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MICROSTRUCTURAL EVOLUTION AND COARSENING KINETICS OF B2-(Fe,Ni)Al IN FERRITIC STEEL OF Fe-14Ni-9Al-7.5Cr-1Mo AT HIGH TEMPERATURES

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ABSTRACT

MICROSTRUCTURAL EVOLUTION AND COARSENING KINETICS OF B2-(Fe,Ni)Al IN FERRITIC STEEL OF Fe-14Ni-9Al-7,5Cr-1Mo AT HIGH TEMPERATURES. Increasing the efficiency of power plant facilities, such as steam power plant, is demanding, and this drives to the development of materials capable for higher temperature operations. Ferritic steels have been widely used for boiler components due to their relatively economical and higher in thermal conductivity. Nevertheless, the application of ferritic steels for boilers is limited only at 630 ℃ due to their weakness in creep strength and corrosion at higher temperatures. Efforts to increase the performance of ferritic steels at higher temperature applications have been intensively carried out, one of which is by developing B2-(Fe,Ni)Al precipitates in the ferrite matrix. This paper discusses the microstructural evolution in alloy of Fe-14Ni-9Al-7.5Cr-1Mo (in wt.%) strengthened by B2-(Fe,Ni)Al precipitates during heating at 800, 900, and 1000 ^oC for 6, 20 and 48 hours. Ageing at 800 ^oC for 6 hours gave rounded cuboidal B2-(Fe,Ni)Al precipitates dispersed homogeneously in the ferrite matrix and changed to rounded shape at longer ageing times. At 1000 ^oC, however, the precipitates had rounded shape at all times of ageing. Coarsening of precipitates occurred during ageing at 800 and 1000 ^oC with the rate constants of 5,7 x 10^4 nm^3/h and 2,1 x10^6 nm^3/h respectively, which is considered relatively low. The highest hardness value of 520 HVN was observed for the sample aged at 1000 °C for 6 hours.

Keywords: Ferritic steels, B2-(Fe,Ni)Al precipitate, coarsening, microstructure evolution.

ABSTRAK

EVOLUSI STRUKTURMIKRO DAN KINETIKA PENGASARAN DARI B2-(FE,NI)AL DALAM BAJA FERITIK PADUAN FE-14NI-9AL-7,5CR-1MO PADA TEMPERATUR TINGGI. *Peningkatan efisiensi pada fasilitas pembangkit listrik seperti pada pembangkit listrik tenaga uap, sangat dibutuhkan dan mendorong untuk pengembangan material yang mampu beroperasi pada temparatur tinggi. Baja feritik sudah digunakan secara luas sebagai komponen boiler karena bernilai ekonomis dan konduktivitas panas yang tinggi. Namun demikian, penggunaan baja feritik sebagai boiler terbatas penggunannya pada temperature 630 ^{o}C karena kelemahannya pada kekuatan mulur dan korosi pada temperatur tinggi. Berbagai usaha untuk meningkatkan unjuk kerja baja feritik pada temperature tinggi telah banyak dilakukan, salah satunya dengan mengembangkan presipitat B2-(Fe,Ni)Al pada matriks ferit. Pada tulisan ini dibahas mengenai evolusi strukturmikro pada paduan Fe-14Ni-9Al-7.5Cr-1Mo (dalam wt.%) yang diperkuat presipitat B2-(Fe,Ni)Al yang dipanaskan pada temperatur 800, 900, dan 1000 ^{o}C selama 6, 20 dan 48 jam. Pada temperature pemanasan 800 ^{o}C selama 6 jam terbentuk presipitas B2-(Fe,Ni)Al rounded cuboidal yang secara homogen terdispersi pada matriks feritnya, dan seiring lama proses pemanasannya bentuknya semakin bundar. Pada temperature 1000 ^{o}C, bentuk presipitat yang bundar terbentuk pada semua rentang waktu. Pengasaran presipitat terjadi selama pemanasan 800 dan 1000 ^{o}C dengan laju konstanta secara berturut-turut sebesar 5,7 x 10^4 nm^3/h dan 2,1 x10^6 nm^3/h, dimana perubahan ini relatif lambat. Nilai kekerasan tertinggi sebesar 520 HVN dicapai pada pemanasan 1000°C selama 6 jam.*

Kata kunci: *baja feritik, presipitat B2-(Fe,Ni)Al, pengasaran, evolusi struktur mikro.*

INTRODUCTION

Global population growth has been predicted grows to reach 9.8 billion in 2050 [1]. This triggers the global electric power increases to about 60% from 2015 to 2050 [2,3] and raises the concerns on energy security and global warming. Efforts have been merged to increase the use of renewable non-fossil fuels. However, predictions show that fosil fuels, especially coal based fuels, will still dominate the electricity production at least until 2050 [3]. One of the solutions for this problem is by increasing the effecinecy of the power plants. Based on Carnot cycle, this can be implemented by increasing the temperature operations of the power plant, but this requires materials wistands to be used at higher temperatures. Nickel based superalloys have shown as the most sophisticated materials for high temperature applications, such as in gas turbine engines as well as steam power generators [4]. Such materials contain relatively high nickel contents that make them expensive. Recently, ferritic iron-based superalloys have attracted lots of attentions for the application in advanced supercritical steam power plants. Beside relatively economical, tese materials have better thermal conductivity and thermal expansion compared with that of nickel-based superalloys [5].

