5. More experiments should be performed to evaluate the corrosion behaviour, such as EIS, Tafel, and SEM observation.
6. More discussion of the corrosion mechanism should be included. The following literatures could provide some useful advises: (1) Mechanically driving supersaturated Fe-Mg solid solution for bone implant: Preparation, solubility and degradation; (2) In-situ deposition of apatite layer to protect Mg-based composite fabricated via laser additive manufacturing.

Reviewer #2: 1. "Abstract" "The results showed that the mechanical properties of the Fe-10Al-25Mn alloy are generally close to AISI 316L stainless steel,…." Not data support this result.

2. "Introduction" the expression is not logical, I can not find the value of this research.

3. Line 50: The Co-Cr alloy-based material was a popular material used for medical implants and dental prosthetics because of its high strength, dependable wear and corrosion resistance [5], and antibacterial [6] ; Line 62-64: This modulus mismatch could prevent the bone healing process…..which has a greater potency in terms of corrosion and wear. It is hard to understand this expression.

4. Line116: "It means that the new alloys being developed must be high strength, wear-resistant, corrosion-resistant, antibacterial and non-toxic.", while in line 60:" However, it has several disadvantages, e.g., steel being denser, stronger, and a higher modulus of elasticity than the human bones. This modulus mismatch could prevent the bone healing process." Sure, there exists conflict for these two expressions. Now that being stronger is a disadvantage, why developing alloys with higher strength?

5. Line 215: the nitrogen content on the surface of the Fe-10Al-25Mn alloy is sensitive to the plasma nitriding temperature. The distance between the atoms of the Fe-10Al-25Mn alloy specimen becomes increasingly tenuous with the rise of the plasma nitriding temperature.

6. Line 298 "It means that the newly developed material will be a reliable replacement for conventional medical stainless steels because it combines the advantages of a stable austenitic structure, improved corrosion and wears resistance compared to the currently used AISI 316L stainless steel. Therefore, along with the Fe-Cr-Ni biomaterial, the Fe-10Al-25Mn alloy can be developed as a prospective biomaterial. However, it still needs further clinical examination for its use." I do not think the conclusion is reliable and reasonable; also no wears resistance was tested.

7. Fig. 7. The corrosion rate of Fe-10Al-25Mn alloy, and the corrosion rate is 0.04 mm/year. How did you get the corrosion rate?

8. Generally, the ingots will not be directly used in applications; deformation is needed for getting uniform microstructure.

Surface characterization of Fe–10Al–25Mn alloy for biomaterial applications
Ratna Kartikasari, Marwan Effendy

	Reviewer's Comments	Response
General response The authors have provided the editorial revisions to the sentences as presented with yellow marks in the main texts. A comprehensive correction has been made by considering the comments and suggestions of both reviewers, including from the editor. Considering the page constraints in the JMRT journal, not all of the results can be fully-added to the revised manuscript. Besides, some descriptions were also removed, considering the urgency of contents.		
Reviewer 1:		
The manuscript reported a FeAlMn alloy for biomedical applications. Major revision is suggested before considering for publish.		
1)	In introduction, authors claimed that the selection of materials should not cause harmful effects, but FeNiCr alloys are known to cause biosafety problems. Please rearranging this part.	The authors have resolved this issue. The metallic biomaterials play a crucial role in reconstructing the human body in orthopedic surgery and dentistry [1]. Some literature noted that at least three implant materials are broadly used for medical purposes, such as stainless steel [2], titanium (Ti) alloys [3], and cobalt-chromium (Co–Cr) based alloys [4]. Because of its high strength, dependable wear and corrosion resistance [5], and antibacterial properties [6], the Co–Cr alloy-based material is considerably used in medical implants and dental prosthetics. The suitability of the biological system of the human body is an essential consideration in developing biomaterials. Therefore, materials should not cause harmful effects and not damage the tissues of the human body. It must be free from toxic products and not trigger allergies, inflammation, or cancer [7]. Furthermore, biomaterials require clinical testing to ensure their use [8][9].
2)	Al is a well-known neurotoxic element and has been reported to cause hepatoxicity and allergic problems, how do authors consider the biocompatibility and biosafety of the Fe-Al-Si alloy.	This research focuses on developing biomaterials of Fe–10Al–25Mn alloy by nitriding to increase the mass transfer of molecules and high-energy nitrogen ions to the material surface and improve the control of process parameters. Although, the content of Al still leaves problems in this research. This comment becomes a valuable consideration for the next stage of our research roadmap. In this regard, this study focuses on developing a biomaterial by eliminating both Cr and Ni contents. New biomaterials are directed towards stainless steel without a nickel to reduce the toxic properties of AISI 316L stainless steel. The biomaterial compounds made are also expected to be more robust and corrosion-resistant compared to pure metal.

3)	The third paragraph regarding the corrosion environment is encouraged to delete, since this part is irrelevant with the discussion of introduction.	It has been deleted as suggested by the reviewer.
4)	ASTM norm should be specified in the manuscript when mentioned	The authors have checked this concern.
5)	More experiments should be performed to evaluate the corrosion behaviour, such as EIS, Tafel, and SEM observation.	The authors would like to thank the reviewer suggestion. Due to the limitation of journal pages on the JMRT, the authors only discuss the main tests in the first part of the manuscript. Meanwhile, the authors are being prepared another paper using different tests as suggested.
6)	More discussion of the corrosion mechanism should be included. The following literatures could provide some useful advises: (1) Mechanically driving supersaturated Fe-Mg solid solution for bone implant: Preparation, solubility and degradation; (2) In-situ deposition of apatite layer to protect Mg-based composite fabricated via laser additive manufacturing.	It has been added to sharpen the critical discussion of corrosion.
Reviewer 2:		
1)	"Abstract" "The results showed that the mechanical properties of the Fe-10Al-25Mn alloy are generally close to AISI 316L stainless steel,…." Not data support this result!!	The authors have addressed this issue by reconstructing the sentences. The results indicated that the treatment of plasma nitriding at temperatures up to 500 °C significantly enhances the corrosion resistance and increases the hardness of the alloy. At 500 °C, the percentage of nitrogen atoms reaches a peak and then decreases. It means that the nitride formation process on the alloy surface occurs more massively. The amount of nitrogen deposited on the surface of the Fe–10Al–25Mn alloy, as well as a thin layer of iron nitride, is noticeable in the SEM-EDS test. Furthermore, the formation of phases due to the nitriding temperature significantly impacts the alloy properties.
2)	Introduction" the expression is not logical, I can not find the value of this research.	Some sentences have been edited to have a better introduction.
3)	Line 50: The Co-Cr alloy-based material was a popular material used for medical implants and dental prosthetics because of its high strength, dependable wear and corrosion resistance [5], and antibacterial [6] ; Line 62-64: This modulus mismatch could prevent the bone healing process.….which has a greater potency in terms of corrosion and wear. It is hard to understand this expression	The authors have reconstructed the sentence to avoid ambiguity. In the last decades, the application of metallic biomaterials in the medical sector has been overgrown, such as in orthopedic implant devices and dentures. With material engineering and the development of the latest technology, millions of people seem to have a new spirit in improving their quality of life. The metallic biomaterials play a crucial role in reconstructing the human body in orthopedic surgery and dentistry [1]. Some literature noted that at least three implant materials are broadly used for medical purposes, such as stainless steel [2], titanium (Ti) alloys [3], and cobalt–chromium

		(Co–Cr) based alloys [4]. Because of its high strength, dependable wear and corrosion resistance [5], and antibacterial properties [6], the Co–Cr alloy-based material is considerably used in medical implants and dental prosthetics. The suitability of the biological system of the human body is an essential consideration in developing biomaterials. Therefore, materials should not cause harmful effects and not damage the tissues of the human body. It must be free from toxic products and not trigger allergies, inflammation, or cancer [7]. Furthermore, biomaterials require clinical testing to ensure their use [8], [9]. Austenitic stainless steel, AISI 316L, is extensively used for orthopedic and prosthetic implant devices. This material has reliable mechanical characteristics at an affordable price. The manufacturing process of this material is also relatively easy compared to others [10]–[12]. The main advantages of SS-AISI-316L are inexpensive, stable mechanical properties and easy manufacturing process. However, problems with medical stainless steel have been discovered in recent decades of clinical use. Firstly, the stainless steel used in medical applications commonly has a higher elastic modulus than bone. This incompatibility of strength or modulus can result in a stress shielding effect, which can impede bone healing. Secondly, stainless steel is considered less able to withstand wet conditions in a fluid body environment, which has a greater potency in corrosion and wear. This could lead to premature fracture and massive corrosion of the implanted device, followed by releasing harmful substances to the human body. A study reported that both nickel (Ni) and chromium (Cr) are among the potential hazards in medical stainless steels [13]. Allergic contact dermatitis affected by Ni is the most common type of metal hypersensitivity reaction. Furthermore, high Cr levels in the human body may cause cancer and other diseases [1].
4)	Line116: "It means that the new alloys being developed must be high strength, wear-resistant, corrosion-resistant, antibacterial and non-toxic.", while in line 60:" However, it has several disadvantages, e.g., steel being denser, stronger, and a higher modulus of elasticity than the human bones. This	The authors have reconstructed the sentence to avoid ambiguity. The main advantages of SS-AISI-316L are inexpensive, stable mechanical properties and easy manufacturing process. However, problems with medical stainless steel have been discovered in recent decades of clinical

