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ANALYSIS OF OXIDE INCLUSIONS ON MEDICAL GRADE 316L STAINLESS STEEL USING LOCAL RAW

I Nyoman Jujur[1*], Joni Sah[1], Agustanhakri Bakri[1], Agus Hadi S. Wargadipura[1]

[1] *Center for Materials Technology Agency for the Assessment and Application of Technology (BPPT), Kawasan Puspiptek, Gedung 224, Tangerang Selatan – Banten 15314, Indonesia*

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ABSTRACT

The type of stainless steel that is most commonly used in bone implants is austenitic 316L stainless steel, which has an excellent corrosion resistance and high strength. The Center for Materials Technology, BPPT, in cooperation with a local industry, is currently undertaking research into integrating, refining and alloying processes for the production of medical grade 316L stainless steel, using raw material originating from the ferronickel of Pomalaa. Natural resources of ferronickel, one of the main raw materials for stainless steel, are locally available in Indonesia. Other alloy metals such as steel scrap, ferro chrome and ferro molybdenum are bought in the market. The charging calculation is done by computer-aided simulation, before the melting processes are carried out. The melting facility used is an induction furnace of 250 kg capacity, following the procedures commonly used in the industry. Chemical composition analysis is done by a spectrophotometer. Tensile and hardness tests are conducted, and a microstructure observation is also carried out using an optical microscope and a scanning electron microscope. The selection of raw material inputs and refining and annealing processes affect the quality of the alloy. In our study, we found various forms of oxide inclusions in the stainless steel microstructure: triangular, hexagonal and spherical. The tensile strength of the specimen of 316L stainless steel casting materials was influenced by the presence of oxide phases.

Keywords: Bone implant; Ferronickel; Medical grade 316L stainless steel; Oxide inclusions

1. INTRODUCTION

The increasing need for stainless steel implants in Indonesia requires a great deal of material engineering research and development activity, using local raw materials. The infrastructures and natural resources needed to produce stainless steel are locally available in Indonesia. The country has abundant reserves of nickel ores, which can be processed into ferronickel. The ferronickel industry facility of PT. Aneka Tambang, at Pomalaa, south-east Sulawesi, can be used to melt stainless steel since it has the capability to produce ferronickel with low sulphur and carbon compositions. The type of stainless steel that is most commonly used in bone implants is austenitics 316L stainless steel, which has an excellent corrosion resistance and high strength (Suhendra, 2005). Some local stainless steel casting manufacturers that use induction furnaces are highly dependent on imported raw materials. A common problem encountered in the smelting of stainless steel using induction furnaces is the effect of inclusion of the oxide phase on the mechanical strength and corrosion resistance properties of the casted product,

* Corresponding author's email: nyoman.jujur@bppt.go.id, Tel. +62-21-75791324, Fax. +62-21-75791327
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especially when it is used in medical equipment parts. The oxide phase has been studied by several other authors, looking at various types of oxide phase in austenitic stainless steel. Non-metallic inclusions such as oxides and sulphides, however, can act as initiation points of corrosion attacks and propagate fatigue attacks by corrosive body fluids. In order to address these issues, the oxygen must be controlled (Winters & Nutt, 2003).

In this work, we investigated the formation of oxide inclusion in the microstructure level and how it affects the tensile strength of medical grade 316 stainless steel that is produced by using Pomalaa ferronickel.

2. EXPERIMENTAL SETUP

Table 1 shows the composition of Pomalaa ferronickel; it has a nickel content of 26.79%, with a relatively high carbon content. The charging calculation was done by computer-aided simulation before the melting processes were carried out. Two types of scraps, 2205 stainless steel scrap and 316L stainless steel scrap, were used for making the medical grade 316L stainless steel alloys. Alloy compositions that used the 2205 scrap and the 316L scrap were designated as casting C1 and casting C2, respectively. According to the charging calculation, C1 used 50 kg of the 2205 scrap, 30 kg of the ferronickel, 0.13 kg of the ferro chrome and 2,675 kg of the ferro molybdenum, while C2 used 200 kg of the 316L stainless steel and 20 kg of the Pomalaa ferronickel.