In fact, ferritic iron base materials have long been used in conventional steam power plants, which are operated at a maximum of 630 ℃, due to their low in creep and high temperature corrosion resistant [6]. Ferritic steel is also used in nuclear power plant for nuclear fuel cladding. Efforts have been foccued to developed new ferritic alloys capable to be used for temperatures higher than 700 °C. Recent studies investigated the development Fe-Ni-Cr based alloys modified by Mo addition to obtain the precipitation strengthening of B2-ordered (Fe,Ni)Al dispersed homogeneously in the ferrite matrix [7]. These materials have been showed better mechanical properties at higher temperatures compared with that of conventional ferritic alloys [8]. These studies also revealed that basically the strengthening behavior of these materials are similar with that found in nickel based superalloys [9]. Further studies have been conducted to develop Fe-Ni-Cr-Al-Mo alloys by taking the advantage of higher oxidation resistance of ferritic Fe-Cr-Al system and higher mechanical properties at high temperatures of Fe-Ni-Al, still by strengthening of more complex B2-ordered (Fe,Ni)Al [10,11]. In these alloys Mo mostly contributes to the solid solution strengthening of the matrix, but it could also influence the surface energy of matrix-precipitet (B2-α-Fe) interphase, and reduces the coarsening rates of the precipitates [12]. As normally valid in most high temperature alloys, both Al and Cr are expected act as the elements that produce protective scales of Al_2O_3 and Cr_2O_3 or spinels for high temperature oxidation and hot corrosion resistant [11].

In addition to high temperature oxidation and hot corrosion, the strength of all materials operated at high temperatures can decrease due to microstructural changes. These microstructural changes occur as the microstructures are essentially metastable. Heating the alloys at high temperatures activated for the atom to diffuse and leads the microstructural changes. For alloys strengthened by second phase precipitates distributed homogeneously in the matrix, coarsening of precipitates increases the distance between precipitates and consequently reduces the strength or hardness of the materials [12]. Corsening rates can be minimized by reducing the diffusivity of precipitate forming elements in the matrix, decreasing the interface energy between both phases and lowering the solubility of alloying elements in the base metals [13].

Alloys of Fe-Ni-Al-Cr-Mo are projected to be operated in power plants, either as boiler tubes or steam turbine blades, at a maximum temperature of [13,14]. However, during application of these materials, malfunctions might occur that make overheating of the parts. Therefore, the microstructures that occur at higher temperatures are necessary in order to know the microstructural evolutions during overheating and enable to predicting the degradation of the microstructures. This study investigated the microstructural evolution of Fe-14Ni-9Al-7.5Cr-1Mo strengthened by B2-(Fe,Ni)Al at 800, 900 and 1000 ^oC. These temperatures are considered as the overheating temperatures for the advanced superctitical steam power plants.

METHODS

The alloy of Fe-14Ni-9Al-7.5Cr-1Mo (otherwise stated all chemical compositions are in weight percent) was prepared by melting the raw materials of low carbon ferronickel shots obtained from PT. Antam Tbk.containing 20.4%Ni and 78.7%Fe with small amount of Cr and C, pure aluminium ingot of 99.98%Al delivered from PT. Inalum, pure chromium chips of 99.5% from Sigma Aldrich dan pure molybdenum (99.90%) and iron (99.90%) from Merck.

After weighing, the raw materials were melted in a mini DC arc furnace at 230 Volt and 80-110 Ampere. To avoid oxidation, prior during melting the air in the furnace was flushed and purged with high purity argon. To obtain relatively homogeneous alloy, the button was melted four times after turned it up and down. The as melted button was then further homogenized by heating the alloy button in a heat resistant tube furnace at 1100 ^oC for 24 hours. To avoid from oxidation, the furnace was purged with high putrity argon.

The as-homogenized alloy button was then cut into several coupon samples of about 1.5 mm x 5 mm x 7 mm using low speed diamond cutter. Heating simulations were conducted by heating the coupon samples in the similar furnace as used for homogenization at 800, 900 and 1000 ^oC for 6, 20 and 48 hours. To reveal the microstructures, all the samples were etched with chemical solution containing 0,1 ml HF, 3,3 ml CH_3COOH, 3,3 ml HNO_3 and 3,3 ml water. Characterization of the as homogenized sample and the heated samples were carried out using optical microscopes and scanning electron microscope (SEM) at which an energy dispersion spectroscopy (EDS) device was attached. The EDS analysis was used to identify the phase faound in the microstructures. The SEM images were used to obtained the average size of B2-(Fe,Ni)Al precipitates in each sample. Vicker hardness test was then done to measure the hardness of all samples.