	modulus mismatch could prevent the bone healing process." Sure, there exists conflict for these two expressions. Now that being stronger is a disadvantage, why developing alloys with higher strength?	use. Firstly, the stainless steel used in medical applications commonly has a higher elastic modulus than bone. This incompatibility of strength or modulus can result in a stress shielding effect, which can impede bone healing. Secondly, stainless steel is considered less able to withstand wet conditions in a fluid body environment, which has a greater potency in corrosion and wear
5)	Line 215: the nitrogen content on the surface of the Fe-10Al-25Mn alloy is sensitive to the plasma nitriding temperature. The distance between the atoms of the Fe-10Al-25Mn alloy specimen becomes increasingly tenuous with the rise of the plasma nitriding temperature.	The authors have reconstructed the sentences.
6)	Line 298 "It means that the newly developed material will be a reliable replacement for conventional medical stainless steels because it combines the advantages of a stable austenitic structure, improved corrosion and wears resistance compared to the currently used AISI 316L stainless steel. Therefore, along with the Fe-Cr-Ni biomaterial, the Fe-10Al-25Mn alloy can be developed as a prospective biomaterial. However, it still needs further clinical examination for its use." I do not think the conclusion is reliable and reasonable; also no wears resistance was tested.	The authors have reconstructed the sentences to avoid ambiguity. It means that the newly developed material will be a reliable replacement for traditionary medical stainless steel. This material combines the advantages of a stable austenitic structure and corrosion resistance. Therefore, the Fe–10Al–25Mn nitride alloy can be developed as a prospective biomaterial.
7)	The corrosion rate of Fe-10Al-25Mn alloy, and the corrosion rate is 0.04 mm/year. How did you get the corrosion rate?	The authors have deleted the sentence due to a typo in the abstract. It should be 0.013 mm/yr. The calculation of corrosion rate is based on the formula used by Li et al. (2015). However, we decided to change the sentence to be more general as follows The results indicated that the treatment of plasma nitriding at temperatures up to 500 °C significantly enhances the corrosion resistance and increases the hardness of the alloy. At 500 °C, the percentage of nitrogen atoms reaches a peak and then decreases. It means that the nitride formation process on the alloy surface occurs more massively. The amount of nitrogen deposited on the surface of the Fe–10Al–25Mn alloy, as well as a thin layer of iron nitride, is noticeable in the SEM-EDS test..
8)	Generally, the ingots will not be directly used in applications; deformation is needed for getting uniform microstructure!	The authors agree with the suggestions. It has been revised some related sentences to improve the contents. Considering the limitations on the number of pages allowed in the JMRT journal, not all experimental results can be fully added to the manuscript. However, this feedback is precious in developing the next manuscript.

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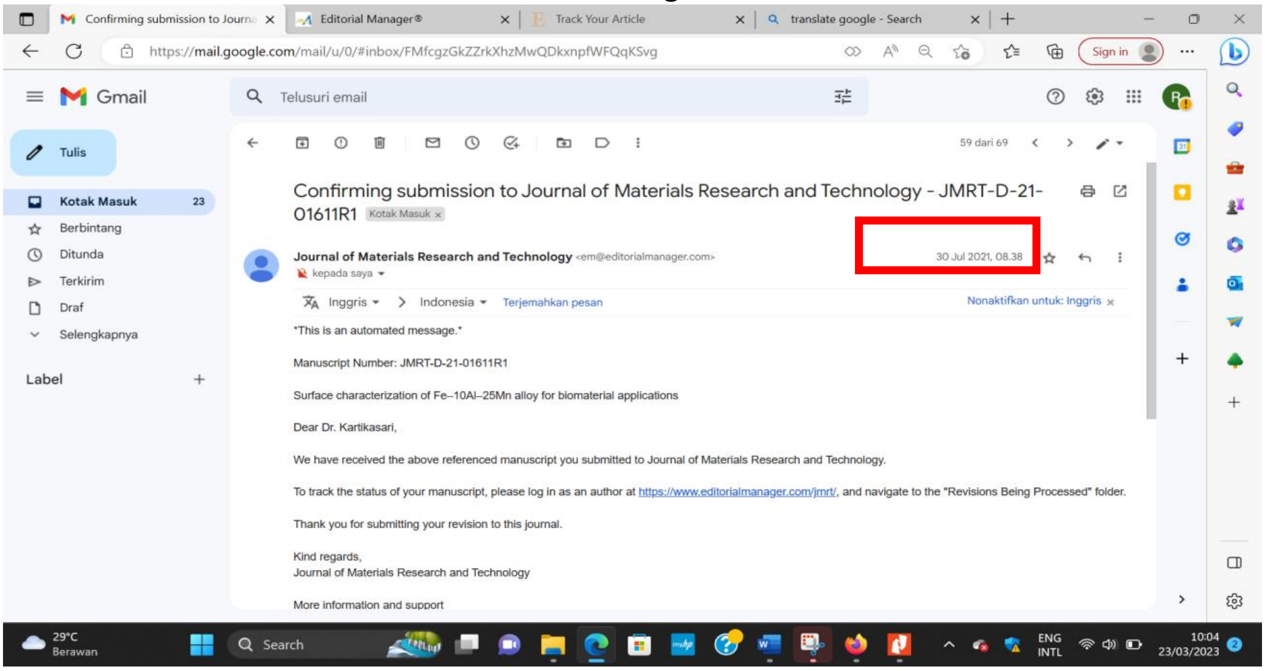

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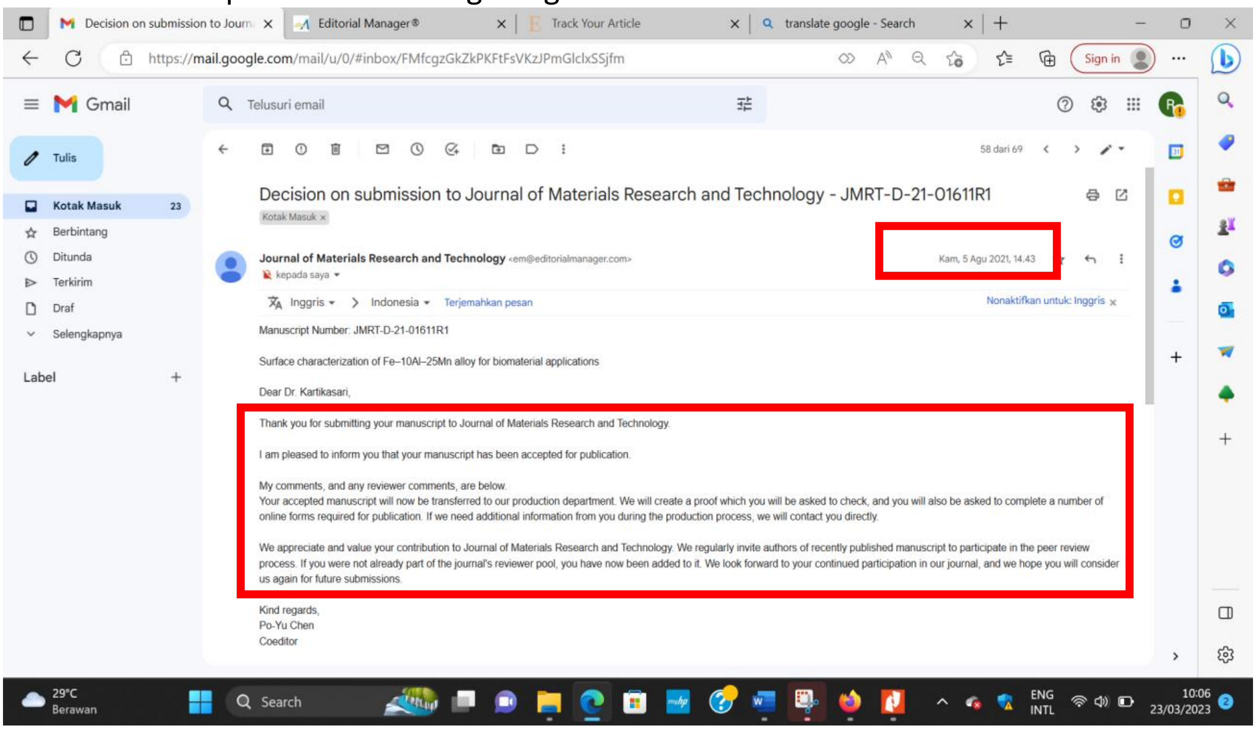

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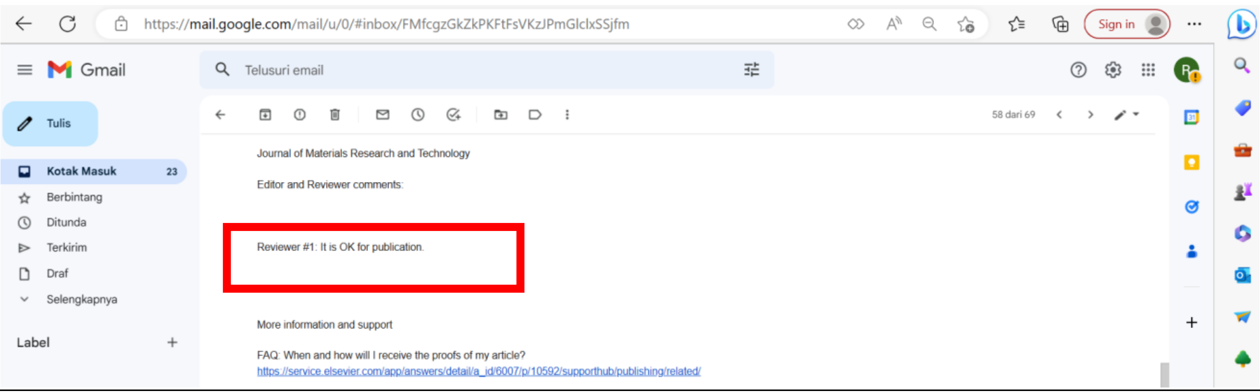
We appreciate and value your contribution to Journal of Materials Research and Technology. We regularly invite authors of recently published manuscript to participate in the peer review process. If you were not already part of the journal's reviewer pool, you have now been added to it. We look forward to your continued participation in our journal, and we hope you will consider us again for future submissions.

