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MICRO AND CRYSTAL STRUCTURE ANALYSIS OF NEW AUSTENITIC STEEL

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ABSTRACT

MICRO AND CRYSTAL STRUCTURE ANALYSIS OF NEW AUSTENITIC STEEL. New austenitic stainless steel code-named A1 has been synthesized using the foundry method. The steel was prepared from the crude minerals mined in Indonesia consisting of ferro scrap, ferro chrome, ferro mangan, and ferro silicon; all of them were in the granular shape. Moreover, a small quantity of titanium was added to this austenitic steel that already has a very low carbon content. The synthesis was started by calculating each raw material quantity from the given specification data with the material balance equation, so that the austenitic composition specification matches the originally conceived specifications. After all of the crude material quantities were determined, each of the crude materials to be used in the alloying process were then weighed separately. The process was continued by inserting the crude materials into an induction foundry furnace that operates on an electromagnetic inducto-thermo system. The stirring was carried out automatically by the system. The now homogenous melting materials were then inserted into the ladle, followed by pouring into the sand casting. Some of the steels was normalized by a homogenization process at 1200 ^{o}C for 20 hours, and additional characterization was carried out afterwards. The microstructure observation shows that the material surface is relatively homogenous but with some porous holes. The X-ray diffraction pattern shows that the material had a fcc crystal structure with lattice parameter of 3.564 Å.

Key words : New Austenitic, Analysis, Microstructure, Crystal structure

ABSTRAK

ANALISIS STRUKTUR MIKRO DAN STRUKTUR KRISTAL BAJA AUSTENITIK BARU. Telah dibuat baja austenitik tahan karat baru yang diberi kode A1 dengan metoda pengecoran. Baja ini dibuat dari bahan-bahan tambang yang digali di Indonesia, yaitu bahan yang terdiri dari *ferro scrap*, *ferro chrome*, *ferro mangan*, *ferro silicon* yang kesemuanya dalam bentuk granular-granular. Austenitik yang dibuat ini juga diberi sedikit titanium, dengan kandungan karbon sangat rendah. Pembuatan dimulai dengan menghitung porsi bahan-bahan tersebut dari data-data spesifikasi yang diberikan dengan persamaan *material balance*, agar spesifikasi komposisi austenitik yang dibuat sesuai dengan yang dikehendaki. Setelah kuantitas dari setiap bahan mentahnya ditentukan, maka dilakukan penimbangan. Pekerjaan dilanjutkan dengan memasukkan bahan-bahan tersebut ke dalam *foundry furnace* induksi yang memiliki sistem pemanasan induksi elektromagnet. Pengadukan dilakukan secara otomatis dari sistem *foundry furnace* tersebut. Kemudian cairan baja yang telah homogen dituang kedalam ledel terus dituang kedalam cetakan pasir. Pada sebagian baja tahan karat austenitik tersebut dinormalisir dengan homogenisasi pada temperatur 1200 ^{o}C selama 20 jam, dan dilakukan karakterisasi awal. Hasil pengamatan struktur mikro menunjukkan bahwa permukaan bahan homogen dengan sedikit porositas. Pola difraksinya menunjukkan bahwa bahan memiliki struktur kristal fcc dengan nilai parameter kisi sekitar 3.564 Å.

Kata kunci : Austenitik baru, Analisis, Strukturmikro, Struktur kristal

INTRODUCTION

The synthesizing of the new austenitic type alloy by powder metallurgy method was effectively and successfully carried out [1,2]. But it is widely as known that the sample material obtained by this procedure was relatively small and so that it posses very limited dimension. This case bring to pass another ensuing problem appears, that is the presence of the restrictiveness in the forming of sample testing; so it will not be able unhampered to form testing sample as well as sample for impact testing and another. For instance smallest standard, sample for impact testing has 6.5 cm long [3]. As another illustration, the sample for neutron

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scattering with the (2 x 2) cm^2 cross section has 4 up to 11 cm long. So that it is needed to make sample material with the relative bigger dimension, in a such away that the test types can also be extended.

So the main purpose of this work is to make the relatively larger samples to overcome the testing restrictiveness in above mentioned.