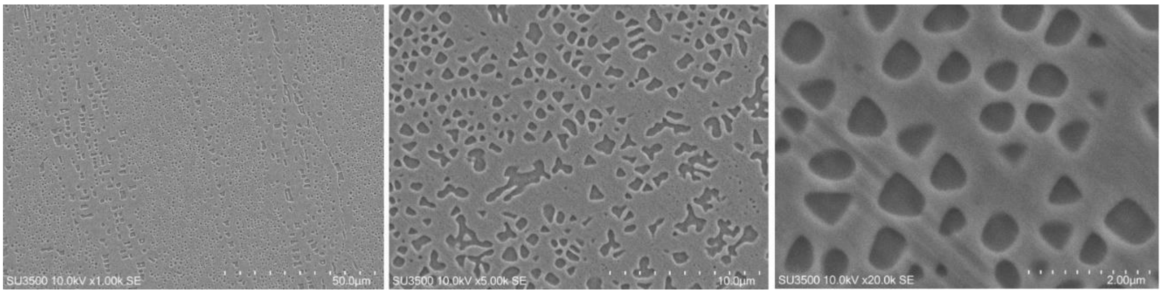
RESULTS AND DISCUSSIONS

Microstructure of As hmogenized Alloy

Images given in Figure 1 show the microstructures of as homogenized Fe-14Ni-9Al-7.5Cr-1Mo at different locations and magnifications. It is clearly seen that the alloy contained B2 precipitates dispersed relatively homogeneously in the matrix ferrite. In the center of the grain, the precipitates mostly are small with equiaxed rounded-cuboid shape, while in the area around grain boundaries they are tend to irregular. A pseudo-terner phase diagram of Fe-Ni-Al with addition of 7.5%Cr and 1%Mo at 1100^oC, as shown in Figure 2, was used to analyse the occurrence of the microstructures. Using this phase diagram as approximation, it is confirmed that based on the chemical composition, the alloy is in the equilibrium phase area for α-Fe and β’.

Addition of Cr in the Fe-Ni-Al alloy system has been reported enlarged the α-Fe area as the temperatures increased, as this element is known as ferrite stabilize element [15]. Therefore, addition of 7.5%Cr in Fe-Ni-Al make the alloy is in α-Fe at 1100 ^oC. Moreover, the addition of Cr into the alloy increases the stability area for β’ phase when the temperatures drop from 1100 ^oC down to

950 ^{o}C, and further decreasing from 850 ^{o}C to 750 ^{o}C. It is predicted that addition of 1% Mo would not significantly change the phase diagram. Precipitates of B2 were formed when the alloy experienced cooling in furnace. It is seen in pseudo-binary of NiAl-Fe, shown in Figure 3 that decreasing temperatures reduces the solubility of Ni and Al in α-Fe, and as consequent, it provides precipitation of B2 in the alloy matrix.



(a) (b) (c)

Figure 1**.** Microstructures of the as-homogenized alloy
(a) in the interior of grains and around grain boundaries
(b) distribution and morphology of B2 precipitates around grain boundaries
(c) distribution and morphology of B2 precipitates in the interior of grain.

The EDS analysis of the as homogenized sample, as shown in Figure 4, confirmed that the precipitates were B2-(Fe,Ni)Al, and known as intermediate phase that has B2 (CsCl) cryatal structure [13]. It is clearly seen that when Cr and Mo are sit in Fe and Ni point lattice, the chemical composition of precipitates are nearly equatomic and represented the chemical composition of B2-(Fe,Ni)Al. Lower aluminium contents were found in the matrix, especially in the area relatively far from the precipitates, as indicated by EDS spot 3.

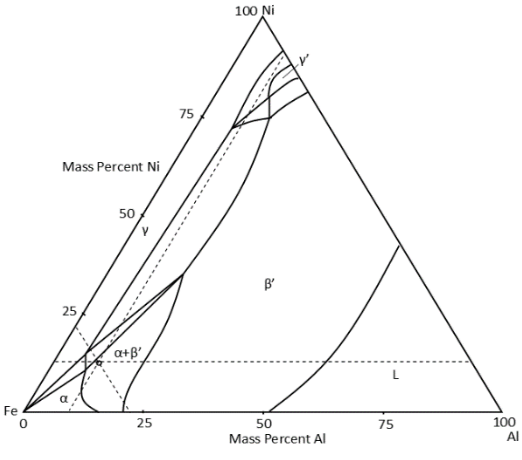


Figure 2. Estimation of pseudo-ternary phase diagram of Fe-Ni-Al-7.5 wt.% Cr at 1100 ^{o}C [15].