Kind regards,
Po-Yu Chen
Coeditor



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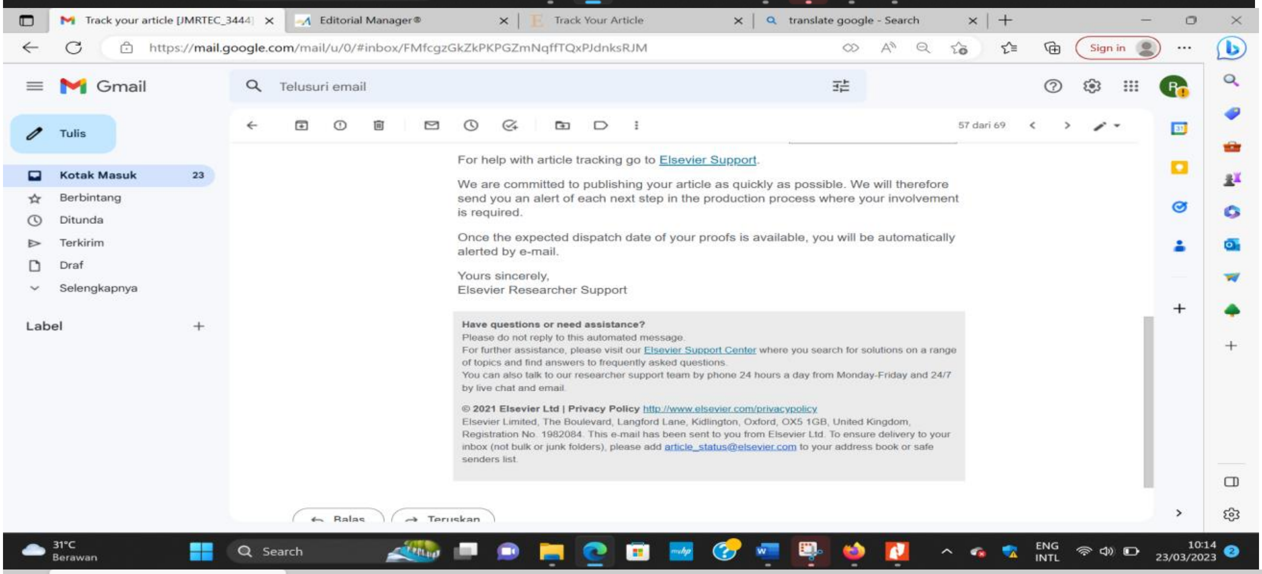

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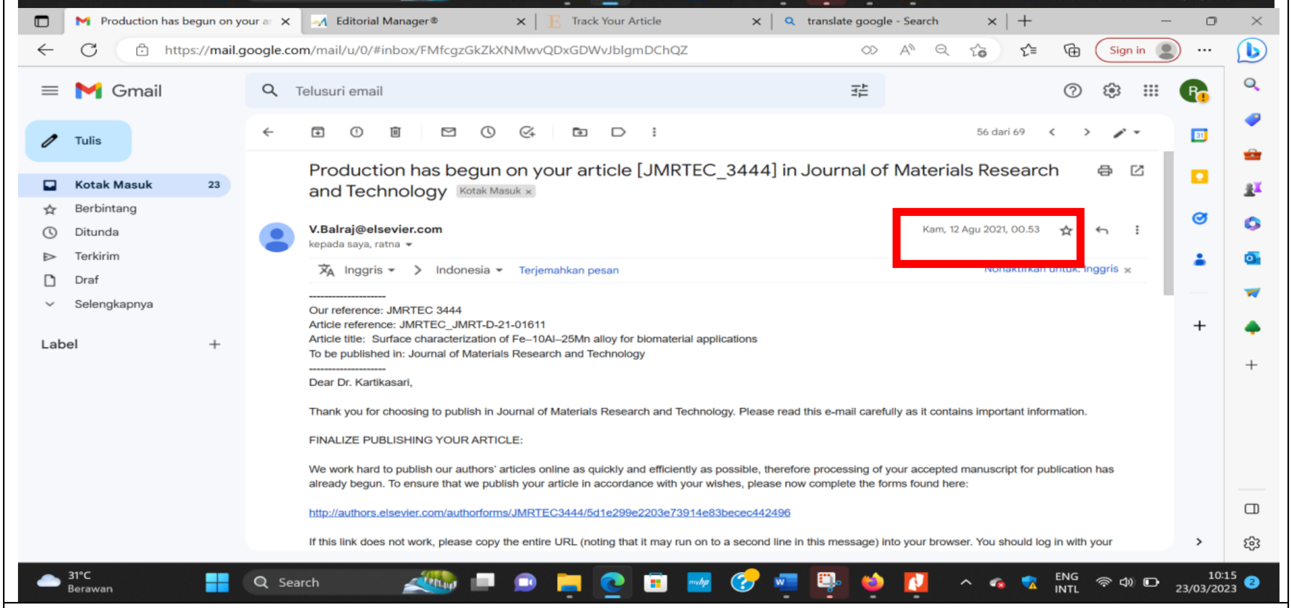

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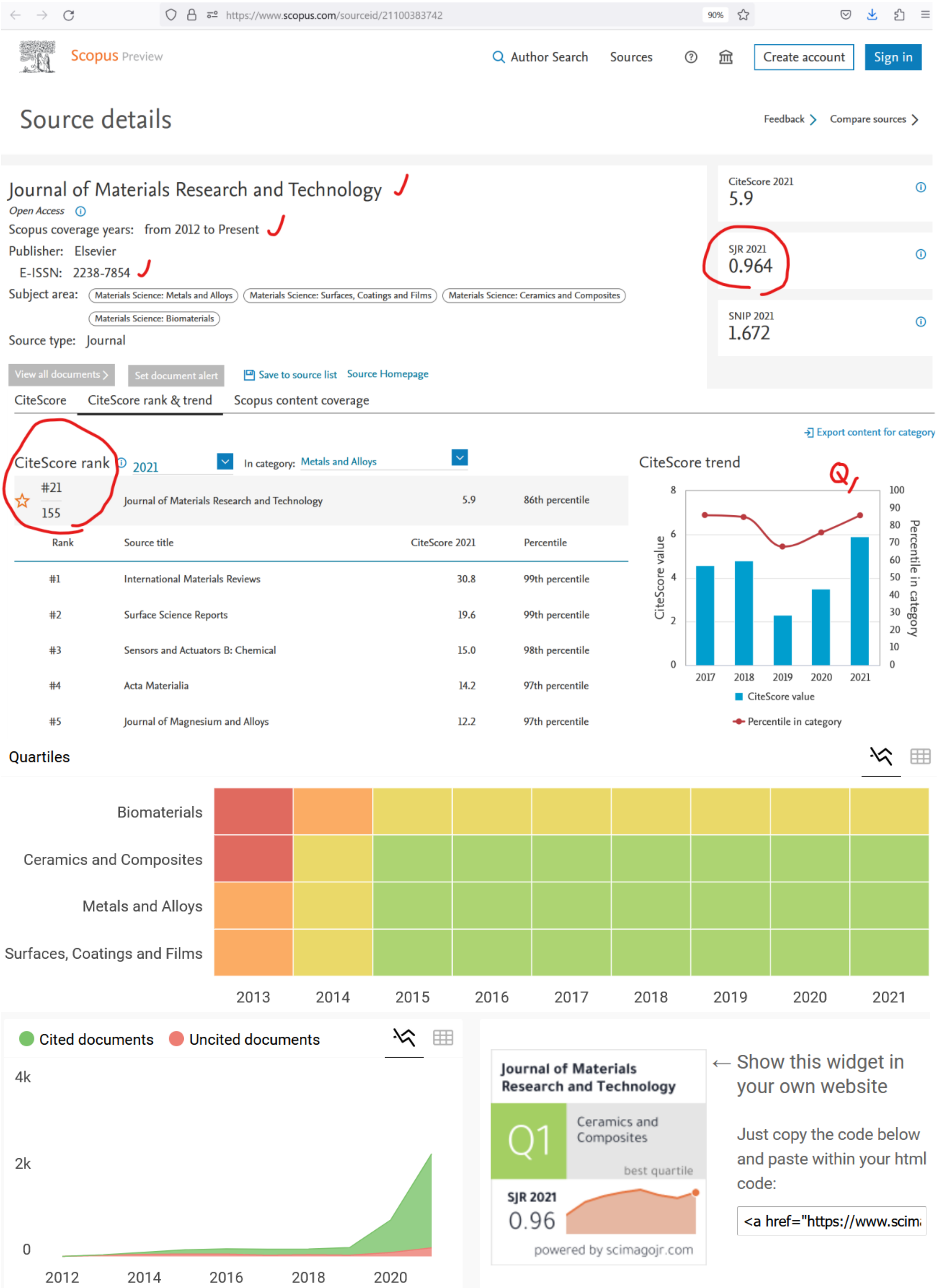

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Original Article

Surface characterization of Fe–10Al–25Mn alloy for biomaterial applications

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ABSTRACT

The austenitic stainless-steel biomaterial, AISI 316L stainless steel, is one of the most widely used for orthopedic or prosthetic implant devices because it is easy to manufacture at a relatively low cost. However, corrosion is a challenging issue related to major alloying compounds with body fluids and wear. Therefore, this study aimed to develop a biomaterial of Fe–Al–Mn alloys by minimizing chromium (Cr) and nickel (Ni) contents. In the research, an attempt has been made to increase the corrosion resistance of Fe–10Al–25Mn alloy using plasma nitriding, which was considered one of the most cost-effective surface treatments. The processes were carried out at various treatment temperatures between 350 and 550 °C, with a pressure of 1.8 mbar in 3 h. Several tests were performed, such as chemical compositions, scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS), hardness, and corrosion. The results indicated that the treatment of plasma nitriding at temperatures up to 500 °C significantly enhances the corrosion resistance and increases the hardness of the alloy. At 500 °C, the percentage of nitrogen atoms reaches a peak and then decreases. It means that the nitride formation process on the alloy surface occurs more massively. The amount of nitrogen deposited on the surface of the Fe–10Al–25Mn alloy, as well as a thin layer of iron nitride, is noticeable in the SEM-EDS test. Furthermore, the formation of phases due to the nitriding temperature significantly impacts the alloy properties.

1. Introduction

In the last decades, the application of metallic biomaterials in the medical sector has been overgrown, such as in orthopedic implant devices and dentures. With material engineering and the development of the latest technology, millions of people seem to have a new spirit in improving their quality of life. The metallic biomaterials play a crucial role in reconstructing the human body in orthopedic surgery and dentistry [1]. Some literature noted that at least three implant materials are broadly used for medical purposes, such as stainless steel [2], titanium (Ti) alloys [3], and cobalt–chromium (Co–Cr) based

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alloys [4]. Because of its high strength, dependable wear and corrosion resistance [5], and antibacterial properties [6], the Co–Cr alloy-based material is considerably used in medical implants and dental prosthetics. The suitability of the biological system of the human body is an essential consideration in developing biomaterials. Therefore, materials should not cause harmful effects and not damage the tissues of the human body. It must be free from toxic products and not trigger allergies, inflammation, or cancer [7]. Furthermore, biomaterials require clinical testing to ensure their use [8,9].