Tabel 1 Chemical composition of Pomalaa ferronickel

C	Si	Mn	P	S	Cr	Ni	Mo	Fe
1.55	0.443	0.065	0.109	0.250	0.55	26.79	0.051	Balance

Synthesis of the 316L stainless steel material consisted of the following processes. First, the steel scraps were melted at a temperature of about 1600°C in a 250 kg induction furnace. After the steel was dissolved homogeneously, the ferronickel, ferro chrome and ferro molybdenum were added until the alloy dissolved. The chemical composition of the melting stainless steel was measured using a spectrophotometer. The alloying process was targeted to meet the chemical composition of the medical grade stainless steels ASTM F 138 (Laing, 1979) and ISO 5832-1, which are used for bone implant applications. Finally, the melting stainless steel alloy was poured into the specimen mold. Preparation of the tensile and hardness specimens were carried out according to the standards of JIS Z 2241; SNI 07-0408 for casting products. Specimens were annealed in the air furnace at a temperature of 1040^{o}C for 45 minutes, and then quenched with water. The rest of the molten liquid metal was used to cast implant products using technology investment casting.

The metal specimen for the microstructural examinations was metallographically prepared by a standard procedure using diamond polishing paste, and then etched by a kalling reagent to reveal grain boundaries. Mechanical tests conducted were the tensile and hardness tests (Vickers), and microstructure observation was also carried out using an optical microscope and a scanning electron microscope (SEM) that was equipped with EDS.

3. RESULTS

The chemical composition requirements for stainless steel bone implants are specified in the ASTM F 138 (Laing, 1979) and ISO 5832-1 specifications. The limits of the chemical composition for bone implants in both the ASTM and ISO standards are nearly identical, with slight differences in maximum silicon and molybdenum content, as seen in Table 2. A reduction in maximum sulphur content, from 0.03% (commercial quality alloy) to 0.01%, has a favorable effect on the volume of sulphide inclusions. A lower phosphorus content provides somewhat better ductility, especially for the majority of surgical implants that are moderately or highly treated with cold work.

Table 2 Chemical requirements (wt %)

	C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	Fe
ASTM F 138	0.03	0.75	2.00	0.025	0.01	17–19	13–15	2.25–3.0	0.1	0.5	Balance
ISO 5832-1	0.03	1.0	2.00	0.025	0.01	17–19	13–15	2.25–3.5	0.1	0.5	Balance

Table 3 compares the chemical compositions of the melting process for C1 and C2, as regards the main elements forming austenitic 316L stainless steel. The content of the main elements for C1 and C2 already meet the standard composition, except that C1 has lower nickel and molybdenum contents than is standard.

Table 3 Chemical composition of 316L stainless steel (wt %)

	C	Si	Mn	P	S	Cr	Ni	Mo	Fe
Casting C1	0.028	0.610	1.20	0.025	0.0039	18.86	10.89	2.13	Balance
Casting C2	0.016	0.321	1.44	0.029	0.0100	18.13	13.59	2.36	Balance

However, as shown in Table 4, the mechanical tensile strength significantly differed between C1 and C2; the final strength of the C1 casting was greater than the value of the ASTM F 138 standard, with a minimum of 490 Mpa. In addition, the yield strength was greater than the ASTM F 138 standard, with a minimum value of 190 Mpa. Elongation of the C1 casting meets the standard value (ASTM > 40), while elongation of the C2 casting does not. The hardness of C1 (139 HV) was higher than C2 (119 HV), but both fulfill the standard value, which is lower than 150 HV.

