AUSTENITIC TYPE STAINLESS STEEL PRODUCTION BY FOUNDRY

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ABSTRACT

AUSTENITIC TYPE STAINLESS STEEL PRODUCTION BY FOUNDRY. Synthesis of austenite stainless steel using extracted minerals from Indonesian mines has been carried out. The starting materials for austenite alloy consist of granular ferro-scrap, nickel, ferro-chrome, ferro-manganese, and ferro-silicon. A small quantity of titanium has been added to it, and an extremely low carbon content is maintained in the alloy. However before the actual alloying work starts, the first important step is to carry out the determination of the fractional amount of each starting material necessary to form an austenite stainless steel alloy as specified. Once the componential fraction of each base alloy-element is determined, the raw materials are weighed on the microbalance. After the fractional quantities of each constituent have been computed, an appropriate amount of these base materials are weighed separately on the microscale. The raw materials are then placed in the induction foundry furnace, which is operated by an electromagnetic inductive-thermal system. The foundry furnace system performs the stirring of the molten materials automatically. The homogenized molten metals are poured down into sand casting prepared in advance. Some of the austenite stainless steel was given heat treatment, followed by preliminary characterization. The microstructure observation concludes that an extensive portion of the sample's surface turns out to be homogenous and that the grain boundaries appear to be well-defined. X-ray diffraction analysis shows that the material belongs to the fcc crystallographic system, which fits in with the austenite class of the alloy. The Vickers scale hardness distribution in the prehomogenized austenite stainless steel is relative high. The experimental fractional elemental composition data acquired by OES method turn out to differ slightly from the theoretical assumption.

Keywords: Stainless Steel, Austenite, synthesis, mining materials.

***ABSTRAK**:*

***PEMBUATAN BAJA TAHAN KARAT JENIS AUSTENIT DENGAN PENGECORAN**. Telah dibuat baja austenitik tahan karat dari bahan-bahan tambang yang digali di Indonesia; Bahan-bahan tambang tersebut berupa ferro scrap, ferro chrome, ferro mangan, ferro silicon dan ferro nikel yang kesemuanya dalam bentuk granular-granular. Austenitic yang dibuat ini juga diberi sedikit titan, serta memiliki kandungan karbon yang sangat rendah. Pembuatan dimulai dengan menghitung porsi bahan-bahan tersebut dari data-data spesifikasi yang diberikan, agar spesifikasi komposisi feritik yang dibuat sesuai dengan yang dikehendaki. Setelah kuantitas dari setiap bahan mentahnya ditemukan, maka dilakukan penimbangan. Pekerjaan dilanjutkan dengan memasukkan bahan-bahan tersebut kedalam foundry furnace induksi yang memiliki sistem pemanasan induksi elektromagnet. Setelah bahan-bahan tersebut mencair, maka pengadukan dilakukan secara otomatis dari sistem foundry furnace tersebut. Kemudian cairan baja yang telah homogen tersebut*

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dituang kedalam cetakan pasir. Pada sebagian baja tahan karat austenitik tersebut diberi perlakukan panas, dan dilakukan karakterisasi awal. Pengamatan struktur mikro menunjukkan bahwa bahan relatif homogen secara luas dan batas butirnya terlihat jelas. Pola difraksinya menunjukkan bahwa bahan memiliki struktur kristal fcc yang sesuai dengan baja jenis austenitik Distribusi kekerasan dalam skala Vickers ingot sebelum homogenisasi relatif tinggi. Pengamatan distribusi unsur dilakukan dengan alat optical emision spectrometry (OES) dan hasilnya agak berbeda dengan design spesifikasi yang dikehendaki.