Austenitic stainless steel, AISI 316L, is extensively used for orthopedic and prosthetic implant devices. This material has reliable mechanical characteristics at an affordable price. The manufacturing process of this material is also relatively easy compared to others [10–12]. The main advantages of SS-AISI-316L are inexpensive, stable mechanical properties and easy manufacturing process. However, problems with medical stainless steel have been discovered in recent decades of clinical use. Firstly, the stainless steel used in medical applications commonly has a higher elastic modulus than bone. This incompatibility of strength or modulus can result in a stress shielding effect, which can impede bone healing. Secondly, stainless steel is considered less able to withstand wet conditions in a fluid body environment, which has a greater potency in corrosion and wear. This could lead to premature fracture and massive corrosion of the implanted device, followed by releasing harmful substances to the human body. A study reported that both nickel (Ni) and chromium (Cr) are among the potential hazards in medical stainless steels [13]. Allergic contact dermatitis affected by Ni is the most common type of metal hypersensitivity reaction. Furthermore, high Cr levels in the human body may cause cancer and other diseases [1].

It is well known that the liquid contains about 0.9% salt in the human body, a pH of 7.4, and a temperature of about 37 °C [14]. Therefore, biomaterials naturally interact continuously with body fluids. This interaction certainly affects the metals and alloys attached to the human body. Naturally, the complexity of the human body with a wet environment triggers corrosion of almost all metal-based biomaterials, which is followed by chemical and electrochemical degradation. Even the most corrosion-resistant materials are difficult to escape from this natural process. Therefore, biomaterials must be biocompatible and non-stimulating by the body systems, non-toxic, and withstand repeated loads in an aggressive body environment [15]. Besides, biomaterials must have physical and mechanical properties that are reliable to replace body systems, be easily formed and produced at a relatively low cost [4].

Recently, high manganese (Mn) austenitic steels with nitrogen alloys have improved strength, toughness, corrosion resistance, and nonmagnetic properties, making them a viable replacement for traditional Cr–Ni stainless steels in orthopedic implants and other medical applications. The development of manganese-based alloys has been reported for bio-absorbable implants [16]. A metal vapor vacuum is used to implant the biocompatible manganese (Mn) compound into the biomedical Mg surface. This is subjected to evaluate the impact of Mn ion cultivation on the corrosion phenomenon of biomedical Mg. This study found that the surface roughness could be reduced by Mn ion implantation. In a recent study using Fe–Mn–C alloys, the increase of Mn contents reduced the mechanical resistance [17]. Another study found that the addition of Ca to Fe–Mn–Si alloys could improve the osteoinduction and osteoconduction processes better than Fe–Mn–Si alloys or standard AISI 316L stainless steel. The ability to degrade at higher corrosion rates appears to be more optimal [18]. In addition, the suitability of Fe–35Mn–5Si as a biodegradable implant has been improved by considering its mechanical and corrosion properties [19].

Metallic biomaterials appear to be required for patients to support diseased tissue for as long as necessary. However, the capability of implants to degrade uniformly under various conditions in the human body to avoid cytotoxic effects and inappropriate tissue responses remains a significant concern. It means that the new alloys being developed must be high strength, wear-resistant, corrosion-resistant, antibacterial and non-toxic. One of the most widely used industrial processes is plasma nitriding, including nitrogen absorption by diffusion into the structure of a material. This approach mainly applies to tools and low alloy steels, considering their most inadequate surface treatments costs. The main benefit of plasma nitriding is to increase the mass transfer of molecules and high-energy nitrogen ions to the surface of the material and to improve the control of process parameters. In this regard, this study focuses on developing a biomaterial by eliminating both Cr and Ni contents. New biomaterials are directed towards stainless steel without a nickel to reduce the toxic properties of AISI 316L stainless steel. The biomaterial compounds made are also expected to be more robust and corrosion-resistant compared to pure metal. In the study, several tests such as microstructure, surface hardness, and corrosion have been realized to evaluate the surface characteristics of Fe–10Al–25Mn alloy after plasma nitriding. The nitride alloys were characterized using a spectrometer, scanning electron microscopy (SEM) combined with energy dispersion spectroscopy (EDS), micro-Vickers hardness and corrosion testing.

2. Materials and methods

2.1. *Materials and samples preparation*

The Fe–10Al–25Mn alloy smelting process was carried out using a high-frequency induction chamber with a capacity of 50 kg. The raw materials used in this study were mild steel scrap, Fe–Mn med C, pure Al, Fe–C. The ingot-shaped Fe–10Al–25Mn alloy casting has a size of 30 m × 30 m × 200 mm. An inductively coupled plasma optical spectrometer was used to examine the chemical compositions of the alloys. Table 1 shows the chemical compositions of the tested specimen.

2.2. *Surface characterization and experiments*

For nitriding, specimens measuring 5 mm × 10 mm × 10 mm were prepared. The reference material was a 2 mm thick AISI 316L stainless steel plate cut into 10 mm × 10 mm squares. Furthermore, the specimen surfaces were smoothed by sandpaper up to 2000 mesh and cleaned by an ultrasonic cleaner using a polishing machine. Plasma nitriding equipment consists of a metal vacuum vessel with an emptiness

Table 1 – The chemical compositions of Fe–10Al–25Mn alloy.

Elements	Fe	Al	Mn	C	P	S	Si	Mo	Cu	Sn	Ti	W
%weight	Balance	10.05	25.05	0.6	0.01	0.01	0.55	0.01	0.12	0.01	0.01	0.01

system, a nitrogen gas input, a 300 1200-V DC high voltage system, and a temperature regulator. The nitriding process was completed in 3 h at 350, 400, 450, 500, and 550 °C with a pressure of 1.8 mbar.

The tests carried out included the composition, microstructures, hardness, and corrosion. The composition test was carried out using Baird FSQ Foundry Spectrovac Spectrometer based on ASTM E2209 standard test. A JEOL type JSM.6360-LA-EDX (JED 2200 series) *Scanning Electron Microscope*-Energy Dispersive X-Ray System were used to examine the microstructures. The mechanical testing was performed by Schmierplan/Libriction plan LA-H-250 RC 16-02/Hardness Tester DIA Testory micro-Vickers method. The Vickers hardness test procedure was based on ASTM E384. Finally, a corrosion polarization test was performed using a CMS 100 Gamry Instrument to quantify the corrosion rate. The ASTM G5 standard was used to determine the polarization potential.

2.3. Data analyses

The results of microstructure tests were qualitatively evaluated based on various temperatures of plasma nitriding. The comparison of shapes, patterns, sizes, and types of microstructures were interpreted in the analyses. Quantitatively, graphs were created to present the effects of hardness and corrosion. The current research data were also analyzed considering the viewpoint of the findings data by other researchers for more advanced analyses.

3. Results and discussion

The Fe–10Al–25Mn alloy has been experimentally investigated to evaluate its potency for biomaterials. The plasma nitriding was realized to give treatments in the surface material. The microstructure change of the surface alloy plays an important role to impact its hardness and corrosion rate. The main results and the advanced discussion are presented as follow.

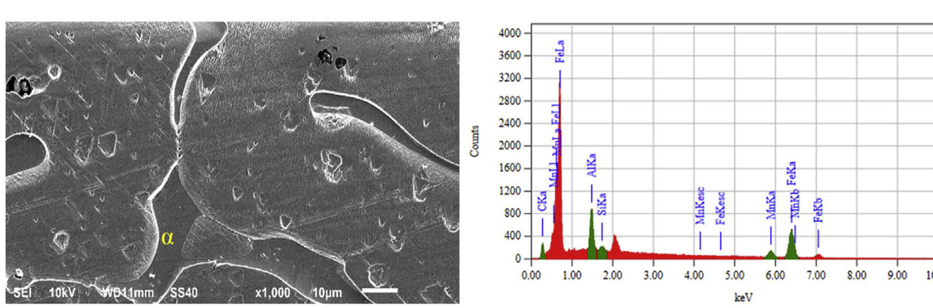
3.1. Microstructures

Fig. 1 gives the SEM micrograph and EDS spot analysis of Fe–10Al–25Mn alloy before plasma nitriding. It is found in Fig. 1(a) that the Fe–10Al–25Mn as-cast alloy has austenite, ferrite, and kappa structure. The structure of austenite tends to be dominant because of the element Mn as an austenite stabilizer. The ferrite structure is related to the Al element as a ferrite stabilizer, while the kappa phase is associated with the relatively high C content, as found by another researcher [20]. As addressed by Chen et al. [21], Mn is dissolved in the Fe system as a solid solution with a disordered FCC structure. The presence of the Al atom in the system changes the disordered FCC structure to an ordered FCC, and the C atom causes the formation of the κ (Fe, Mn)$_3$AlC phase [22]. The κ phase is seen around the α/γ duplex system (see Fig. 2).

Based on the magnification of the SEM micrograph in Fig. 2, both the austenite structure and the κ form lamellas. It is similar to the findings of another research [21]. Thus, the aluminum content of 7.5% is a ferrite phase stabilizer, and 20% manganese is an austenite stabilizer, and a high enough C content encourages the kappa phase formation.

Austenite remains stable at low Al and high C compositions, while κ-carbide remains stable at high C and high Al compositions. Thus, in austenite, κ-carbide precipitation is part of the dispersion of both C and Al. It is in line with the research findings by Kim et al. [23]. Based on the measurement of EDS composition, the Fe–10Al–25Mn alloy contains no nitrogen, as shown in Fig. 1(b).

Fig. 3(a) depicts the SEM test results on the nitride cross-section of the Fe–10Al–25Mn by plasma nitriding process. As previously found by Manfridini et al. [24], the nitride layer consists of γ–Fe(N), Fe_4N and AlN compounds. Fe_4N tends to be dark, whereas AlN is bright. The dominant austenite phase in the Fe–10Al–25Mn alloy encourages the formation of the AlN nitride. This result is similar to that of a study carried out by Chen [25]. The nitride layer on the transverse surface of the Fe–10Al–25Mn alloy produced by plasma nitriding at a temperature of 350 °C is unclear. The higher the nitriding



(a) SEM micrograph (b) EDS

Fig. 1 – The microstructure of Fe–10Al–25Mn alloy.

