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FERRITIC STAINLESS STEEL SYNTHESIS FROM MINING MATERIALS

A.K. Jahja
PTBIN-BATAN, PPTA Serpong, Kws. PUSPIPTEK, Tangerang 15314, Indonesia
e-mail: ay_khan@yahoo.com

ABSTRACT:

Synthesis of ferritic stainless steel using Indonesian minerals as base or raw materials has been carried out; the mineral base materials are ferro-scrap, ferro-chrome, ferro-manganese and ferro-silicon, all of them are in granular morphology. An additional but minute quantity of titanium is also added as an alloying element, and in addition the formed alloy would have a very low carbon content. The synthesis was started by first calculating the necessary quantity of each base alloying component so that the ferritic composition specification is satisfied. Once the componential fraction of each base alloy-element is determined, the raw materials are weighed on the microbalance. The activity was continued by inserting the raw materials into the induction foundry furnace, which is controlled by an electromagnetic inducto-thermo system. The melting raw materials are then stirred automatically by the foundry furnace system. Finally the homogenized melting mixture was poured down into sand casting. Some of the ferritic stainless steels were ′normalized′ by homogenization at 1200 ^{o}C for 20 hours, and some characterizations was carried out. The microstructure observation shows that the material′s surface is homogenous and the grain boundaries appear to be somewhat diffuse. Analysis of the X-ray reflections shows that the material has a bcc crystal structure. The hardness distribution of this ingot ferritic stainless steel in Vickers scale before homogenization is relative high around of 320; meanwhile the Vickers hardness after homogenization is found to be around 220 VS. The elemental distribution measurement is carried out using the optical emission spectrometry (OES) apparatus and the results differ from the determined specification.

Keywords: *Ferritic Stainless Steel, Synthesis, Mining Materials*

INTRODUCTION

After initial experiments on the synthesis of ferrite materials have been carried out, using powder metallurgy methods [1,2,3], the next activities would involve the synthesis of high-temperature resistant ferrite alloys using foundry methodology, with the same composition as the powder samples but with a larger dimensions. The main argument behind the synthesis of non-corrosive foundry ferrite steels being the dimensions of powder ferrites are quite small and therefore this is a restriction with respect to characterization of these samples. Therefore it is necessary to synthesize larger-sized samples, in order to perform additional characterization on these samples.The stainless ferritic samples are synthesized from natural granular minerals whose composition is very well known. This way the cost of synthesis could be kept at a minimum, because no highly expensive powder materials are involved in the synthesis. This ferrite material is synthesized from ferro-scrap, ferro-chrome, ferro-manganese, ferro-silicon with a minute amount of titanium added to it. Consequently, calculation of the material's balance must first be carried out in accordance with the desired materials' composition. This is only possible if the reliable average elemental composition specifications of the natural minerals to be used are known. Therefore the specific data pertaining to each material must be requested from the suppliers. After the appropriate quantities of the raw components have been determined, the next step is to weigh the necessary amounts using the microbalance, followed by the final step of submitting the weighted materials to the foundry master to be synthesized. The entire alloying process consists of first constructing a silica-sand casting using a bentonite binder and a little water; next, the lining work is carried out using an appropriate material, in an appropriate environment, e.g. acid-, base- or neutral environment. The choice of which depends very much on the type of the alloy material to be made. For stainless alloys this means that a neutral type lining is required. Once the lining process is finished, the lining wall is sintered. The alloying process could be started only after all the due preliminary processes have been completed. The final product is an ingot of stainless ferrite alloy. The ferrite ingot is homogenized at 1200 ^{o}C in order to flush away the existing carbides and to induce the growth of grains which will soften the material. The finishing of the cast ingot is carried out further by grinding and machining. The next stage is performing additional preliminary characterization on the ingots. These include activities 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). 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 ferrite alloy confirms its bcc 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 compared to the desired specification.

MATERIALS AND METHODS

Base materials

The primary base materials used to produce the ferrite stainless steel consist of ferro scrap, ferro-chrome, ferro-manganese, ferro-silicon; all of them are in granular form and all are minerals extracted from domestic mines; this condition is very advantageous, because instead of having to purchase expensive powder chemicals from foreign producers, a much cheaper alternative domestic materials are available. The main base materials specifications are listed in Table-1 below.

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

	Fe	Cr	Mn	Si	C	Al	S	P
Fe scrap (LC)	99.17	-	0.5	0.3	0.03	-	-	-
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

Casting and Lining materials

Casting and lining materials consist of silica sand and bentonit binder, mixed with a little water. The support materials are used cans (of slightly larger size) and pattern material made of wood. Since lining for stainless steels is carried out in a neutral environment, the lining material is chosen to be alumina (Al_2O_3) with a specific type of binder.

Processing materials

The materials used in the alloying process are feldspar and chalk. Feldspar functions as slack remover material, the main task of this material is to remove slack (impurity) from the main material, which is the stainless steel. Chalk is used as air blocker in order to prevent oxygen flow from the air, so oxidation would not occur or at least be reduced. This way alloying could proceed unhindered.