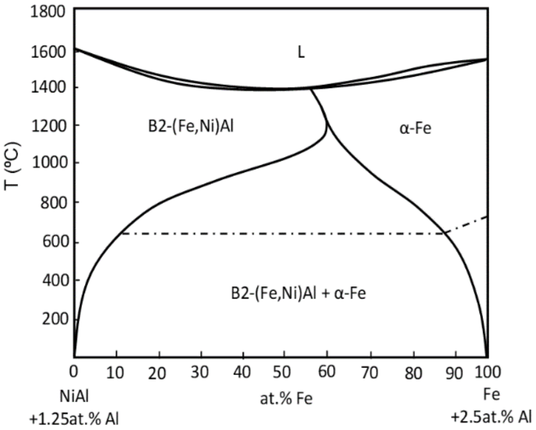
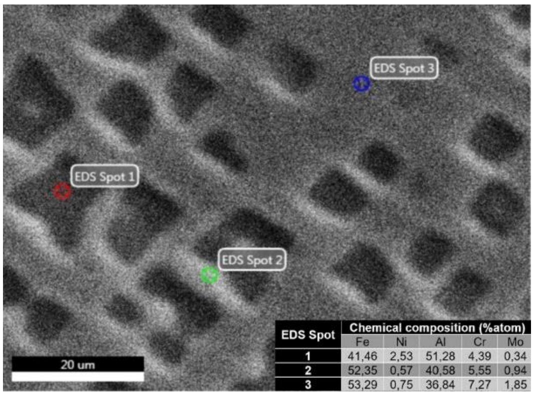


Figure 3. Pseudo-binary phase diagram of 48,75Ni-51,25Al up to 97,5Fe-2,5Al [16].

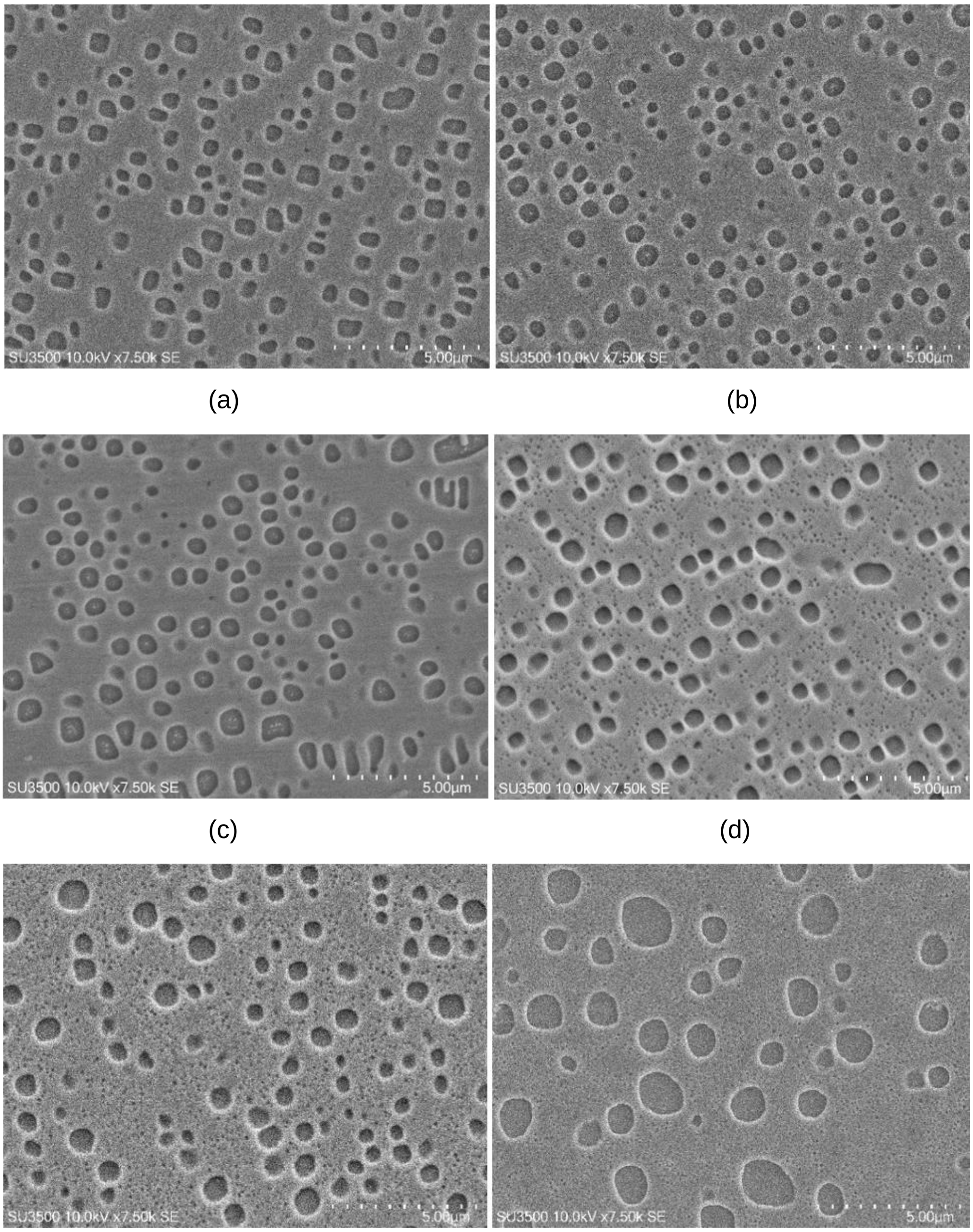


EDS Spot	Chemical composition (%atom)				
	Fe	Ni	Al	Cr	Mo
1	41,46	2,53	51,28	4,39	0,34
2	52,35	0,57	40,58	5,55	0,94
3	53,29	0,75	36,84	7,27	1,85

Figure 4. Microstructure and EDS analysis of as-homogenized sample.