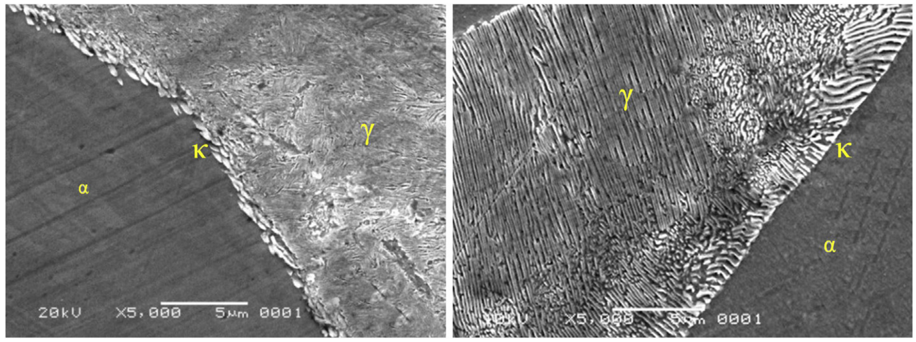


Fig. 2 – The SEM micrograph Fe–10Al–25Mn alloy high magnification.

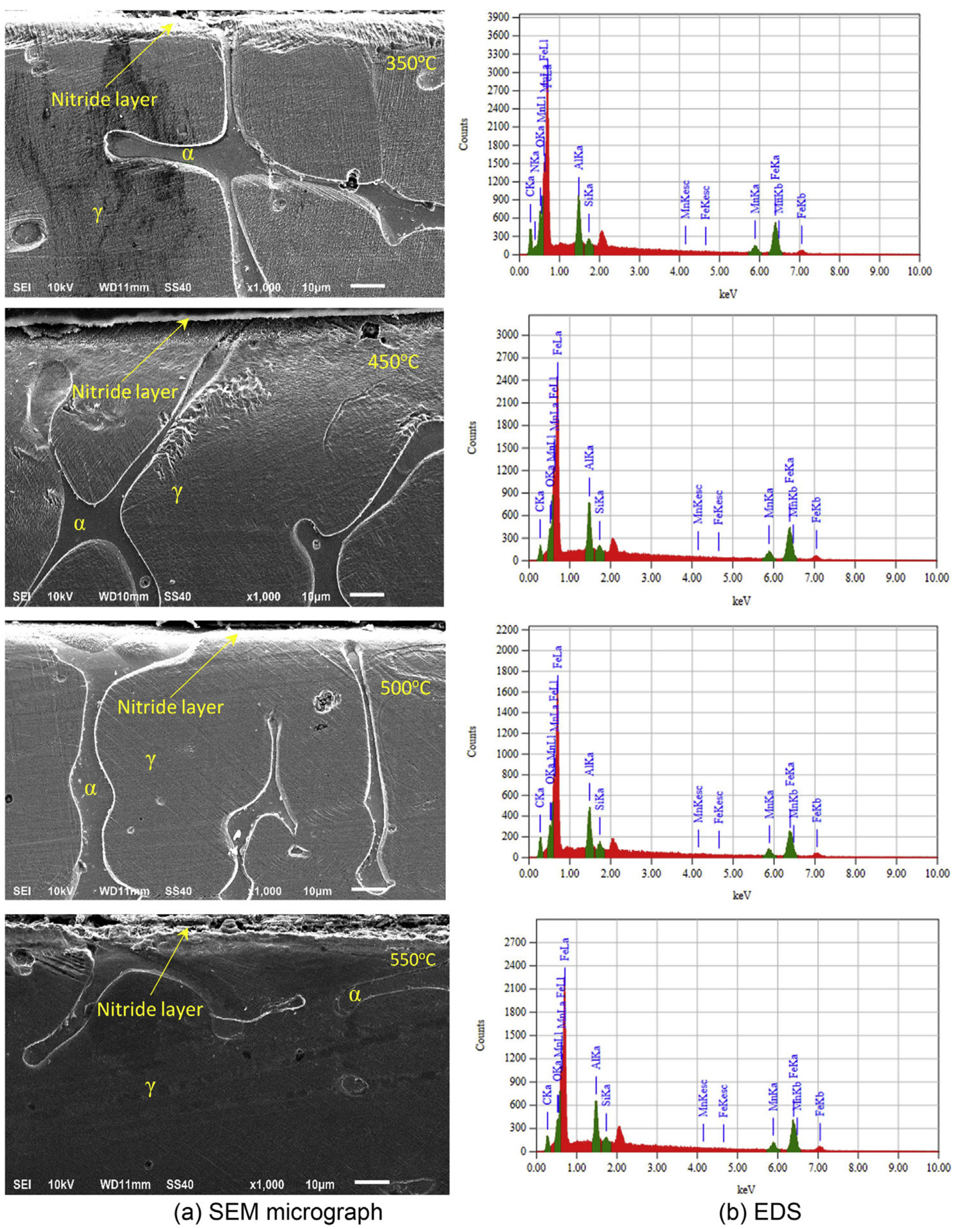


Fig. 3 – The microstructure of Fe–10Al–25Mn nitride alloy.

temperature, the thicker the nitride layer. This finding denotes that the higher the plasma nitriding temperature, the more nitrogen diffuses the Fe–10Al–25Mn alloy surface, forming a nitride compound.

Fig. 3(b) provides the EDS test results on the surface of the Fe–10Al–25Mn alloy after the nitriding process. There is a thin layer of iron nitride on the Fe–10Al–25Mn alloy surface and a percentage of nitrogen deposited. The nitrogen content on the surface of the Fe–10Al–25Mn alloy is sensitive to the plasma nitriding temperature. The distance between the atoms of the Fe–10Al–25Mn alloy specimen becomes increasingly tenuous with the rise of the plasma nitriding temperature. It is due to nitrogen atoms diffuse more easily into the Fe crystal system. The increased nitriding temperature also causes the atoms to vibrate in a position of instability. This causes it more straightforward for nitrogen atoms to enter and diffuse between the atoms making up the Fe–10Al–25Mn alloy. The nitrogen atom then binds with Fe to form the intermetallic compound Fe_3N and Fe_4N, as Chen found [1]. When the nitrogen atom meets Al, it creates the intermetallic AlN compound.

Fig. 4 displays N content on the surface of the Fe–10Al–25Mn alloy after nitriding treatment at various temperatures between 350 and 550 °C. The percentage of nitrogen atoms increases from 350 to 500 °C and declines after 550 °C. This decrease is related to the nitriding temperature, proportional to the depth of nitrogen atoms in the specimen. The distance between the particles in the sample stretches as the nitriding temperature increases at 500 °C. Thus, It is easier for nitrogen atoms to diffuse onto the surface of the specimen to form a layer of iron and aluminum nitride. The distance between the atoms would be even greater if the nitriding temperature is increased to 550 °C. The percentage of nitrogen atoms on the specimen surface decreases as the specimen surface diffuses deeper below the cut surface. The nitriding process at 350–550 °C yields in α'–Fe(N) with a percentage of nitrogen atoms up to 19%, whereas at 19–21%, nitrogen levels cause the formation of Fe_4N iron nitride phase. The amount of nitrogen atoms deposited on the specimen surface significantly impacts the percentage of the Fe_4N phase formed in its region. This finding agrees well with another research [24].

3.2. *Surface hardness*

Fig. 5 indicates the surface hardness test results of the Fe–10Al–25Mn alloy after plasma nitriding. The surface hardness of the Fe–10Al–25Mn alloy after plasma nitriding at 350 °C is 445.6 VHN. The higher the plasma nitriding temperature, the more hardness increases until it reaches a maximum of 680.3 VHN at 500 °C. The hardness decreases up to 30% after attaining a peak point at a temperature of 550 °C. It is relevant to the EDS test results, where the percentage of nitrogen atoms reaches a maximum at 500 °C and then decreases significantly. The nitride phase formed on the surface also has a powerful effect on the surface hardness of the Fe–10Al–25Mn alloy after plasma nitriding. Treatment temperature up to 450 °C causes the material surface of the Fe–10Al–25Mn alloy nitride phase to be α'–Fe(N). The surface changes to Fe_4N when the temperature is 500 °C. Unsurprisingly, Meka et al. found something similar from the results of their study [26].

Fig. 6 shows the hardness distribution test results of the Fe–10Al–25Mn alloy after the plasma nitriding process. At all plasma nitriding temperatures, the deeper the hardness decreases. At a distance of 10 μm from the surface, the decrease in hardness is not significant, only around 2.5%. This finding reveals that nitrogen atoms diffuse quickly up to a distance of 50 μm and form nitride compounds. However, there is a significant decrease in hardness at a distance of 100 and 150 μm. It means that the nitrogen atom experiences a substantial energy reduction. It can be related to the collisions with particles on the surface to reduce its penetration depth. At a distance of 250 μm, the hardness is equivalent to a Fe–10Al–25Mn alloy that does not undergo nitriding. Nitrogen atoms show no more diffusion in the penetration of N in Fe–N up to 70 μm, as found by other studies [24,26].

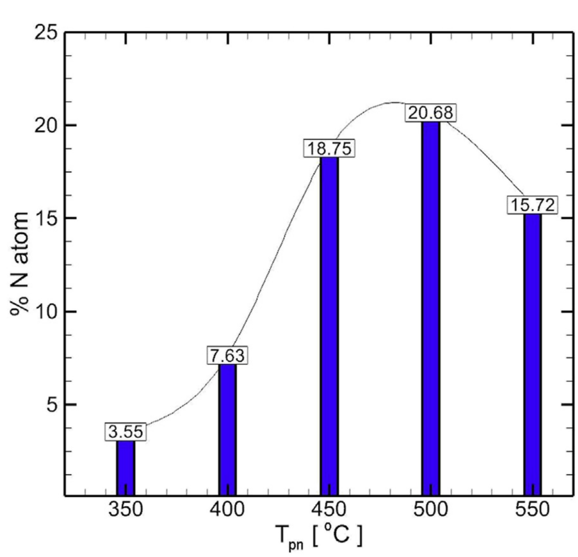


Fig. 4 – The N content on the surface of the Fe–10Al–25Mn Mn alloy.