***Kata kunci**: Baja Tahan Karat**,** Austenit, pembuatan, pengecoran*

INTRODUCTION

Austenite alloys have been synthesized in previous experiments by using powder metallurgy technique [1,2,3] . In this work, the synthesis is continued by preparation of the austenite alloys for high temperature application, but using the foundry technique instead. This way one would readily obtain samples having the same composition as the powder samples but larger in size. It is a well known fact that the physical size of powder austenites are quite small, and so these samples are sometimes difficult to characterize due to their limited physical size. Therefore to perform additional characterization on these samples, samples with larger size are needed, and here is where the foundry method comes in. In this work natural granular minerals having very well known composition are used as starting materials in the synthesis of stainless austenite samples. By evading the use of costly powder samples, a more economical way of alloying is thus achieved. The starting materials for austenite alloy consist of ferro-scrap, ferro-silicon, ferro-manganese, ferro-chrome and ferro-nickel. A small quantity of titanium has been added to it. However before the actual alloying work starts, the first important step is to carry out the determination of the fractional amount of each starting material necessary to form an austenite stainless steel alloy as specified. Therefore an accurate information on the average chemical constitution of these natural minerals must be available. This information should be available and could be obtained from the minerals′ supplier upon request. Once the fractional quantities of each constituent have been computed, an appropriate amount of these base materials are weighed separately on the microscale. Finally, the methodically gained fractional components are handed over to technician in the workshop to be alloyed. The alloying process actually is not just one step, but made up of several steps. A mould made of silica-sand is first constructed by mixing a bentonite binder with a little water. The finished mould must now be lined-up with a certain choice of material depending on the appropriate acid-, base- or neutral environment. A neutral type lining is standard requirement if the desired product is stainless alloy. As a final step, the lining wall is now sintered. The preliminary steps outlined above must be followed before alloying could start. The obtained final product is stainless austenite alloy in ingot form. The austenite ingot is homogenized at 1200 ^{o}C in order to rinse out the existing carbides and to stimulate grain-growth that will cause softening in the austenite. The final step is to trim the finished alloy by grinding and machining. Preliminary characterization on the finished austenite product would constitute most of the next steps in this work. To obtain conclusive result of the austenite samples' mechanical and structural properties several measurements are carried out such as microstructure investigation, x-ray diffraction to investigate the crystal structure, pre-homogenization hardness distribution measurement and elemental composition in

the bulk using optical emission spectrometry (OES). The results are as follows: micrograms show that the alloy's surface is relatively homogenous to a wide extent and its grain-boundaries appear somewhat diffuse. The x-ray diffraction pattern of the austenite alloy confirms its fcc crystalline structure. Hardness tests produce relatively high values of hardness distribution (Vickers scale) in this pre-homogenized stainless ingot. Elemental distribution observation using the OES instrument shows a slightly different result in quantity compared to the desired specification.

The primary raw materials used to alloy the austenite stainless steel are granular ferro scrap, nickel, ferro-chrome, ferro-manganese, and ferro-silicon; these minerals have been extracted from domestic mines; this situation has created an economical advantage because there is no need to purchase expensive imports, since much cheaper alternatives are available domestically. The main raw materials specifications are listed in Table 1 below

MATERIALS AND METHODS

1,Rawmaterials

Table 1: Specification of the raw materials used to build the austenite stainless steel (w %)

	Fe	Ni	Cr	Mn	Si	C	Al	S	P
Fe scrap (LC)	99.17		-	0.5	0.3	0.03	-	-	-
Ni	-	100.0	-	-	-	-	-	-	-
FeCr (LC)	28.486		70.46	-	0.94	0.073	-	0.01	0.03
FeMn(MC)	23.044		-	75.0	0.52	1.3	-	0.006	0.13
FeSi (LC)	24.714		-	-	75.0	0.118	0.14	0.023	0.005

2. Casting materials

Casting and lining materials consist of silica sand and bentonit binder, mixed with a little water. The support materials are used containers (of slightly larger size) and pattern material made of wood. Production of stainless steels requires a neutral environment, and this calls for an alumina (Al_2O_3) lining material used with a specific type of a ramming binder.

3.Processing materials

The materials used in the alloying process are feldspar and chalk. Feldspar is used in order to separate slack or impurity from the austenite stainless steel ingots (main material). Chalk functions to impede the flow of oxygen from the atmosphere to prevent oxidation to occur. This way the alloying procedure could proceed unhindered. Unless these steps are carried out, the raw components would simply coagulate and the resulting steel would not have a homogenous composition.

4. Equipment

An ITB made thermal-induction furnace is the main alloying furnace used in this work. This furnace melts the raw materials by vibrations generated via electromagnetic wave. An optical microscope is used to generate optical micrograms; structural studies are accomplished using the Shimadzu X-ray diffractometer XD-610, and an indentation Vickers hardness-tester is available for hardness tests.

Elemental composition profile was obtained with a 1996 Swiss made *optical emission spectrometry* (OES).

