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EFFECT OF AGING TREATMENT ON THE MICROSTRUCTURES AND HARDNESS OF Fe-Ni-Cr SUPERALLOY

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ABSTRACT

Aging treatment was conducted on Fe-Ni-Cr superalloy to observe its effect on the microstructures and hardness of the material. Solution treatments were carried out at 900°C, 975°C, 1050°C, and 1125°C followed by water quenching. The samples were further treated with a double aging treatment at 720°C and 650°C for 4 hours and 12 hours respectively. Materials characterization techniques such as SEM, XRD, and optical microscopy were used to analyze the heat-treated samples. The Fe-Ni-Cr superalloy formed mainly a dendritic austenitic structure with $Cr_{23}C_6$ precipitated along the grain boundaries. Increase in solution treatment temperature results in dissolution of chromium carbide, coarser grain, and lower hardness for non-aged samples. Double aging treatment produced more chromium carbide, higher hardness, but no apparent change in grain size. Neither quenching nor aging treatments caused any phase transformation. The highest hardness value of 220.4 Hv was recorded for the sample solution treated at 1125°C for 2 hours and water quenched, followed by 2-step aging at temperature 720°C and 650°C for 4 hours and 12 hours respectively, and air cooled.

Keywords: Fe-Ni-Cr superalloy; solution treatment; aging; precipitation.

INTRODUCTION

Heat resistant material is one type of superalloy that is extensively used in engineering design especially in high temperature applications. The high temperature strength of all superalloys is based on the principle of a stable face-centered cubic (FCC) matrix combined with either precipitation or strengthening and solid-solution strengthening (ASM Handbook, 1991). Iron-nickel-chromium (Fe-Ni-Cr) alloy is one type of superalloy which is being used as heat resistant material. The combination of iron, nickel, and chromium provides the characteristics needed for this material to operate in a high temperature environment. Aging treatment prior to solution treating is one of the most common treatments used for superalloys (Moniruzzaman, Rakib, & Matin, 2012). Usually solution treating will be the first step in the heat treatment process. The temperature for solution treating depends on the desired properties. Higher solution-treating temperatures will result in some grain growth and more extensive dissolving of carbides. Lower solution-treating temperatures dissolve only a small amount of carbide and without grain growth (ASM Handbook, 1991). After solution treating, the material will usually undergo quenching. The purpose of this quenching is to maintain, at room temperature, the supersaturated solid solution obtained during solution treating.

Quenching permits a finer age-hardening precipitate size. Cooling methods commonly used include oil and water quenching as well as various forms of air or inert gas cooling (ASM Handbook, 1991). The temperature for solution treating and the type of quenching medium depends on the property desired on the metal via the treatment process. Rapid cooling will decrease the diffusion rate while slow cooling will increase the diffusion rate of the material, which will result in different types of microstructure according to the present phase in the structure. Prior to quenching, the dissolve carbides will be in a metastable state where aging treatment is conducted to promote homogeneous carbide precipitation in matrix as the hardening phase. After aging, the resulting microstructure consists of large grains that contain the aging phases and a heavy concentration of carbides in the grain boundaries.

HEAT TREATMENT

In order to study the effects of aging treatment on the microstructure and hardness of Fe-Ni-Cr superalloy, the types of heat treatments selected were solution treating followed by aging. Table 1 shows the temperature and time for the respective heat treatment. Four samples underwent solution treating followed by double aging while another four underwent solution treatment (water quenched) only in order to serve as controlled samples.

Table 1. Double aging treatment.