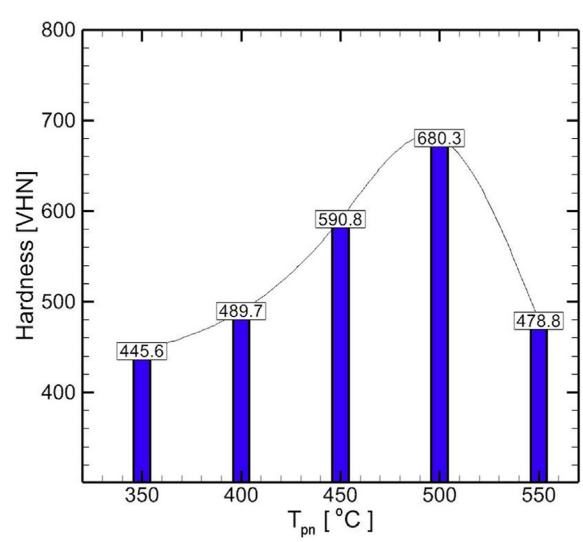


Fig. 5 – The surface hardness of Fe–10Al–25Mn alloy at various nitriding temperatures.

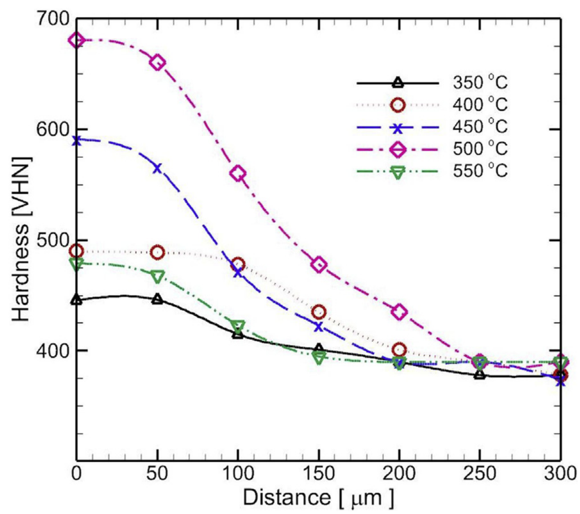


Fig. 6 – The cross-surface hardness distribution of Fe–10Al–25Mn alloy after plasma nitriding.

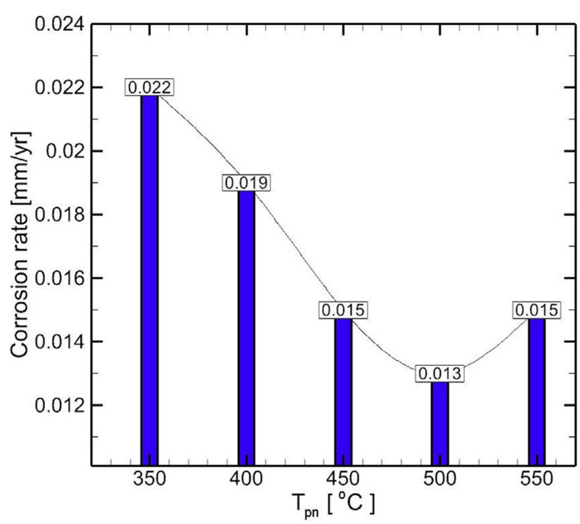


Fig. 7 – The corrosion rate of Fe–10Al–25Mn alloy at various nitriding temperatures.

3.3. *Corrosion rate*

Fig. 7 represents the corrosion rate of the Fe–10Al–25Mn alloy. The calculation of the corrosion rates adopts the formula used by Li et al. [27]. The corrosion resistance increases and reaches the lowest value at 500 °C when plasma nitriding temperature is higher. It means that the corrosion resistance of Fe–10Al–25Mn alloy increases and gets a maximum value at 500 °C. The corrosion resistance of this alloy is due to the formation of nitride on its surface after plasma nitriding, which becomes more apparent, more massive, and thicker as the temperature of the nitride layer rises. Fe_4N, Fe_2N, and AlN compounds make up this nitride layer. Thus, the nitride compound on the alloy surface increases the superficies properties, especially hardness and corrosion resistance. According to Chen et al. [21], corrosion in this nitride layer appears to cross-grain boundaries and take the form of pitting. Another study found that alloying Fe with Mg reduces its corrosion resistance [28]. Mg corrosion is affected by the production of hydroxide (OH^-) and an increase in pH. On the other side, the increase in Al content leads to higher noble corrosion.

4. Conclusion

Investigation of Fe–10Al–25Mn alloy has been carried out experimentally. It can be summarized that plasma nitriding can increase the corrosion resistance of Fe–10Al–25Mn alloy. Higher plasma nitriding temperature increases corrosion resistance and reaches a maximum of 500 °C with γ–Fe(N), Fe_4N and AlN structures on the surface. Plasma nitriding also increases the surface hardness of the alloy. Plasma nitriding at a temperature of 500 °C produces the highest hardness. It means that the newly developed material will be a reliable replacement for traditionary medical stainless steel. This material combines the advantages of a stable austenitic structure and corrosion resistance. Therefore, the Fe–10Al–25Mn nitride alloy can be developed as a prospective biomaterial.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Original Article

Surface characterization of Fe–10Al–25Mn alloy for biomaterial applications

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ABSTRACT

The austenitic stainless-steel biomaterial, AISI 316L stainless steel, is one of the most widely used for orthopedic or prosthetic implant devices because it is easy to manufacture at a relatively low cost. However, corrosion is a challenging issue related to major alloying compounds with body fluids and wear. Therefore, this study aimed to develop a biomaterial of Fe–Al–Mn alloys by minimizing chromium (Cr) and nickel (Ni) contents. In the research, an attempt has been made to increase the corrosion resistance of Fe–10Al–25Mn alloy using plasma nitriding, which was considered one of the most cost-effective surface treatments. The processes were carried out at various treatment temperatures between 350 and 550 °C, with a pressure of 1.8 mbar in 3 h. Several tests were performed, such as chemical compositions, scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS), hardness, and corrosion. The results indicated that the treatment of plasma nitriding at temperatures up to 500 °C significantly enhances the corrosion resistance and increases the hardness of the alloy. At 500 °C, the percentage of nitrogen atoms reaches a peak and then decreases. It means that the nitride formation process on the alloy surface occurs more massively. The amount of nitrogen deposited on the surface of the Fe–10Al–25Mn alloy, as well as a thin layer of iron nitride, is noticeable in the SEM-EDS test. Furthermore, the formation of phases due to the nitriding temperature significantly impacts the alloy properties.

1. Introduction

In the last decades, the application of metallic biomaterials in the medical sector has been overgrown, such as in orthopedic implant devices and dentures. With material engineering and the development of the latest technology, millions of people seem to have a new spirit in improving their quality of life. The metallic biomaterials play a crucial role in reconstructing the human body in orthopedic surgery and dentistry [1]. Some literature noted that at least three implant materials are broadly used for medical purposes, such as stainless steel [2], titanium (Ti) alloys [3], and cobalt–chromium (Co–Cr) based

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alloys [4]. Because of its high strength, dependable wear and corrosion resistance [5], and antibacterial properties [6], the Co–Cr alloy-based material is considerably used in medical implants and dental prosthetics. The suitability of the biological system of the human body is an essential consideration in developing biomaterials. Therefore, materials should not cause harmful effects and not damage the tissues of the human body. It must be free from toxic products and not trigger allergies, inflammation, or cancer [7]. Furthermore, biomaterials require clinical testing to ensure their use [8,9].

Austenitic stainless steel, AISI 316L, is extensively used for orthopedic and prosthetic implant devices. This material has reliable mechanical characteristics at an affordable price. The manufacturing process of this material is also relatively easy compared to others [10–12]. The main advantages of SS-AISI-316L are inexpensive, stable mechanical properties and easy manufacturing process. However, problems with medical stainless steel have been discovered in recent decades of clinical use. Firstly, the stainless steel used in medical applications commonly has a higher elastic modulus than bone. This incompatibility of strength or modulus can result in a stress shielding effect, which can impede bone healing. Secondly, stainless steel is considered less able to withstand wet conditions in a fluid body environment, which has a greater potency in corrosion and wear. This could lead to premature fracture and massive corrosion of the implanted device, followed by releasing harmful substances to the human body. A study reported that both nickel (Ni) and chromium (Cr) are among the potential hazards in medical stainless steels [13]. Allergic contact dermatitis affected by Ni is the most common type of metal hypersensitivity reaction. Furthermore, high Cr levels in the human body may cause cancer and other diseases [1].

It is well known that the liquid contains about 0.9% salt in the human body, a pH of 7.4, and a temperature of about 37 °C [14]. Therefore, biomaterials naturally interact continuously with body fluids. This interaction certainly affects the metals and alloys attached to the human body. Naturally, the complexity of the human body with a wet environment triggers corrosion of almost all metal-based biomaterials, which is followed by chemical and electrochemical degradation. Even the most corrosion-resistant materials are difficult to escape from this natural process. Therefore, biomaterials must be biocompatible and non-stimulating by the body systems, non-toxic, and withstand repeated loads in an aggressive body environment [15]. Besides, biomaterials must have physical and mechanical properties that are reliable to replace body systems, be easily formed and produced at a relatively low cost [4].

Recently, high manganese (Mn) austenitic steels with nitrogen alloys have improved strength, toughness, corrosion resistance, and nonmagnetic properties, making them a viable replacement for traditional Cr–Ni stainless steels in orthopedic implants and other medical applications. The development of manganese-based alloys has been reported for bio-absorbable implants [16]. A metal vapor vacuum is used to implant the biocompatible manganese (Mn) compound into the biomedical Mg surface. This is subjected to evaluate the impact of Mn ion cultivation on the corrosion phenomenon of biomedical Mg. This study found that the surface roughness could be reduced by Mn ion implantation. In a recent study using Fe–Mn–C alloys, the increase of Mn contents reduced the mechanical resistance [17]. Another study found that the addition of Ca to Fe–Mn–Si alloys could improve the osteoinduction and osteoconduction processes better than Fe–Mn–Si alloys or standard AISI 316L stainless steel. The ability to degrade at higher corrosion rates appears to be more optimal [18]. In addition, the suitability of Fe–35Mn–5Si as a biodegradable implant has been improved by considering its mechanical and corrosion properties [19].