Microstructures of As-heated Samples

Figure 5 show the microstructures of Fe-14Ni-9Al-7,5Cr-1Mo alloy after heated at 800, 900 and 1000 °C for 6 and 48 hours. The micrographs show clearly that increasing temperature made the morphology of B2 precipitates in the area relatively in the interior of grains have changed from rounded-cuboidal to fully globular. Moreover, the microstructures also indicate that the size of the precipitates increased with temperatures and times. .



(a) (b)

(c) (d)

(e) (f)

Fig. 5. Microstructures of alloy samples after heating
(a). at 800°C for 6 hours (b). at 800°C for 48 hours
(c). at 900°C for 6 hours (d). at 900°C for 48 hours
(e). at 1000°C for 6 hours (f). at 1000°C for 48 hours

Characterization with higher magnifications has revealed that other precipitates occurred inside of B2 precipitates. An example of this result has been shown by a sample of alloy heated at 800 ^{o}C for 48 hours, as shown in Figure 6.

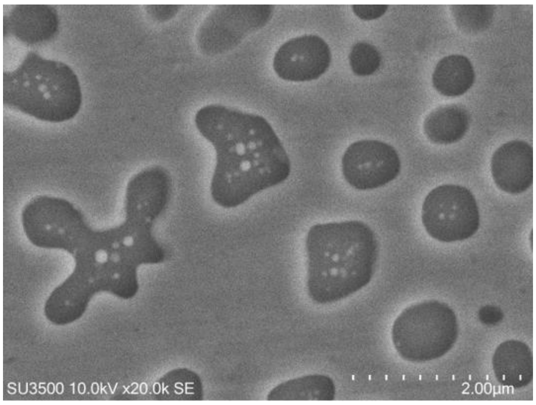


Figure 6. Microstructure of alloy sample heated at 800^{o}C for 48 jam showing heararchy precipitates in the B2.

This microstructure represents the area between interior and grain boundary that contained both globular and irregular B2 precipitates.

The histograms that display the size distribution of B2 precipitates in the sample heated at 800^{o}C for 6 and 48 hours are depicted in Figure 7. The histograms show that the size distribution of B2 precipitates heated for longer times, *i.e,* 48 hours was larger compared with that heated for shorter times, *i.e.,* 6 hours. Heating at 800 ^{o}C caused the average size of B2 precipitates increased from 479,6 ± 9,5 nm when heated for 6 hours to 505,8 ± 9,3 nm when heated at 48 hours. These facts indicate that coarsening of B2 precipitates has significantly occured at 800^{o}C. Moreover, it was found that, beside increasing size, the B2 precipitates increased their volume fractions, from 17,90% when heated for 6 hours to 19,07% when heated for 48 hours.

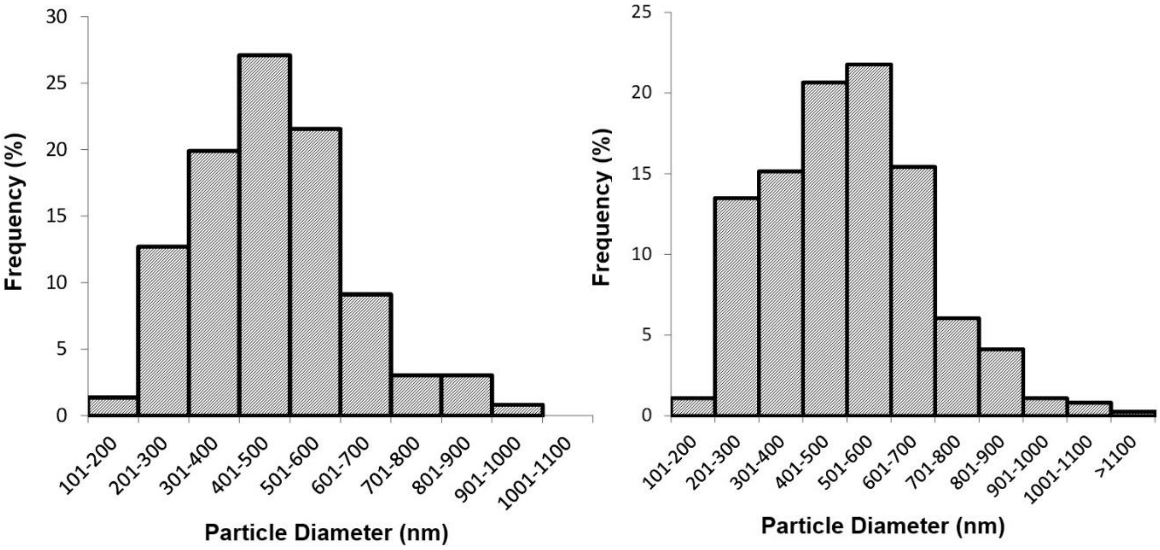


Figure 7. Size distribution of B2 precipitates in the sample heated at 800^{o}C for
(a) 6 hours
(b) 48 hours.