Solution treating temperature (^oC) for 2 h (water quench)	Reheating temperature(^oC) for 4 hour (air cool)	Aging temperature for 12 h (air cool)
900	720	650
975	720	650
1050	720	650
1125	720	650

All samples prepared for heat treatment were cut into 10 x 10 x 10 mm dimensions. The cutting process was conducted using an abrasive cutter with a continuous supply of coolant in order to avoid microstructure change from excessive heat. The heat-treated samples were analyzed by several materials characterization techniques: optical microscopy, scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX), x-ray diffraction (XRD), volume fraction analysis and a hardness test. As for metallurgical investigation, the specimen was mounted using a hot mounting process. The mounted specimen then underwent a surface finishing process before being inspected under an image analyzer and SEM. The surface finishing process included grinding by using abrasive silicon carbide paper with an increasing number sequence, started from 240, 320, 400, 600, 800, 1200, and 2400. Once grinding was completed, where only a soft scratch was detected on the surface, the specimen underwent a polishing process with alumina solution in order to produce a deformation-free surface which was flat, scratch-free, and had a mirror appearance. Finally, etchant was applied to the specimen surface to reveal the microstructure of the specimen. The etchant used was a combination of 45 ml glycerol, 30 ml hydrochloric acid, and 15 ml nitric acid.

A hardness test was conducted using a Vickers Hardness machine on the specimen before and after the heat treatment process. The load used was 30 kgf and indentation duration was 15 seconds. In order to obtain an accurate and reliable result, five indentations were made in each specimen and the average reading was taken. Grain size is the diameter of individual grains on the microstructure of the material. However, Fe-Ni-Cr superalloy has a dendritic structure, so the grain size was measured according to dendritic arm spacing (DAS). Technically, the measurement is conducted perpendicularly along the dendrite and DAS is calculated by dividing the total length by the number of dendrites intercepted. The average value was calculated from 15 measurements on different dendrites. The volume fraction of the carbide was measured using image analyzer software, Omnimet. SEM micrographs were analyzed by measuring the area fraction of carbide on the microstructure. The area fraction is the fraction of the carbide area with respect to the matrix area. According to ASTM E1245-03, the relationship between the volume fraction and area fraction is as follows:

$$V_V = A_A = \frac{A_i}{A_T} \quad (1)$$

where

V_V = the volume fraction
A_A = the area fraction of the inclusion or constituent
A_i = the area of the detected feature
A_T = the measurement area (field area)

RESULTS AND DISCUSSION

Microstructure Investigation

Optical micrographs of water quenched and aged samples show that the microstructure retains an austenitic dendritic structure prior to heat treatment. However, grain coarsening was observed as the solution treatment temperature increased. Figures 1 and 2 show the optical micrographs of water quenched and aged samples. As shown in Figure 1, the austenitic dendritic structure was observed on water quenched samples as the solution temperature increased from 900^{o}C to 1125^{o}C. In terms of dendritic size, a slight increase of dendritic arm spacing was observed as the temperature increased from 900^{o}C to 1125^{o}C. Meanwhile, the aged samples shown in Figure 2 also show similar phenomena in terms of microstructural changes and dendritic coarsening. This finding is similar to that of Wang (2009). The grain size measurement of Fe-Ni-Cr superalloy was conducted by measuring the dendritic arm spacing (DAS). Figure 3 shows the DAS of the heat-treated samples. Both water quenched and aged samples show an increasing trend of DAS as the temperature increases. This is because the grain size is controlled by a thermally activated process through the migration of the grain boundary, where the free energy of the boundaries acts as the major driving force for grain growth (Koul & Pickering, 1982). Thus, an increase in solution temperature provides more free energy to the boundary, which promotes grain growth and results in grain coarsening. SEM micrographs for both heat treatments show the presence of chromium carbide, $Cr_{23}C_6$ which is determined by EDX analysis. Figure 4 shows the EDX analysis of $Cr_{23}C_6$. SEM micrographs of the heat treated samples are shown in Figures 5 and 6.