Metallic biomaterials appear to be required for patients to support diseased tissue for as long as necessary. However, the capability of implants to degrade uniformly under various conditions in the human body to avoid cytotoxic effects and inappropriate tissue responses remains a significant concern. It means that the new alloys being developed must be high strength, wear-resistant, corrosion-resistant, antibacterial and non-toxic. One of the most widely used industrial processes is plasma nitriding, including nitrogen absorption by diffusion into the structure of a material. This approach mainly applies to tools and low alloy steels, considering their most inadequate surface treatments costs. The main benefit of plasma nitriding is to increase the mass transfer of molecules and high-energy nitrogen ions to the surface of the material and to improve the control of process parameters. In this regard, this study focuses on developing a biomaterial by eliminating both Cr and Ni contents. New biomaterials are directed towards stainless steel without a nickel to reduce the toxic properties of AISI 316L stainless steel. The biomaterial compounds made are also expected to be more robust and corrosion-resistant compared to pure metal. In the study, several tests such as microstructure, surface hardness, and corrosion have been realized to evaluate the surface characteristics of Fe–10Al–25Mn alloy after plasma nitriding. The nitride alloys were characterized using a spectrometer, scanning electron microscopy (SEM) combined with energy dispersion spectroscopy (EDS), micro-Vickers hardness and corrosion testing.

2. Materials and methods

2.1. Materials and samples preparation

The Fe–10Al–25Mn alloy smelting process was carried out using a high-frequency induction chamber with a capacity of 50 kg. The raw materials used in this study were mild steel scrap, Fe–Mn med C, pure Al, Fe–C. The ingot-shaped Fe–10Al–25Mn alloy casting has a size of 30 m × 30 m × 200 mm. An inductively coupled plasma optical spectrometer was used to examine the chemical compositions of the alloys. Table 1 shows the chemical compositions of the tested specimen.

2.2. Surface characterization and experiments

For nitriding, specimens measuring 5 mm × 10 mm × 10 mm were prepared. The reference material was a 2 mm thick AISI 316L stainless steel plate cut into 10 mm × 10 mm squares. Furthermore, the specimen surfaces were smoothed by sandpaper up to 2000 mesh and cleaned by an ultrasonic cleaner using a polishing machine. Plasma nitriding equipment consists of a metal vacuum vessel with an emptiness

Table 1 – The chemical compositions of Fe–10Al–25Mn alloy.

Elements	Fe	Al	Mn	C	P	S	Si	Mo	Cu	Sn	Ti	W
%weight	Balance	10.05	25.05	0.6	0.01	0.01	0.55	0.01	0.12	0.01	0.01	0.01

system, a nitrogen gas input, a 300 1200-V DC high voltage system, and a temperature regulator. The nitriding process was completed in 3 h at 350, 400, 450, 500, and 550 °C with a pressure of 1.8 mbar.

The tests carried out included the composition, microstructures, hardness, and corrosion. The composition test was carried out using Baird FSQ Foundry Spectrovac Spectrometer based on ASTM E2209 standard test. A JEOL type JSM.6360-LA-EDX (JED 2200 series) *Scanning Electron Microscope*-Energy Dispersive X-Ray System were used to examine the microstructures. The mechanical testing was performed by Schmierplan/Libriction plan LA-H-250 RC 16-02/Hardness Tester DIA Testory micro-Vickers method. The Vickers hardness test procedure was based on ASTM E384. Finally, a corrosion polarization test was performed using a CMS 100 Gamry Instrument to quantify the corrosion rate. The ASTM G5 standard was used to determine the polarization potential.

2.3. *Data analyses*

The results of microstructure tests were qualitatively evaluated based on various temperatures of plasma nitriding. The comparison of shapes, patterns, sizes, and types of microstructures were interpreted in the analyses. Quantitatively, graphs were created to present the effects of hardness and corrosion. The current research data were also analyzed considering the viewpoint of the findings data by other researchers for more advanced analyses.

3. Results and discussion

The Fe–10Al–25Mn alloy has been experimentally investigated to evaluate its potency for biomaterials. The plasma nitriding was realized to give treatments in the surface material. The microstructure change of the surface alloy plays an important role to impact its hardness and corrosion rate. The main results and the advanced discussion are presented as follow.

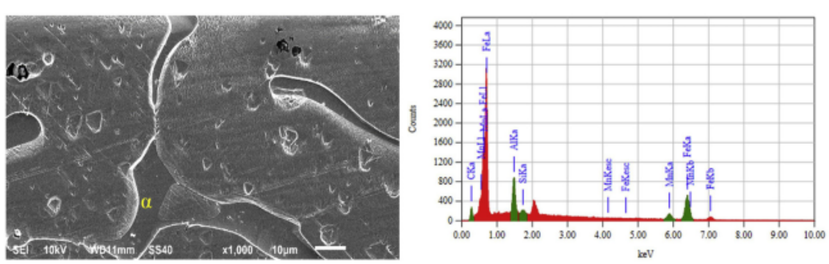
3.1. *Microstructures*

Fig. 1 gives the SEM micrograph and EDS spot analysis of Fe–10Al–25Mn alloy before plasma nitriding. It is found in Fig. 1(a) that the Fe–10Al–25Mn as-cast alloy has austenite, ferrite, and kappa structure. The structure of austenite tends to be dominant because of the element Mn as an austenite stabilizer. The ferrite structure is related to the Al element as a ferrite stabilizer, while the kappa phase is associated with the relatively high C content, as found by another researcher [20]. As addressed by Chen et al. [21], Mn is dissolved in the Fe system as a solid solution with a disordered FCC structure. The presence of the Al atom in the system changes the disordered FCC structure to an ordered FCC, and the C atom causes the formation of the κ (Fe, Mn)$_3$AlC phase [22]. The κ phase is seen around the α/γ duplex system (see Fig. 2).

Based on the magnification of the SEM micrograph in Fig. 2, both the austenite structure and the κ form lamellas. It is similar to the findings of another research [21]. Thus, the aluminum content of 7.5% is a ferrite phase stabilizer, and 20% manganese is an austenite stabilizer, and a high enough C content encourages the kappa phase formation.

Austenite remains stable at low Al and high C compositions, while κ-carbide remains stable at high C and high Al compositions. Thus, in austenite, κ-carbide precipitation is part of the dispersion of both C and Al. It is in line with the research findings by Kim et al. [23]. Based on the measurement of EDS composition, the Fe–10Al–25Mn alloy contains no nitrogen, as shown in Fig. 1(b).

Fig. 3(a) depicts the SEM test results on the nitride cross-section of the Fe–10Al–25Mn by plasma nitriding process. As previously found by Manfridini et al. [24], the nitride layer consists of γ–Fe(N), Fe_4N and AlN compounds. Fe_4N tends to be dark, whereas AlN is bright. The dominant austenite phase in the Fe–10Al–25Mn alloy encourages the formation of the AlN nitride. This result is similar to that of a study carried out by Chen [25]. The nitride layer on the transverse surface of the Fe–10Al–25Mn alloy produced by plasma nitriding at a temperature of 350 °C is unclear. The higher the nitriding



(a) SEM micrograph (b) EDS

Fig. 1 – The microstructure of Fe–10Al–25Mn alloy.

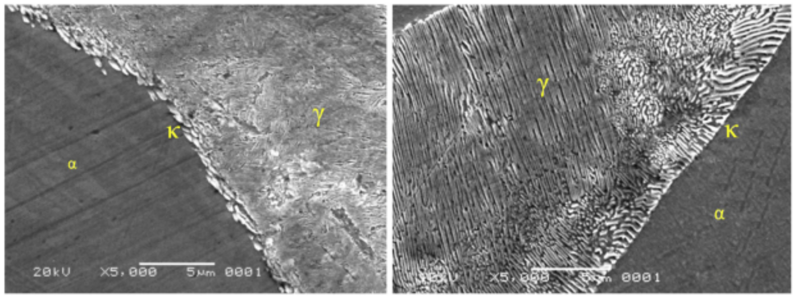


Fig. 2 – The SEM micrograph Fe–10Al–25Mn alloy high magnification.

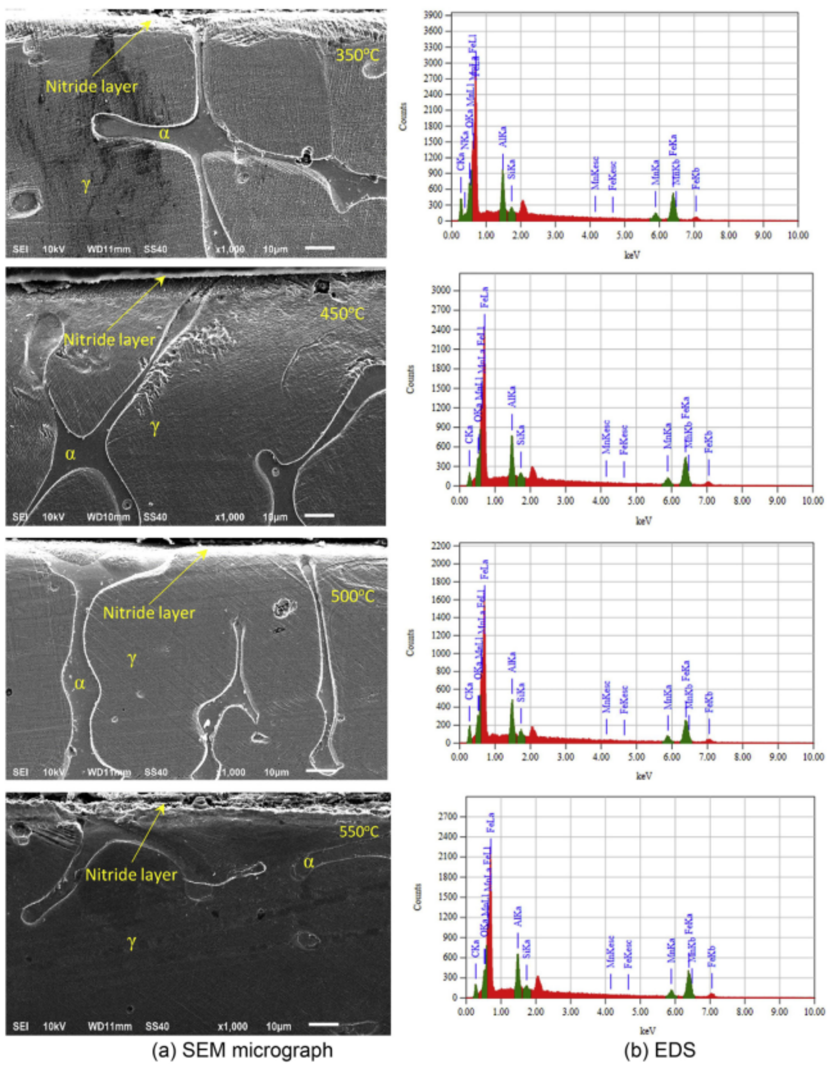


Fig. 3 – The microstructure of Fe–10Al–25Mn nitride alloy.

temperature the thicker the nitride layer. This finding denotes that the higher the plasma nitriding temperature, the more nitrogen diffuses the Fe–10Al–25Mn alloy surface, forming a nitride compound.