Table 4 Tensile properties

	Ultimate strength (Mpa)	Yield strength (Mpa)	Elongation
Casting C1	531.16	288.12	42
Casting C2	418.00	215.70	37

The difference in the final strengths of the C1 and C2 casting is allegedly affected by the influence of raw materials and annealing during the manufacturing process. The observation of the castings' microstructures through the optical microscope is shown in Figure 1, where the differences between C1 and C2 can be clearly observed. The grain size of C1 appears smaller than that of C2, and the surface looks very clean, but C2's microstructure shows a larger grain size, and it can also be observed that there are black spots spread evenly in the grains or their boundaries. C1's microstructure shows the formation of the delta-ferrite phase. Traditionally, the effects of different alloying elements on phase balance have been quantified by using the nickel and chromium equivalent numbers, the formula for which was proposed by Schaeffler. Based on the calculations of these equivalent numbers, it is indeed appropriate that this phase is delta-ferrite (Schaeffler, 1949). The differences in grain size between C1 and C2 is suspected to have been caused by the formation of the delta ferrite phase on C1.

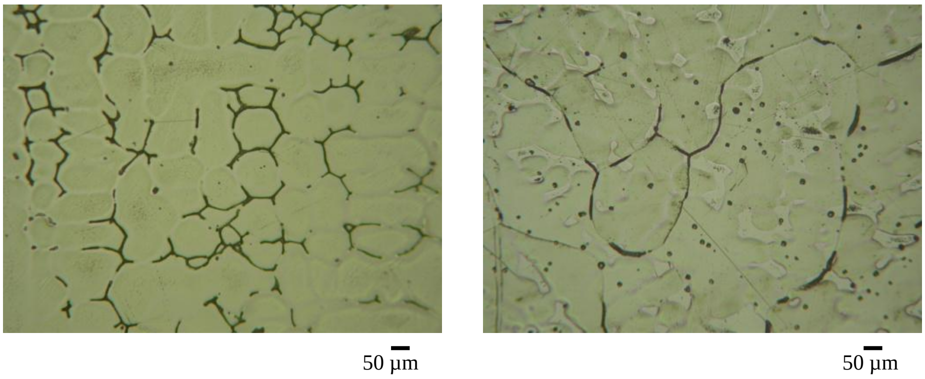
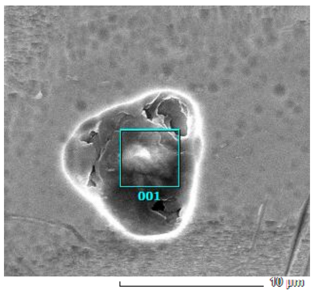
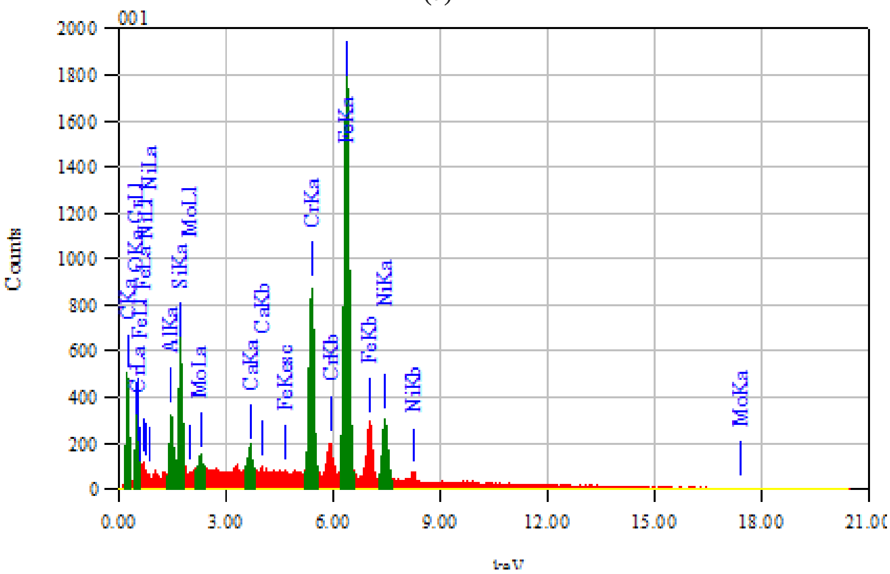