Equipment

The main alloying furnace used in this work is an inducto-thermo furnace made by ITB. The way this furnace works is by melting the raw materials using the material vibrating method via electromagnetic wave until it melts. The optical microgram is obtained using an optical microscope; A Shimadzu x-ray diffractometer XD-610 is available for structural studies, and an indentation Vickers hardness-tester has been utilized also. Elemental composition profile was obtained with a 1996 Swiss made *optical emission spectrometry* (OES).

Methods

The sample preparation for the alloying process starts from the computation of all the materials' component fraction in accordance with the specification of the material to be synthesized. The fractional component is determined by weight, and the quantitative amount of each component is then weighed on the microbalance. Simultaneously, a casting made of sand-silica is prepared using a bentonit binder and a little water. This step is followed by lining the induction furnace according to the chosen condition. In this case, since a stainless steel is the desired end-product (SS), the furnace must be in a neutral condition, and therefore an alumina material (Al_2O_3) is chosen as the lining material.

For the microstructural investigation a standard procedure was followed [4]. The characterization is accomplished using an optical microscope. The crystalline space group is verified using x-ray diffraction method, 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

Result of microstructural characterization is presented in figure 1(a). In the microgram, it is evident that the alloying process does produce a stainless ferrite, after etching using a killing reagent, showing a relatively homogenous surface microstucture. Because of the relatively even distribution of the alloy′s homogeneity, the grain-boundaries appear to be diffuse. With different magnification similar microstructure pattern is shown in figure 1(b). Also here the grain boundaries appear to be diffuse.

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 carbides which have 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 microgram of the sample's microstructure is shown in Figure 2. In this diagram, the appearance of the grain-boundaries is very diffuse or unclear. There is a chance that this is an indication that both the homogenization process and grain growth in the sample have widened.

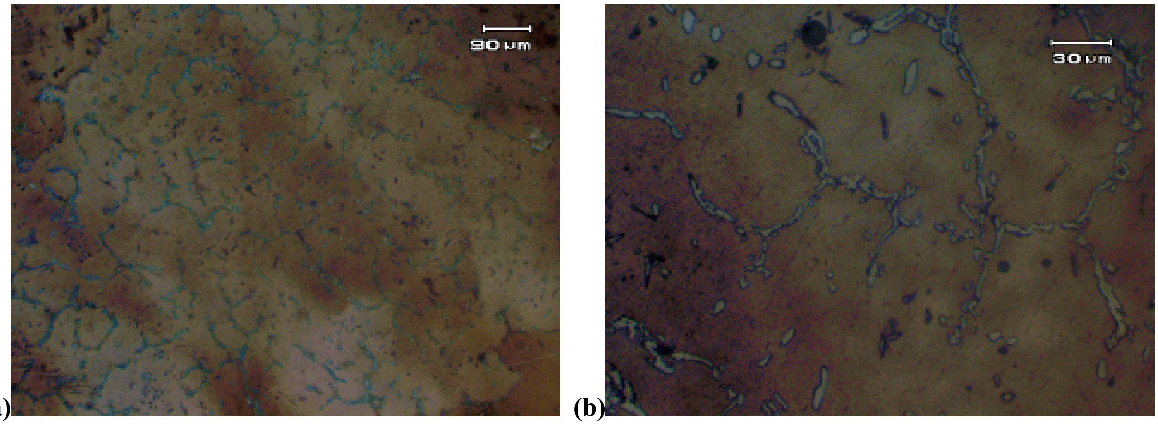
(a) (b)


Figure 1: Optical Micrograms of original ferritic ingot. Sample was made by foundry method.

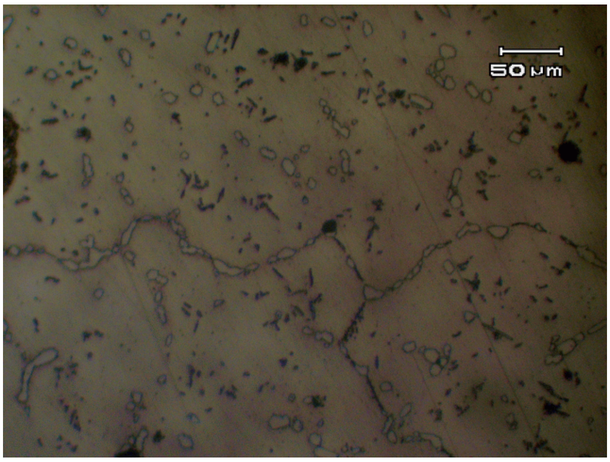


Figure 2: Optical micrgram of new ferritic sampel after homogenization at 1200^{o}C for 20 h.