By foundry as an alternative method the new austenite alloys using non standard certain composition have been synthesized, and these new alloys were designated with the code name of A1 [4]. Initially, the A1 samples were prepared to support the nuclear power plant research activity (abbreviated by PLTN), especially research involving the vessel and heat exchanger materials. Composition wise, this material could find a broader and more general (or multi purpose) application. By coating A1 austenite with ceramic materials, for instance carbide ceramics such as wolfram carbide, silicon carbide or oxide based ceramics such as chrome oxide, silicon oxide etc which turn the A1 alloy into a heat resistant material, the material could also function as a rocket's nose tip and combustion chamber.

In order to implement the raw materials were eligible economy-priced, that were to say mining output gneiss granular. If each of raw materials has definite specification of the ascertainable mixed elements so by certain composition target, the respective quantity of the raw materials can be determined. This paper is a part of the early characterization of the material synthesized.

THEORY

In general, synthesis of materials using the foundry method is not in any way simpler when compared to the powder metallurgy method [5]. However, several advantages could be gained from the foundry method, namely that the dimensions of the materials are larger, and the materials' shape could vary according to the prepared or available moulds, in such a way that even complicated shapes would pose no problem. The foundry method uses an induction furnace, and does not use the ordinary heating furnace which utilizes the supercanthal heat element or the carbide silicon heat element. By full utilization of Indonesian local granular materials will lead to more economical process when compared for instance to another procedure where the starting bulk materials have actually to be imported from abroad, and at the same time this method by passes the sometimes intricate bureaucratic red tape encountered in the process of obtaining import permits**.** The first step started with the preparation of base materials and designing the desired compositions**.**

Therefore it is necessary to calculate the elemental components of the base materials, in order to achieve the desired compositions. Calculation of the component elements is carried out using the materials balance equation. The next step is preparing the mould,

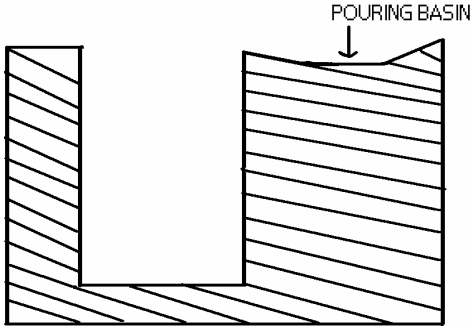

Figure 1. Mould pattern used in casting

which is composed of silicate sand with bentonite as binder or from scorched sand and bentonite binder [6, 7]. The mould pattern diagram is shown in Figure 1. This type of pattern prevents bubble formation in the material during synthesis and the materials extremities do not become brittle as a result.

The next step is to prepare the induction furnace with lining materials. Because the desired end product is qualitatively on a par with stainless steels, beta alumina is chosen as the lining materials. Once the lining procedure is finished, the induction furnace is sintered.

Mould pattern used in casting and all its instrumentation is given a thorough inspection, including the cooling system. The foundry method also needs other processing materials as complements. One of these is feldspar used as slack remover. The main function of the slack remover is to collect and pull all the ensuing impurities out to float upward to the surface to close the steel liquid [7]; another process materials is solid caporite acting as oxygen mask from the air [7]. Because the type of furnace used is of the induction type, the melting process is carried out by setting all the atoms in the molecular parts of the material in vibration using electromagnetic wave, the ensuing tremendous anharmonic vibration in the materials would readily break up the molecular bonds in the materials. To make the process more effective, the minimum amount of the raw or crude materials needed to fill the induction furnace should be at least seventy five percent of the furnace's entire volume capacity. In the synthesis of materials using foundry method, several points need attention. One, evaporation fraction factor of each constituent element needs particular attention, for example chrome's fraction is two percent, nickel is one percent. Therefore this aspect needs to be incalculated in the materials balance calculation.

EXPERIMENTAL METHODS

Materials

In this work, the new alloy code is named A1, constitutes seven main different elements, iron, nickel, chrome, manganese, silicon, carbon, and titanium, each with a specified weight percentage tabulated in Table 1.

Table 1. Targeted composition of material alloying elements in weight percent.

Elements	Ni	Cr	Mn	Si	C	Ti
w. %	20.0	21.0	2.0	1.5	0.08 ≤	0.08

The A1 austenite steels are synthesized from minerals extracted in domestic (Indonesian) mines. The materials consist of ferro scrap, nickel, ferro chrome, ferro mangan, ferro silicon all are in granular shapes. A small amount of titanium is also added to this austenite, which is specially designed with a very low carbon content. The quality of this austenite A1 is expected to be equivalent to that of stainless steels therefore the neutral type lining is chosen in the preparation of austenite A1. Therefore alumina (Al_2O_3) becomes a suitable lining material of choice. All of the raw materials content designed in weight % are listed in Table 1 of the previous paper [1,2]. The etchant materials used in sample preparation is oxalate acid.