Figure 1 Optical photograph of the C1 and C2 casting specimens

The phase microstructure of the C2 casting, in accordance with SEM-EDAX observations, is shown in Figure 2. The carbide's triangular shape is in accordance with Hong's observations (Hongans et al., 2003), which stated that carbides are triangular. The peak which is evident in this phase is Fe, O, C, Al, Si, Cr and Ni. Based on the proportions of the various elements, we can see that the compositions of O, Al, Ca and Si are significant in amount, which indicates the occurrence of oxide inclusions.



(a)

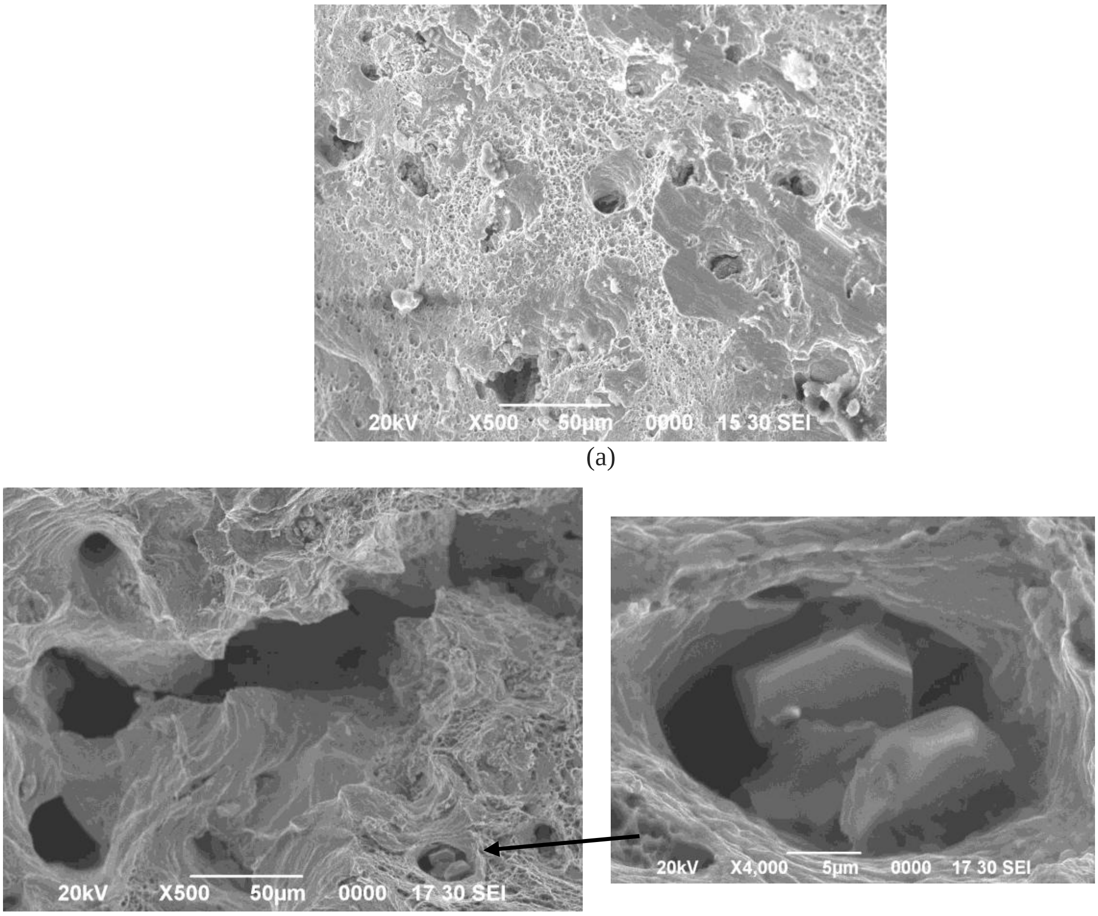


(b)

Figure 2 SEM-EDAX result of oxide phase: (a) oxide phase; (b) XRF of oxide phase

Various forms of oxide inclusions and their chemical compositions have been detected; for example, hexagonal, triangular and spherical. According to Li, a few Al_2O_3-MgO-MnO complex inclusions were observed (Gang et al., 2011).