Figure 8 shows the detail microstructure of alloy sample heated at 1000^{o}C for 48 hours. It is seen clearly seen that two types of B2 precipitates were found in the sample, *i.e.,* large primary B2 and small secondary B2 precipitates. The size distribution of B2 particles in the alloy sample heated at 1000^{o}C for 6 hours and 48 hours are given in Figure 9 (a) and (b), respectively. Two bimodal distribution was identified in the histogram of sample heated at 1000 ^{o}C for 6 hours with two peak sizes of 540 nm and 140 nm. Larger primary B2 precipitates formed based on coarsening processes while smaller secondary B2 precipitates occurred during cooling. Heating at 800 °C, however, did not provide secondary precipitates.

Figure 8 also shown that the distribution of smaller B2 precipitets in the α-Fe matrix is relatively homogeneous. It has been reported that the solubilities and diffusivities of Ni and Al in the α-Fe matrix in ternary Fe-Ni-Al systems at 800 °C is significantly lower than that at 1000 °C [15]. Therefore, precipitation of secondary B2 precipitates easier during cooling from 800 °C compared with that from 1000 °C. Moreover, involvement of Cr in the Fe-Ni-Al systems increases the area of β’ [15]. Size distribution of B2 precipitets in the sample heated at 1000°C for 6 and 48 hours are presented in Figure 9 (a) and (b), respectively. Similar with that occur at 800 °C, the size distribution of B2 precipitates at 1000 °C for 48 hours is wider compared with that in 6 hours. The average size of B2 precipitates increased from 529,5 ± 9,2 nm when heated for 6 hours to 942,1 ± 9,8 nm when heated for 48 hours. This coarsening of B2 precipitates followed by increasing of their volume fraction from 20,46% when heated at 1000 °C for 6 hours to 22,93% when heated for 48 hours

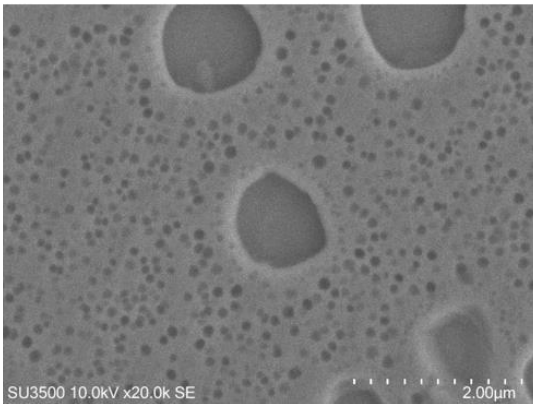


Figure 8. Microstructure of alloy sample heated at 1000 °C for 48 hours.

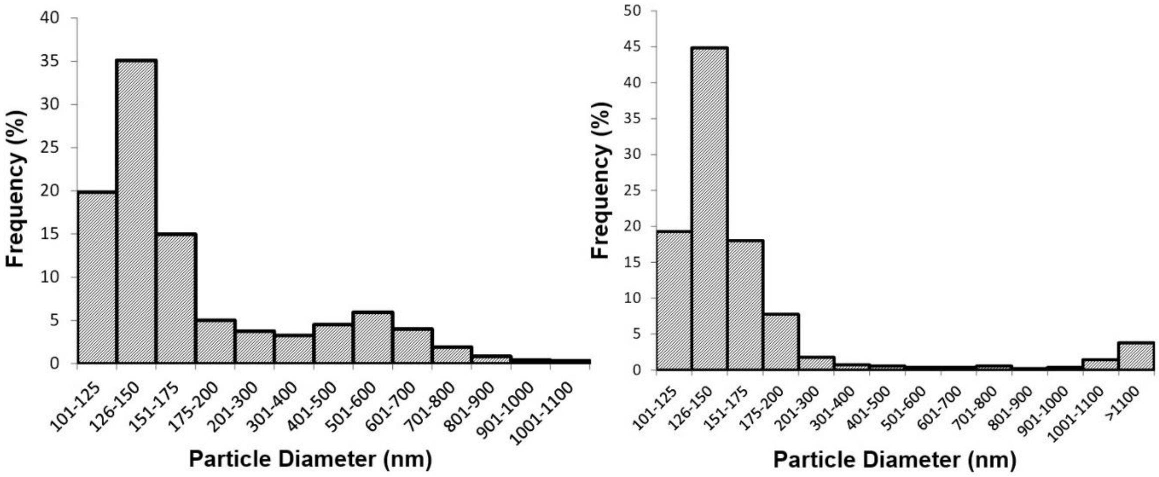


Figure 9. Size distribution of B2 precipitates in the sample heated at 1000 °C for
(a) 6 hours
(b) 48 hours.