Apparatus

The foundry-technique was used in the synthesis process. Furnace for foundry used is *inducto thermo furnace*, thermoline model F47920-26-80, build by Telimex Research Centre in the joint cooperation with Bandung Institute of Technology. The microstructural observations are carried out using optical microscope of the type UFX-DX (Japan), and also scanning electron microscope (SEM) equipped with an EDS. Meanwhile the diffraction intensity pattern was obtained using the Shimadzu X-ray diffractometer XD-610.

Methods

The first step in material design is to determine or to design the composition of the materials in Question [1], the next step is to construct the sand mould. The mould is constructed using the gating system outline, the mould materials are silicate sand or baked sand, with a bentonite binder and a little water with the help of a wooden pattern. The following steps include lining the inductor furnace wall using alumina as base materials, with it certain parts are coated with ramming materials.

Concurrently, raw materials needed in the alloy preparation are being prepared (in accordance with the elemental composition of the raw materials). In the case of austenite, for example, the crude materials in question is prepared in accordance with the precalculated composition of the desired end materials. Because the majority of these materials are not in a pure condition, the elemental quantity of the crude materials is determined using the material balance equation, this is to satisfy the compositional requirements in the design. After each of the elements have been weighed, the process of melting and mixing the different elements could begin. Preparation is carried out at the average temperature of 1600-1700 °C. Alloy preparation is followed by normalization using the homogenization method at 1200 °C for 20 hours. There are two objectives

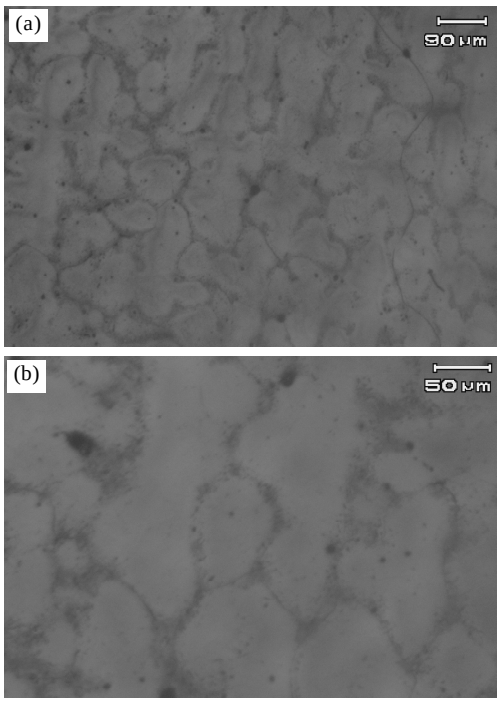

Figure 2. (a). Original austenitic ingot optical microgap. (b). The same austenitic optical micrograph from the same sample with the differrent magnification

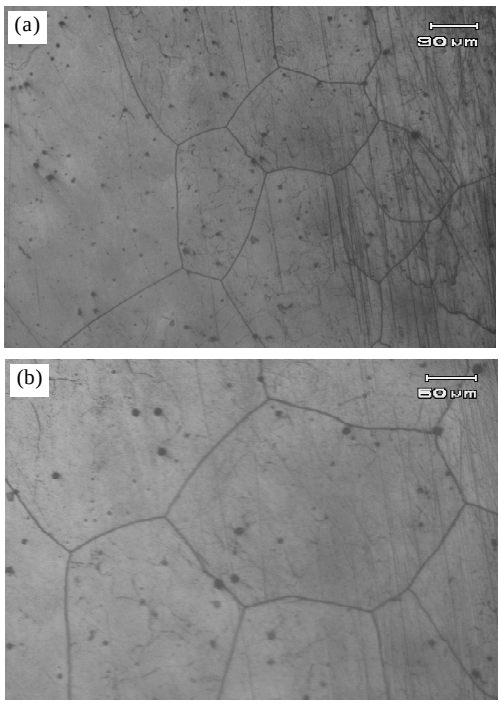

Figure 3. (a) Optical micrograph of new austenitic sampel after homogenization at 1200 °C for 20 h. (b). The same optical micrograph of new austenitic sampel after homogenization at 1200 °C for 20 h, with the differenet magnification.