5. Methods

After the fractional quantities of each constituent component from the base materials

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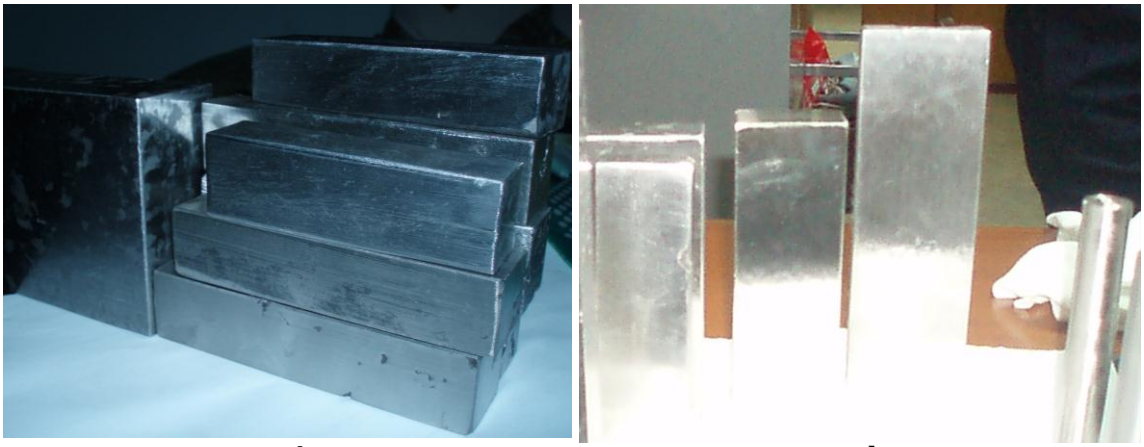
have been computed, an appropriate amount of these base materials are weighed separately on the microscale. Meanwhile, a mould made of silica-sand is simultaneously constructed by mixing a bentonite binder with a little bit water. The finished mould must now be lined-up with a certain choice of material depending on the appropriate acid-, base- or neutral environment. A neutral type lining is standard requirement if the desired product is stainless alloy. As a final step, the lining wall is now sintered. Production of stainless steels requires a neutral environment, and this calls for an alumina (Al_2O_3) lining material used with a specific type of ramming binder.

For the microstructural investigation a standard procedure was followed[4] . The characterization is accomplished using an optical microscope and a SEM microscope with EDS. The crystalline space group is verified by collecting reflection intensities using an x-ray diffractometer, and the hardness testing is carried out using the Vickers indentation method. Optical Emission Spectrometry (OES) equipment is employed in the elemental composition measurement, and the sample is specially prepared by spark erosion method to have a dimension of 2.5 x 2.5 x 12 cm^3.

RESULTS AND DISCUSSION

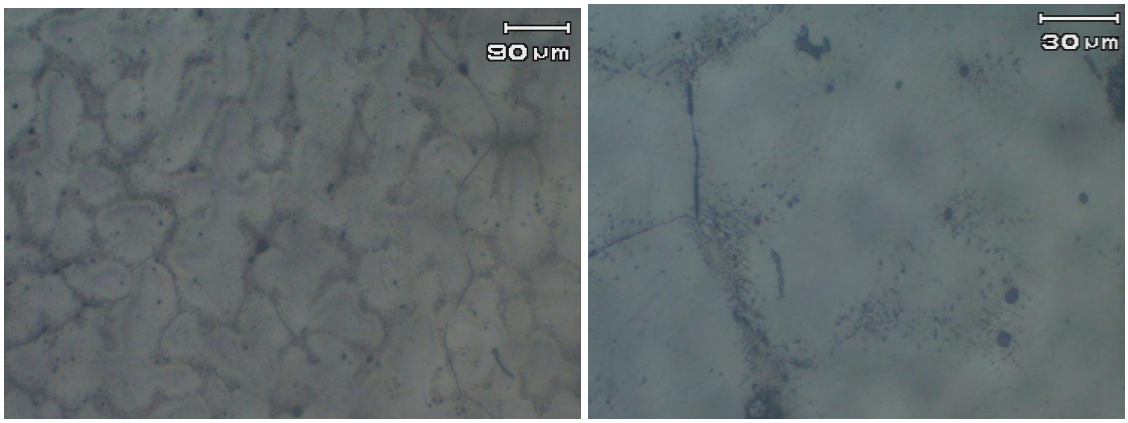
Samples heap foundry result is shown in Fig. 1a, mean while Fig. 1b shows the homogenized samples that underwent hot rolled to comply with a request of ASTM 185, and then cleaned by machining.