Kinetics of B2-(Fe,Ni)Al Coasening

Heating the alloy at both lowest highest temperatures, i.e., 800 and 1000 °C, respectively caused the coarsening of B2 precipitates, indicated by increasing the average diameter the precipitates with times. Coarsening behaviour has been considered as one of the most important parameters for the alloy to be used at high temperatures. Therefore, coarsening kinetics of high temperature materials should be evaluated as the measure of high temperature stability of the microstructures. This coarsening of precipitates occurs as consequent of the alloy system to reduce its free energy. The coarsening process in high temperature alloys has been studied intensively [12], and diffusion control processes has been confirmed. Therefore, the higher the temperarures, the higher the mobility or diffusion of precipitates forming elements in α-Fe the matrix and this makes higher rate of coarsening.

Coarsening kinetics is normally expressed by the Lyfshitz-Slyozov-Wagner (LSW) equation as follows [17] .

$$(\overline{r}_t)^3 - (\overline{r}_o)^3 = kt \qquad (1)$$

In which $\overline{r}_t$ and $\overline{r}_o$ are average radius of precipitates at time t and initial time, respectively, while k is coarsening rate constrant. When $(\overline{r}_t)^3 - (\overline{r}_o)^3$ is ploted to t then a liniear relationship would be obtained. Figure 10 shows the coarsening kinetic plots for heating at 800 and 1000^{o}C, from which the slopes of the curves that indicate the coarsening rate constant (k) could be obtained.

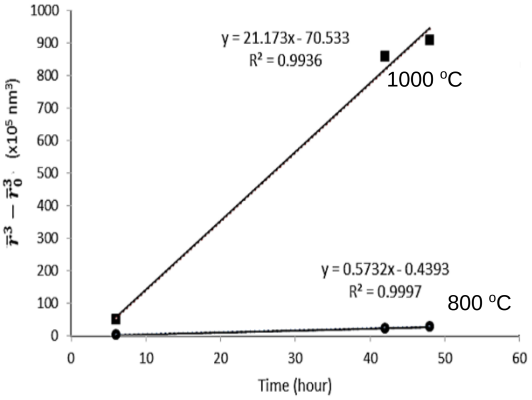


Figure 10. Relationship between ($\overline{r}_t^3 - \overline{r}_o^3$) and time of heating the alloy at 800 and 1000 ^{o}C.

The coarsening rate constants of the alloy heated at 800 and 1000 ^{o}C were 5,6x10^4 nm^3/h and 2x10^6 nm^3/h, respectively, indicated that coarsening at 1000 ^{o}C was much higher compared with that occurred at 800 ^{o}C, due to higher Ostwald ripening processes [11].

Coarsening study done by Stallybrass [8] has shown that the coarsening rate of B2 in ferritic Fe-Ni-Al-Cr alloys at 750 and 900 ^{o}C were 3x10^4 nm^3/h and 2x10^6 nm^3/h, respectively. Therefore, it is concluded that addition of molybdenum in Fe-Ni-Al-Cr ferritic systems reduced the coarsening rates of B2 precipitates at high temperatures.

It has been normally accepted that the coarsening rate coantant depends strongly on three parameters, *i.e.,* diffusivity of B2 forming elements in the α-Fe matrix, the interface energy of the precipitates and solubility of elements in the matrix [18]. Higher temperatures increase the diffusion of elements based on Archeniuss-type equation as follows [18].

$$D = D_o.\exp(\frac{-Q}{RT}) \qquad (2)$$

Where D is diffusion, Q is activation energy, R is gas constant and T is temperature. Therefore, the diffusion rate of aluminium, as the prime B2 forming element, would increase exponentially with temperatures. Consequently, the coarsening rate at 1000 ^{o}C would much higher compared with that occur at 800 ^{o}C.

Hierarchical Precipitation

The experiments have shown that hierarchical precipitation occurred in the sample of Fe-14Ni-9Al-7.5Cr-1Mo alloy heated at 800^{o}C, but this was not found in the sample heated at 900 and 1000 ^{o}C. Figure 11 (a) and (b) shown the ultra fine precipitets occurred inside either in the globe or irregular of B2 precipitates.

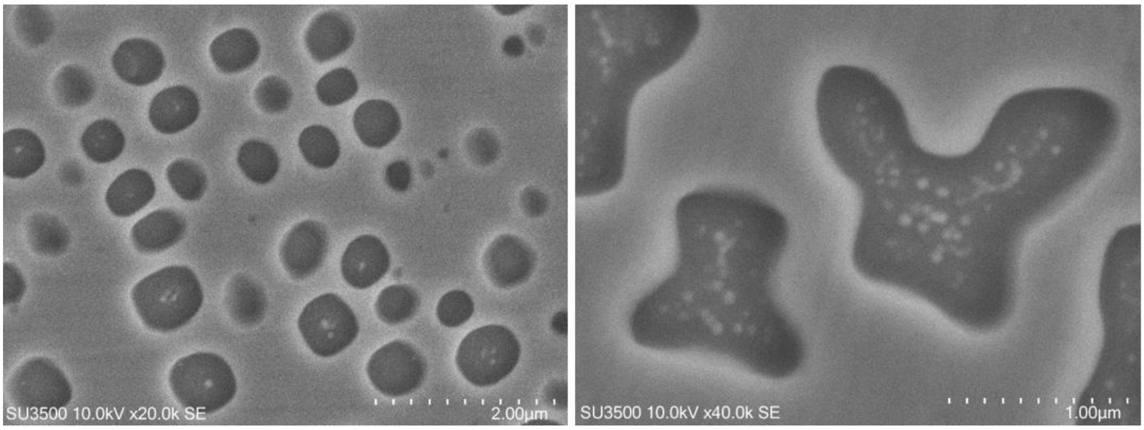


Figure 11. Hierarchical precipitates found in the sample heated at 800 ºC for 48 hours
(a) in the interior of the matrix
(b) in the area around grain boundaries.