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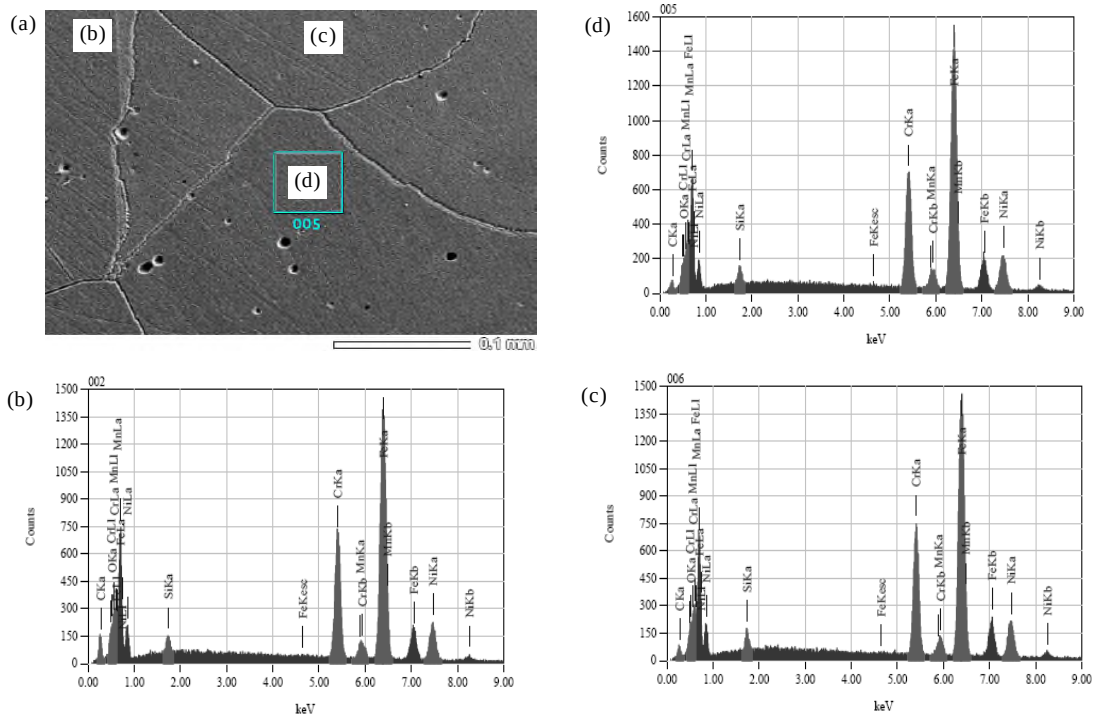

Figure 4. SEM micrograph and the supplemental EDS results: a. Microstructure of A1 ingot sample; Here it is clearly shown that the surface is relatively homogenous with a minor part of the surface showing a grey colour covering a narrow area on the left hand side bordered by the irregular line running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

of homogenization, first it is used to dissolve any formed carbides if any, and the other is to let grains to grow in order to reduce the materials hardness [8,9]. The synthesized A1 austenite stainless steel A1 is then subjected to various examinations, such as optical and SEM/EDS microscopy to obtain structural micrograph of the material; continous and step counting X-Ray diffraction to obtain information on the materials crystal structure analyzed using the Rietveld analysis method.

RESULTS AND DISCUSSION

Ingots of austenite steels obtained from this process show dendrite type microstructure [10, 11], which actually follows the existing cooling cycle pattern, resulting from the temperature pattern on the mould surface. This should be evident in Figure 2(a), recorded using an optical microscope. The grain boundaries are not sharply defined instead a kind of irregular equiaxed pattern is presented caused mainly by a cooling propagation process inside the material. If the micrograph is further enlarged (for example Figure 2(b)) which presents a smooth surface; the visible spots are possibly left over from rinse water trapped in the surface's furrows after the etching process. Other possible explanation would be, that these are dirt materials adhering to the surface after wiping it with unsanitary cloth. Other source could be the tissues used to dry the samples.

The obtained ingots remained to be normalized through homogenization at 1200 ^{o}C for 20 hours, in order to flush away any carbides which could be formed in the process, and also to let the grains grow to reduce the materials hardness.