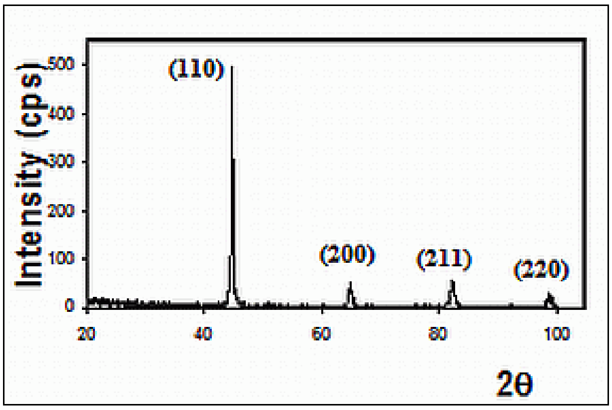


Figure 3: F1 ferritic material X-ray diffraction pattern shows the bcc crystal structure

X-ray diffraction pattern of the cast stainless steel is shown in Figure 3. A Cu-Kα target wavelength is used, and moreover by using an estimated value of lattice-constant obtained by calculation from a similar sample prepared by powder metallurgy method and the help of the bcc-crystallographic model, it becomes possible to index the X-ray reflection peaks. The results strongly indicate that the reflection peaks indices do support the bcc crystallographic system, therefore verifying the assumptions made previously that this sample is a ferrite.

In figure 4, the results of Vickers hardness test on both the original pre-homogenized and 'normalized' homogenized foundry-alloyed samples are shown; in this experiment, indentation is carried out at ten separate and equidistantly spaced (1.0 mm) distribution points. The average value of the observed hardness is 320 on the Vickers scale; 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′ ferrite stainless-steels is also shown to be in the range of 220 on the Vickers scale. Whereas the average hardness value of ferrite stainless-steels is in the range 163-220 on the Vickers scale [6, 7, 8].

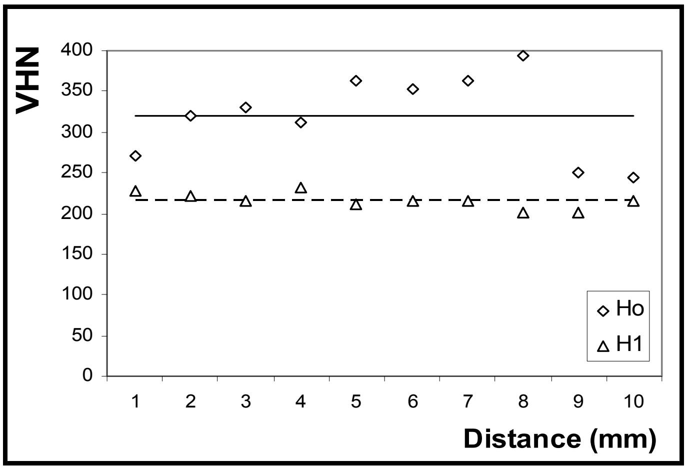


Figure 4: Ingot Vickers hardness number; the lines shows the average material hardness value respectively; Ho and H1 shows material hardness before and after homogenization respectively.

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 ferrite. The experimental results are presented in table-2. The first row contains the requested or the computed theoretical composition, the second provide information on the OES observed elemental composition. Elements such as Ni, Al, S and P contained in the first row, are unwanted elements and are considered to impurities in the synthesized sample. This could be explained from the fact, that the raw-materials' specification do mention those elements to be present in the materials.

Table 2: Comparison of the computed standard elemental composition and the actual OES experimental composition in foundry-cast ferrite stainless steel in percent-weight (w. %).

Composition	Fe	Cr	Si	Mn	C	Ti	Ni	Al	S	P
Standard	75.34	21.0	1.5	2.0	≤ 0.08	0.03	-	-	-	-
OES results	72.65	23.464	2.33	0.911	0.079	0.012	0.09	0.081	0.02	0.02

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 (chemical) 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 both 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. The presence of other impurities, such as aluminum, nickel, sulfur and phosphor should also be taken into account. The presence of small amount of nickel and aluminum is tolerable. It may even improve the mechanical properties of the sample and even increase its corrosion-résistance. The most undesirable impurity is primarily sulfur, since it may cause corrosion and is able to penetrate the bulk directly from the atmosphere by permeation; It is fortunate though that the amount of sulfur is within the tolerated range, or smaller than 0.03 w. % [5].

CONCLUSION

Synthesis of a new ferrite stainless steel with both high temperature- and at once corrosion resistant characteristics has been successfully carried out. This characteristic is brought about by the high chrome- and low nickel, manganese and silicon contents of the sample. The X-ray diffraction pattern confirms the bcc crystal structure of the sample and when combined with OES data which verifies the large amount of chrome present in the sample, it is concluded that this alloy is a ferrite. Experimental hardness data shows that the Vickers-scale (VS) hardness value in the pre-homogenized ingots of this material is quite high at around 320 VS, but has since been successfully lowered by using homogenization method to a value of 220 VS. OES measurements results indicate that the actual quantity of elements present in the sample differs slightly from the standard specifications. Finally, the resulting alloy is described as a non-standard alloy, simply because the fabrication design does not follow any of the available AISI standards.

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