Previous studies reported that these nano particles had crystal structure of $L2_1$-Ni_2TiAl [19,20] found in ferritic Fe–Al–Cr–Ni–Ti. Further study is needed to evaluate these hierarchical precipitates in the Fe-Ni-Al-Cr-Mo ferritic alloys. Nevertheless, it is predicted that the hierarchical precipitates found in this study is in an $L2_1$-Ni_2Al type crystal structure. Partition of elements, such as Fe, Cr and Mo might occur in this nano particles.

Effect of Microstructures on Hardness

The mechanical properties of the alloys was measured based on its hardness at room temperature after the alloy was heated at different temperatures and times. It is expected that the hardness is affected by the precipitets volume fraction and size. The higher the volume fraction of the precipitates the higher the hardness of the alloy. In addition, the smaller the precipitates size, in certain volume fraction, makes the distance between particles shorter, and consequently increases the hardness of the alloy as the barrier for dislocation pinnings stronger. It was confirmed that the size of precipitates were mush smaller compared with the indentor, as shown in Figure 12. Therefore, it is confirmed that the hardness measured represents the average hardness of the matrix α-Fe and precipitates of B2.

Figure 13 show the hardness variation of Fe-14Ni-9Al-7.5Cr-1Mo alloy after heated at 800, 900 and 1000 ºC for different times. The curves indicate that at all temperature concerns, the hardness of the alloy decreased with time. Coarsening of B2 precipitates is believed responsible for these decreasing mechanical properties of the alloy. In addition, higher hardnesses were found in the sample heated at higher temperatures. The reason for this fact is that secondary nano particles B2 could occur at higher temperatures but no such particles were found at lower temperatures. Therefore, it is confirmed that fine secondary B2 precipitates contribute significantly on the mechanical properties of the alloy.

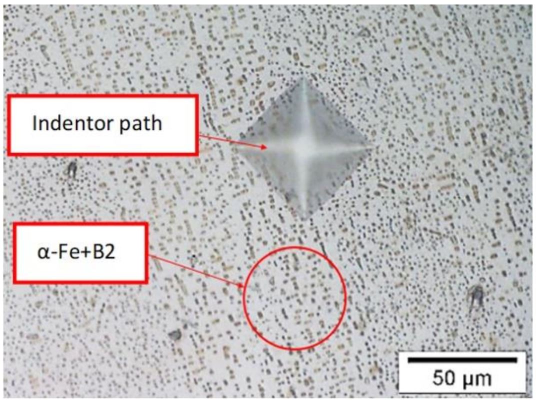


Figure 12. Indentation parth on the sample heated at 1000 ºC for 48 hours.

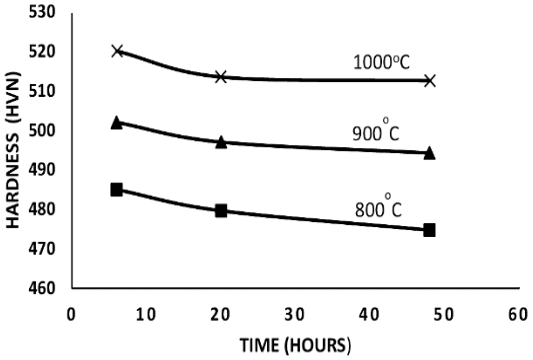


Figure 13. Hardness of alloy after heated at different temperatures and time

CONCLUSION

Microstructural evolutions of Fe-14Ni-9Al-7.5Cr-1Mo alloy when heated at 800^{o}C, 900 and 1000 ^{o}C for different times have been discussed. The alloy is confirmed had both relatively large primary and fine secondary B2-(Fe,Ni)Al precipitates distributed homogeneously in the α-Fe matrix. This secondary B2 precipitates occurred only in the sampel heated at 1000^{o}C. These both B2-type precipitates act as srengthener or hardener in the alloy. During heating at all temperatures, the precipitates experienced coarsening, with coarsening rate constant of 5,6x10^{4} nm^{3}/h and 2x10^{6} nm^{3}/h at 800 and 1000 ^{o}C, respectively. In addition, hierarchical precipitation was found when the alloy heated at 800 ^{o}C. However, no such hierarchical precipitation occurred at 1000 ^{o}C.

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MAIN CONTRIBUTOR STATEMENT

Main contributors in this manuscript are Eddy Agus Basuki and Syaiful Bachri.

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