Microgram in Figure 2a shows result of micro structural characterization. In the microgram, it is evident that the alloying process does produce a stainless austenite, after etching using a kalling reagent, showing a relatively homogenous surface micro stucture of the dendritic type. Because of the relatively even distribution of the alloy's homogenity, the grain-boundaries appear to be diffuse. A similar microstructure pattern but with a different magnification is shown in figure 2-b. Also here as well as in the dendritic pattern generally, the grain boundaries appear to be diffuse and generally difficult to detect[6,7].



A **b**

Figure 1 a.Austenitic samples heap foundry result. b. homogenized foundry result samples that underwent hot rolled, and then machined



a **b**

Figure. 2. a. Original austenitic optical micro-graph. It seems a equiaxed dendritic pattern has developed in the surface′s microstructure. **b.** Another austenitic optical micrograph, using a different magnification. It seems that in the dendritic pattern the grain boundary in the picture is not well defined.

Normally, foundry-made alloys are characterized by a relatively high hardness value; therefore the hardness should be lowered. This is achieved by the so called normalization method, and if necessary by homogenization process. The purpose of the homogenization method is twofold; the first is to eliminate any carbide which has formed in the sample. The second aim is to grow grains as to soften the sample. This is the reason of why the bulk sample undergoes a homogenization process at 1200 ^{o}C for 20 hours. Post-homogenization optical micrograph of the sample's microstructure is shown in figure 3. In this micrograph, the appearance of the grain-boundaries is very sharp or well-defined. It is possible that this is an indication both the homogenization process and grain growth in the sample have already widened out. The visible black spots or dots are surface porosities.

Fig. 3. Optical micrograph of normalized non standard austenitic sample after homogenization at 1200 ^{o}C for 20 h.

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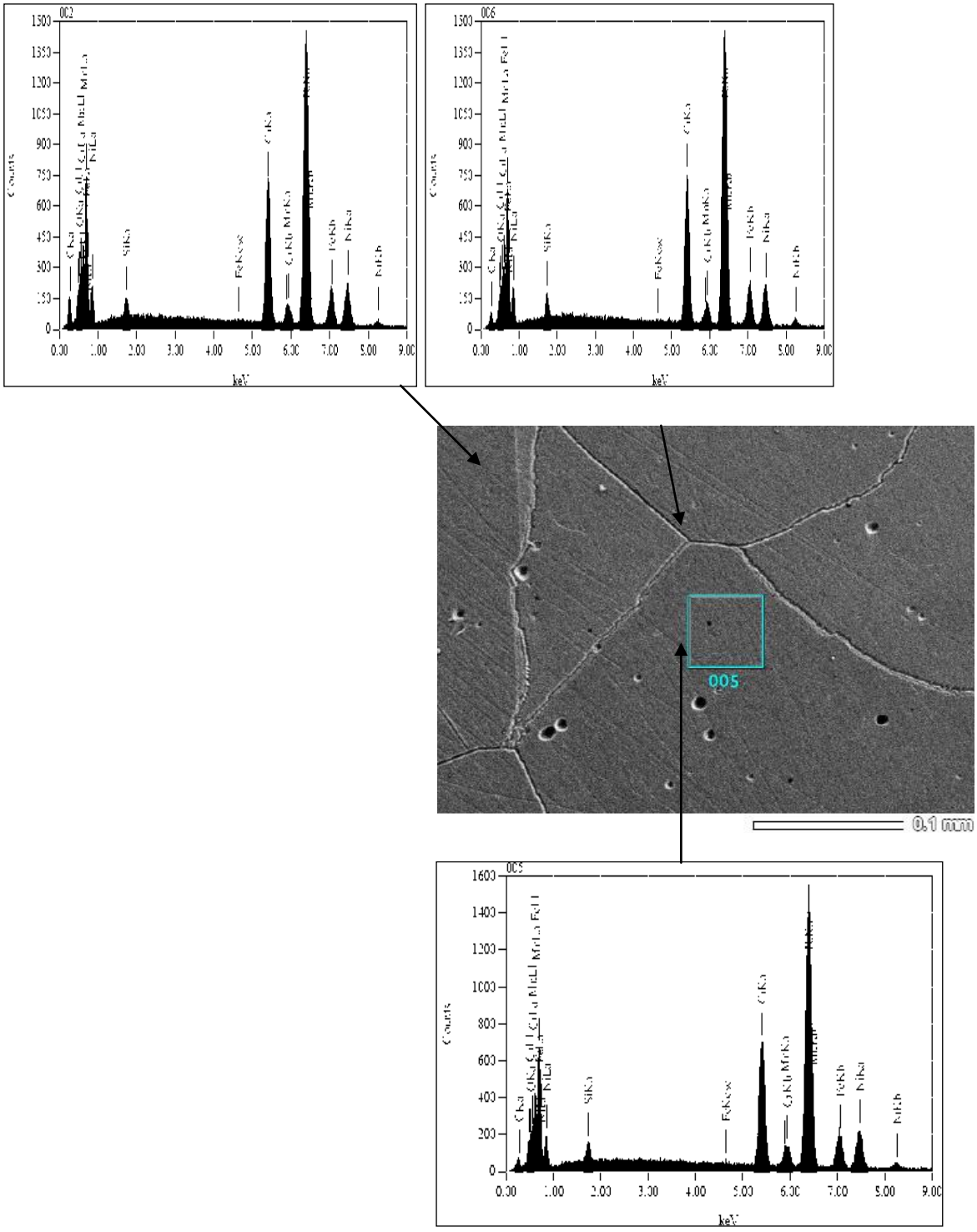