Fracture photographs show cracked flows passing through several voids in the fracture surface, as seen in Figure 3b. The oxide phase can be seen inside the void, where the void diameter is about 20–30 µm (Figure 3c).



(a)

(b) (c)

Figure 3 Fractography of stainless steel 316 L: (a) microvoids; (b) crack at microvoids; and (c) oxide phase at microvoids

Figure 4 shows the distribution of spherically shaped oxide inclusions in fracture photographs of the C2 casting.

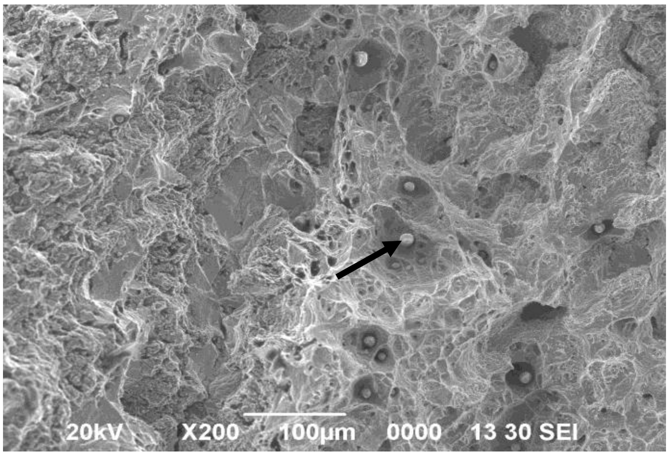


Figure 4 Fractography of 316L stainless steel: the distribution of spherically shaped oxide inclusions

4. DISCUSSION

Godec observed that such inclusions are combinations of Al_2O_3, SiO_2 and CaO particles (Godec, 2011). The results of the current work show similar findings to those obtained by Godec. Most of the inclusions observed by the SEM-EDAX were spherical, and from the XRD analysis, we can conclude that the oxide inclusions are complex phases; further observation of this type of oxide phase is needed.

The tensile strength of the specimen from the C2 casting is lower than that from the C1 casting, as it is influenced by the presence of oxide phases. This condition is supported by the analysis of C2's fracture surface; as shown in Figure 3, oxide phases and voids exacerbate the decrease in tensile strength. The oxide phases are always surrounded by a wall that is so clean as to indicate that voids exist around the oxide phases. The mechanism of void formation needs to be further investigated. It is postulated that voids are formed due to excessive diffusion of elements into the oxide phase. Our results are similar to those of Godec, who found that these inclusions degrade the mechanical properties of the stainless steel, and thereby reduce the ductility of the cast metal; additionally, this increases the risk of mechanical failure and/or corrosion of the final product (Godec, 2011).

In order to improve the quality of 316L stainless steel manufacturing, the formation of inclusions must be controlled through a de-oxidation process. Adding Al and Ca / Si is the best way of reducing the oxygen, increasing the stainless steel's cleanness (Ahmadi et al., 2006) and improving its strength through a change in the distribution of pores (Durowoju et al., 2012).

5. CONCLUSION

In this researh, we investigated the medical grade 316L stainless steel casting implant product, using local Pomalaa ferronickel. Complex inclusions of Al_2O_3, SiO_2 and CaO were found from the SEM-EDAX microstructure analysis. Triangular, hexagonal and spherical oxide inclusion structures were also found. From the tensile strength measurement, it was found that strength values are influenced by oxide inclusions. Therefore, oxide inclusions in the medical grade 316L stainless steel casting product must be controlled.

6. ACKNOWLEDGEMENT

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