The optical microstructure of the normalized austenitic ingots is shown in Figure 3(a**)**. In this micrograph, the grain-boundaries are clearly visible. On the right hand side of this micrograph, a few parallel lines resembling scratchs are visible. The small dots are probably porous holes. Figure 3(b**)** is a repeat of the same microgram of the material, but with a different magnification.

SEM/EDS microgram obtained from microstructure analysis of the normalized gray (homogenized) foundry processed A1 austenite steels is shown in Figure 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 Figure 4(**c)** carries a resemblance with EDS result shown in Figure 4(b)**,** which generally fits the elemental composition of bulk region; EDS results from the slight grey region shown in Figure 3(d) show that this

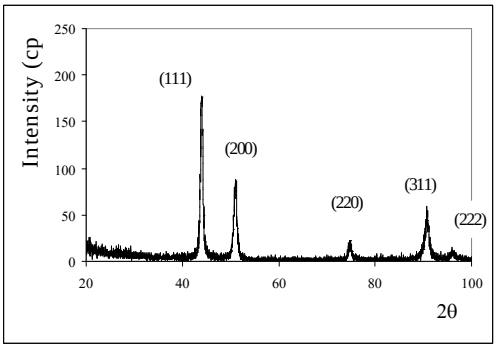

Figure 5. A1 austenitic material X-ray diffraction pattern shows the fcc crystal structure

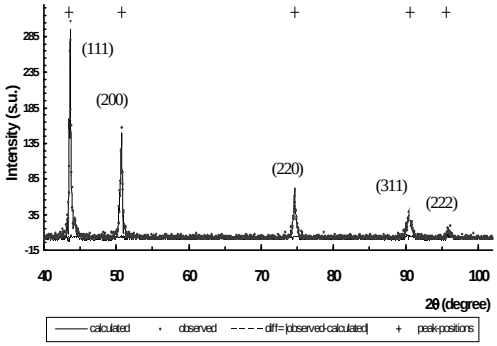

Figure 6. X-ray diffraction pattern as a result of Rietveld analysis; 2θ stands for angel between incidence and scattered rays

particular region contains a larger quantity of carbon (C), therefore in this minor region the formation of carbides, silicon carbide or chrome carbide could be expected.

The X-ray diffraction intensity of the austenite sample is shown in Figure 5. The X-Ray diffraction intensity was measured using a Cu target, and a Cu-Kα wavelength of 1.54 Å. Indexing of the diffraction peaks is carried out based on fcc unit cell pattern. Peak identification and peak position [1,2] allows the lattice parameter to be calculated, and a lattice parameter of 3.564 Å is finally obtained for this new austenite sample.

The Rietveld analysis results of the diffraction intensity pattern from step counting X-Ray measurement are shown in Figure 6, the refinement results are almost equal to the manual analysis′ results mentioned above, and some crystal parameters calculated by Rietan are listed in Table 2.

The good fit of the Rietveld refinement with the experimental diffraction intensity shows that the cubic *Fm3m* space group previously chosen [4] for this sample is correct and therefore confirms the austenitic phase of this self prepared sample. Therefore this research group is successful in developing samples of its own design.

From the resulting pattern, it could be concluded that by a simple manual analysis there is a proven resemblance between the *step counting* pattern and the continous *counting* pattern. The lattice parameters

Table 2. Some of Rietveld analysis results

Rwp	Rp	R_E	R_I	Lattice Par. (Å)	Debye Temp. Factor	Density (gcm^{-3})
17,67	17,03	13,61	20,89	3,564± 2.66687E-03	3.05 x 10^{-5}	8.1

match each other for the first three digits. It can also be compared between the pattern of Figure 4 with the pattern in Figure 3.

CONCLUSIONS

Based upon the deep and broad analysis of experimental data presented, it could be concluded that the new austenitic alloy produced in this work fits in with the stainless steel category of alloys. The phase morphology formed in the samples takes on the form of normal fragmented grains form, and posses a better prospect as a structure material than that of alloys classified in the same class. This new type of alloy has been expected 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 new steel is an austenite class steel. The discussion above also shows that the synthesized new materials given code as A1 is stainless steel austenitic type. The main indicator of the austenite phase is the fcc crystallographic structure with the lattice parameter of 3.564 Å, as verified by X-ray diffraction technique. From the Rietveld analysis also known that the density of this newly synthesized materials is around of 8.1 g cm^{-3}.

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