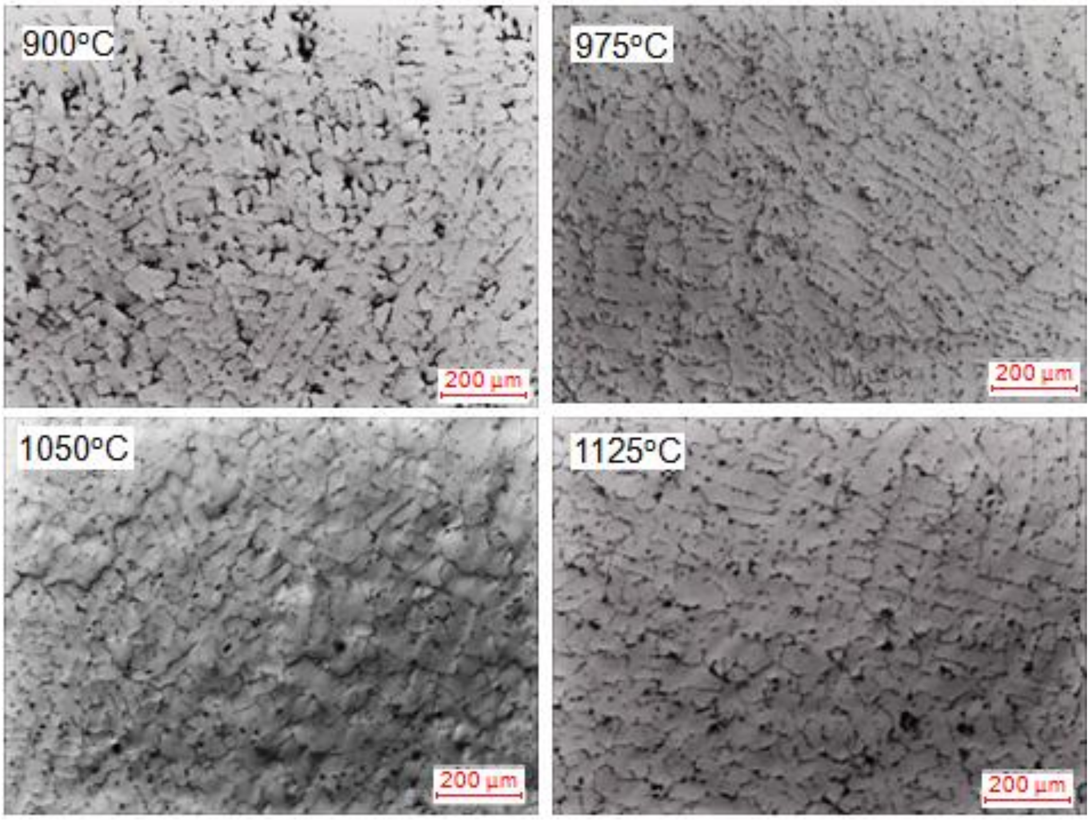


Figure 1. Optical micrographs of water quenched samples.