Fig. 3(b) provides the EDS test results on the surface of the Fe–10Al–25Mn alloy after the nitriding process. There is a thin layer of iron nitride on the Fe–10Al–25Mn alloy surface and a percentage of nitrogen deposited. The nitrogen content on the surface of the Fe–10Al–25Mn alloy is sensitive to the plasma nitriding temperature. The distance between the atoms of the Fe–10Al–25Mn alloy specimen becomes increasingly tenuous with the rise of the plasma nitriding temperature. It is due to nitrogen atoms diffuse more easily into the Fe crystal system. The increased nitriding temperature also causes the atoms to vibrate in a position of instability. This causes it more straightforward for nitrogen atoms to enter and diffuse between the atoms making up the Fe–10Al–25Mn alloy. The nitrogen atom then binds with Fe to form the intermetallic compound Fe_3N and Fe_4N, as Chen found [1]. When the nitrogen atom meets Al, it creates the intermetallic AlN compound.

Fig. 4 displays N content on the surface of the Fe–10Al–25Mn alloy after nitriding treatment at various temperatures between 350 and 550 °C. The percentage of nitrogen atoms increases from 350 to 500 °C and declines after 550 °C. This decrease is related to the nitriding temperature, proportional to the depth of nitrogen atoms in the specimen. The distance between the particles in the sample stretches as the nitriding temperature increases at 500 °C. Thus, It is easier for nitrogen atoms to diffuse onto the surface of the specimen to form a layer of iron and aluminum nitride. The distance between the atoms would be even greater if the nitriding temperature is increased to 550 °C. The percentage of nitrogen atoms on the specimen surface decreases as the specimen surface diffuses deeper below the cut surface. The nitriding process at 350–550 °C yields in α'–Fe(N) with a percentage of nitrogen atoms up to 11%, whereas at 19–21%, nitrogen levels cause the formation of Fe_4N iron nitride phase. The amount of nitrogen atoms deposited on the specimen surface significantly impacts the percentage of the Fe_4N phase formed in its region. This finding agrees well with another research [24].

3.2. Surface hardness

Fig. 5 indicates the surface hardness test results of the Fe–10Al–25Mn alloy after plasma nitriding. The surface hardness of the Fe–10Al–25Mn alloy after plasma nitriding at 350 °C is 445.6 VHN. The higher the plasma nitriding temperature, the more hardness increases until it reaches a maximum of 680.3 VHN at 500 °C. The hardness decreases up to 30% after attaining a peak point at a temperature of 550 °C. It is relevant to the EDS test results, where the percentage of nitrogen atoms reaches a maximum at 500 °C and then decreases significantly. The nitride phase formed on the surface also has a powerful effect on the surface hardness of the Fe–10Al–25Mn alloy after plasma nitriding. Treatment temperature up to 450 °C causes the material surface of the Fe–10Al–25Mn alloy nitride phase to be α'–Fe(N). The surface changes to Fe_4N when the temperature is 500 °C. Unsurprisingly, Meka et al. found something similar from the results of their study [26].

Fig. 6 shows the hardness distribution test results of the Fe–10Al–25Mn alloy after the plasma nitriding process. At all plasma nitriding temperatures, the deeper the hardness decreases. At a distance of 10 μm from the surface, the decrease in hardness is not significant, only around 2.5%. This finding reveals that nitrogen atoms diffuse quickly up to a distance of 50 μm and form nitride compounds. However, there is a significant decrease in hardness at a distance of 100 and 150 μm. It means that the nitrogen atom experiences a substantial energy reduction. It can be related to the collisions with particles on the surface to reduce its penetration depth. At a distance of 250 μm, the hardness is equivalent to a Fe–10Al–25Mn alloy that does not undergo nitriding. Nitrogen atoms show no more diffusion in the penetration of N in Fe–N up to 70 μm, as found by other studies [24,26].

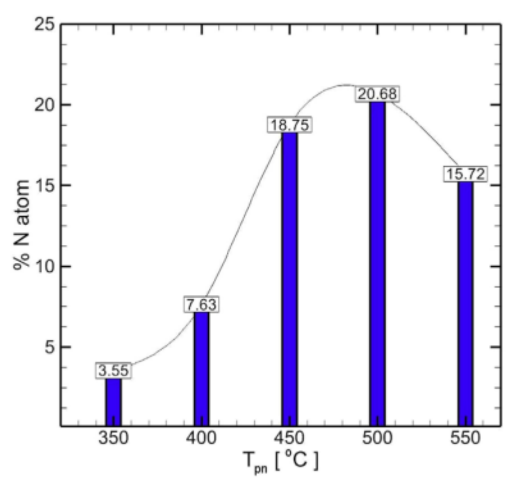


Fig. 4 – The N content on the surface of the Fe–10Al–25Mn Mn alloy.

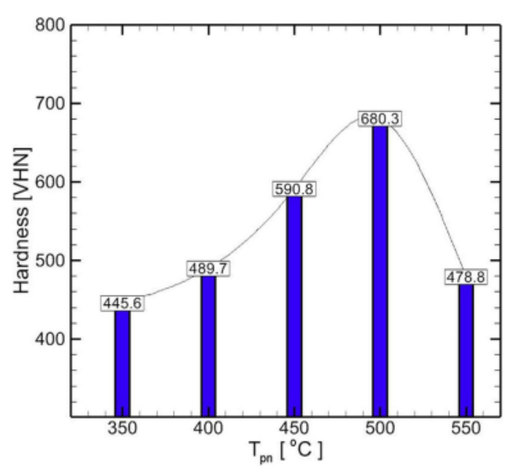


Fig. 5 – The surface hardness of Fe–10Al–25Mn alloy at various nitriding temperatures.

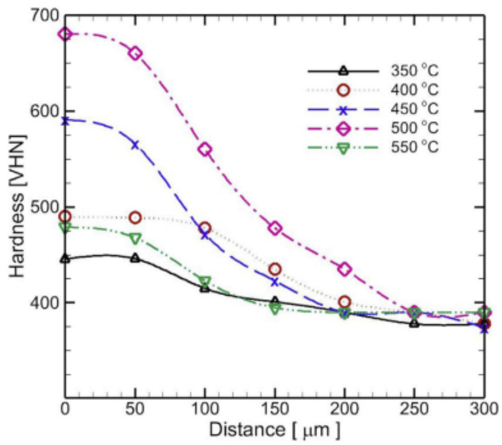


Fig. 6 – The cross-surface hardness distribution of Fe–10Al–25Mn alloy after plasma nitriding.

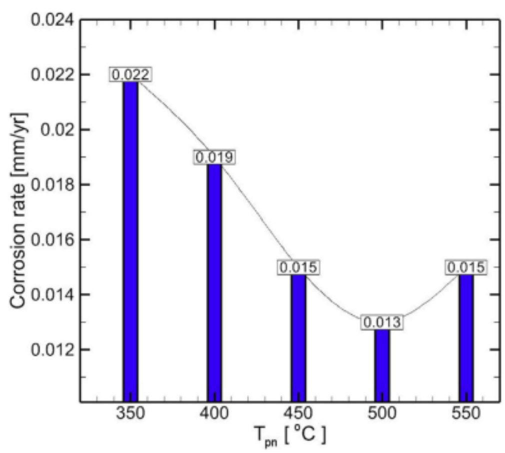


Fig. 7 – The corrosion rate of Fe–10Al–25Mn alloy at various nitriding temperatures.

3.3. *Corrosion rate*

Fig. 7 represents the corrosion rate of the Fe–10Al–25Mn alloy. The calculation of the corrosion rates adopts the formula used by Li et al. [27]. The corrosion resistance increases and reaches the lowest value at 500 °C when plasma nitriding temperature is higher. It means that the corrosion resistance of Fe–10Al–25Mn alloy increases and gets a maximum value at 500 °C. The corrosion resistance of this alloy is due to the formation of nitride on its surface after plasma nitriding, which becomes more apparent, more massive, and thicker as the temperature of the nitride layer rises. Fe_4N, Fe_2N, and AlN compounds make up this nitride layer. Thus, the nitride compound on the alloy surface increases the superficies properties, especially hardness and corrosion resistance. According to Chen et al. [21], corrosion in this nitride layer appears to cross-grain boundaries and take the form of pitting. Another study found that alloying Fe with Mg reduces its corrosion resistance [28]. Mg corrosion is affected by the production of hydroxide (OH^-) and an increase in pH. On the other side, the increase in Al content leads to higher noble corrosion.

4. Conclusion

Investigation of Fe–10Al–25Mn alloy has been carried out experimentally. It can be summarized that plasma nitriding can increase the corrosion resistance of Fe–10Al–25Mn alloy. Higher plasma nitriding temperature increases corrosion resistance and reaches a maximum of 500 °C with γ–Fe(N), Fe_4N and AlN structures on the surface. Plasma nitriding also increases the surface hardness of the alloy. Plasma nitriding at a temperature of 500 °C produces the highest hardness. It means that the newly developed material will be a reliable replacement for traditionary medical stainless steel. This material combines the advantages of a stable austenitic structure and corrosion resistance. Therefore, the Fe–10Al–25Mn nitride alloy can be developed as a prospective biomaterial.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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