Figure.4. SEM micrograph and the supplemental EDS results: **a**. Microstructure of A1 ingot sample; Here it is clearly shown that the surface is relatively homogenous with a minor part of the surface showing a grey colour covering a narrow area on the left hand side bordered by the irregular line running from top to bottom; b. EDS results from a narrow but long top to from the central region.

SEM-EDS micrograph obtained from microstructure analysis of the normalized (homogenized) foundry processed A1 austenite steels is shown in Fig.-4. It shows a relatively homogenous surface with a few spots and a slight grey part of the surface at the bottom, and no grain boundaries are visible; this means that grain growth has set in the material and the growth has already spread out, causing a decrease in the material hardness and an increase in ductility. EDS results shown in Figure-4.b clearly indicating a close relationship between the samples' condition with respect to the elemental map and the OES (*optical emission spectrometry*) results. Similarly, EDS results shown in Fig.-4.**c** carries a resemblance with EDS result shown in Fig.-4.b**,** which generally fits the elemental composition of bulk region; EDS results from the slight grey region shown in Fig.-4.d shows that this particular region contains a larger quantity of carbon (C) could be expected.

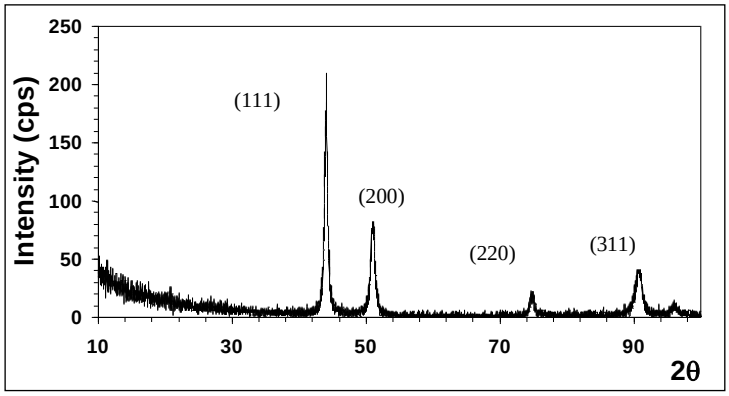

Figure 5. X-ray reflection intensity of the sample code-named A1, produced by foundry method. The reflection peaks fit the fcc crystallographic model confirming the sample's austenite class.

In Fig. 5, the X-ray reflection intensity of the cast stainless austenite is presented. A Cu-Kα target wavelength is used. Indexing of the X-ray reflection peaks is made possible by using an estimated value of lattice-constant obtained by calculation from a similar sample prepared by powder metallurgy method and the aid of the fcc-crystallographic model[1]. The observed reflection peaks indices do sustain the previously held fcc crystallographic system assumption, therefore verifying the austenite character of this sample.

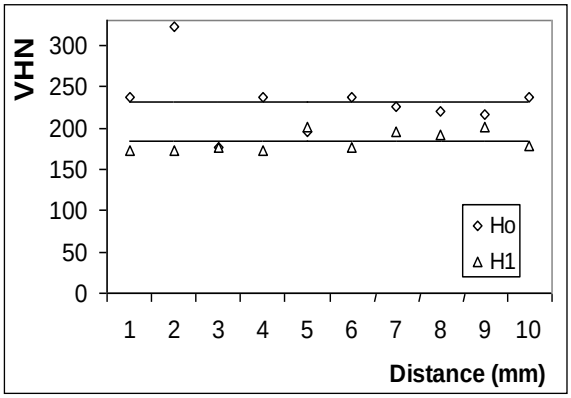

Figure. 6. Ingot Vickers hardness number; the lines shows the average material hardness value respectively; Ho and H1 shows material hardness before and after homogenization treatment, respectively

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In figure 6, the results of Vickers hardness tests on both the original pre-homogenized and 'normalized' homogenized foundry-alloyed samples are shown; in this experiment, indentation is carried out at ten separate points with that of equidistantly spaced (1.0 mm) distribution. The average value of the observed hardness is 230 on the Vickers scale for original casting sample; this means that the material's hardness is still high and needs to be lowered to ″normal″ value by homogenization method, it is expected that with decreasing hardness, the ductility would also increase. In figure-4 the average hardness value of ′normalized′ austenite stainless-steels is also shown to be in the range of 180 on the Vickers scale. Whereas the average hardness value of austenite stainless-steels is in the range 130 - 190 on the Vickers scale [5].

The Optical Emission Spectrometry (OES) instrument operating on the basis of the spark erosion method has been utilized to assess the elemental composition of the stainless austenite. The experimental results are presented in Table-2. The first row contains the requested or the computed theoretical or wanted composition, the second provide information on the OES observed elemental composition. Elements such as V, Sn and P contained in the second row, are unwanted elements and are considered as impurities in the synthesized sample. This could be explained from the fact, that the raw-materials' specification do mention that those impurities are present in the materials.

Tabel 2. Comparison of the computed wanted elemental composition and the actual OES experimental composition in foundry-cast austenite stainless steel in weight percent (w. %).

Composition	Fe	Ni	Cr	Si	Mn	C	Ti	V	Sn	P
wanted / designed	55.34	20.0	21.0	1.5	2.0	$\leq$ 0.08	0.08	-	-	-
OES results	55.98	18.32	23.46	2.55	0.075	0.073	0.003	0.037	0.006	0.002

The results presented in Table-2 show that the computed and the experimental OES elemental composition values quantitatively differ from each other. Firstly, this is caused by the basic (raw) material itself. The raw materials (chemicals) used in this work are original minerals acquired from Indonesian mines, and in this case the specific elemental composition information is provided by the supplier. Although this specific information is reliable, some criticism is due, mainly because these minerals are mined directly from the interior of the earth the specific information supplied is mainly statistical in nature. Therefore it is understandable that any specific information so provided, contains some deviations or standard deviations from the actual data. So it is understood, that academically some of the elemental composition values are actually either higher or lower than those values indicated in the spec-sheet. The second factor is attributed to the limitation in the instrumental accuracy. Also, the basic operational characteristic of the OES which is based upon the spark erosion process is a contributing factor. The third factor comes from the sample itself. The bulk sample does not necessarily have a clean surface, since many additional elements originating from the air outside the sample and considered to be non-ideal external addition, may contaminate the bulky sample during the cooling process. OES based result for example, shows that chrome and silicon are present in the quantity exceeding the specifically designed composition, whereas nickel, manganese and titanium are present in a far lower quantity than the amounts

specified. In particular, the quantity of silicon present could be attributed to statistical deviation from the basic-material′s specification; On the other hand this could also arise from the fact that the tested sample's surface is not sufficiently sterile, for example the presence of silicon carbide in the slag material could significantly affect the amount of silicon in the sample. Other impurities are also present, such as vanadium, phospor and stannum. The presence of negligible amount of unwanted impurities such as vanadium and phosphor is of no serious concern. Stannum itself may even help to improve the mechanical properties of the sample, such as machineability.

CONCLUSION

Based upon the deep and broad analysis of experimental data presented, it could be concluded that the Non Standard austenitic alloy produced in this work fits in with the stainless steel category of alloys. This Non Standard type of alloy has been shown to be both high-temperature – and corrosion resistant, because of its high chrome and nickel contents, with only a small quantity of silicon and manganese. Both the steel′s fcc crystallographic system and its high nickel content indicate that this Non Standard steel is an austenite class steel. The measured hardness data, show that the ingot′s Vickers-scale hardness before homogenization is relatively high. The experimental fractional elemental composition data acquired by OES method turn out to differ slightly from the theoretical assumption.

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