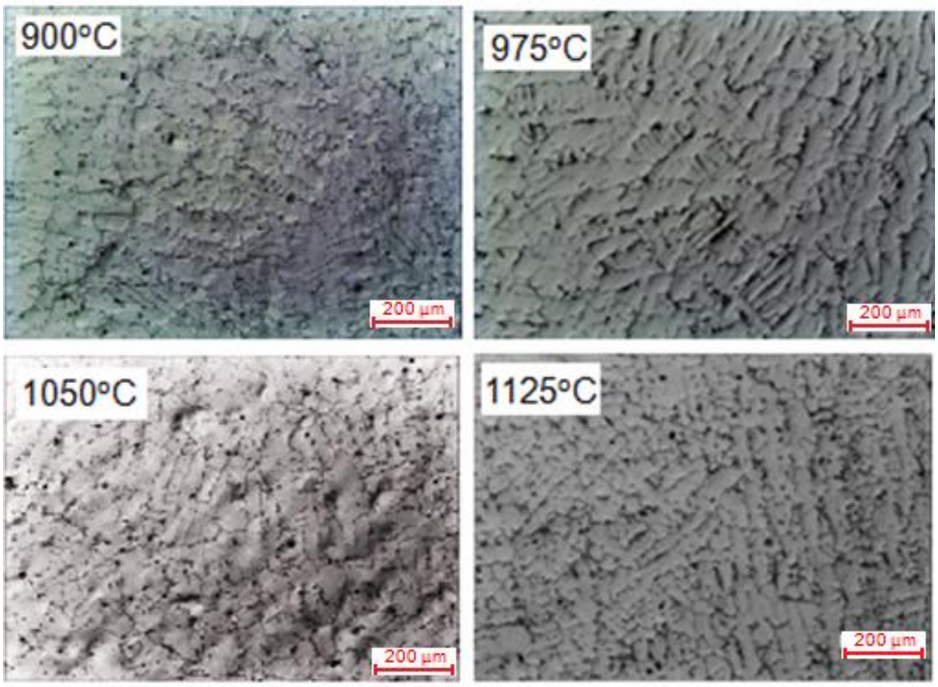
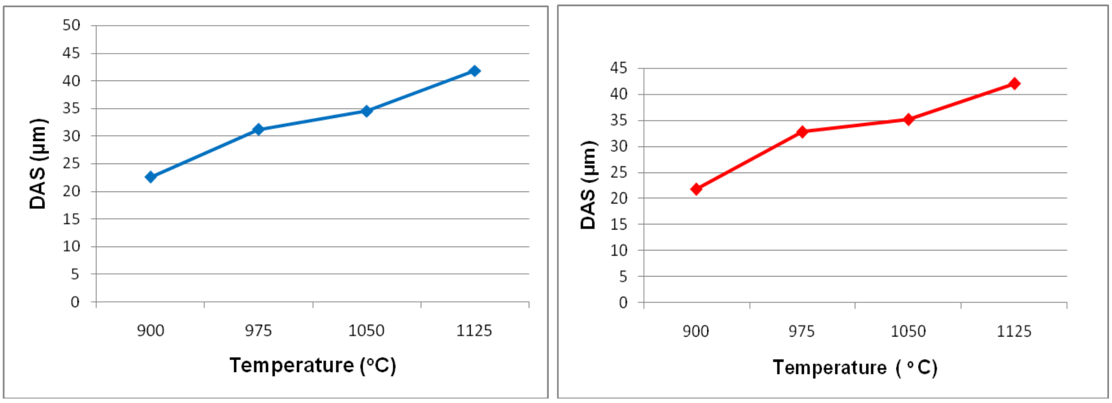


Figure 2. Optical micrographs of aged samples.



(a) (b)

Figure 3. Effect of temperature on DAS of (a) water quenched and (b) aged samples.

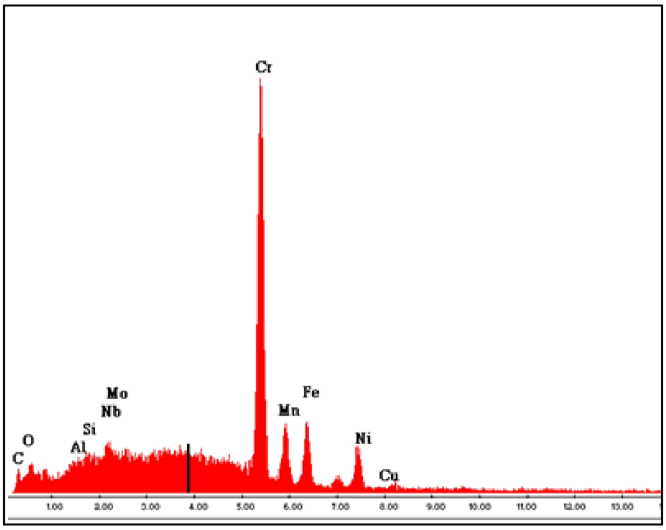


Figure 4. EDX analysis of $Cr_{23}C_6$.

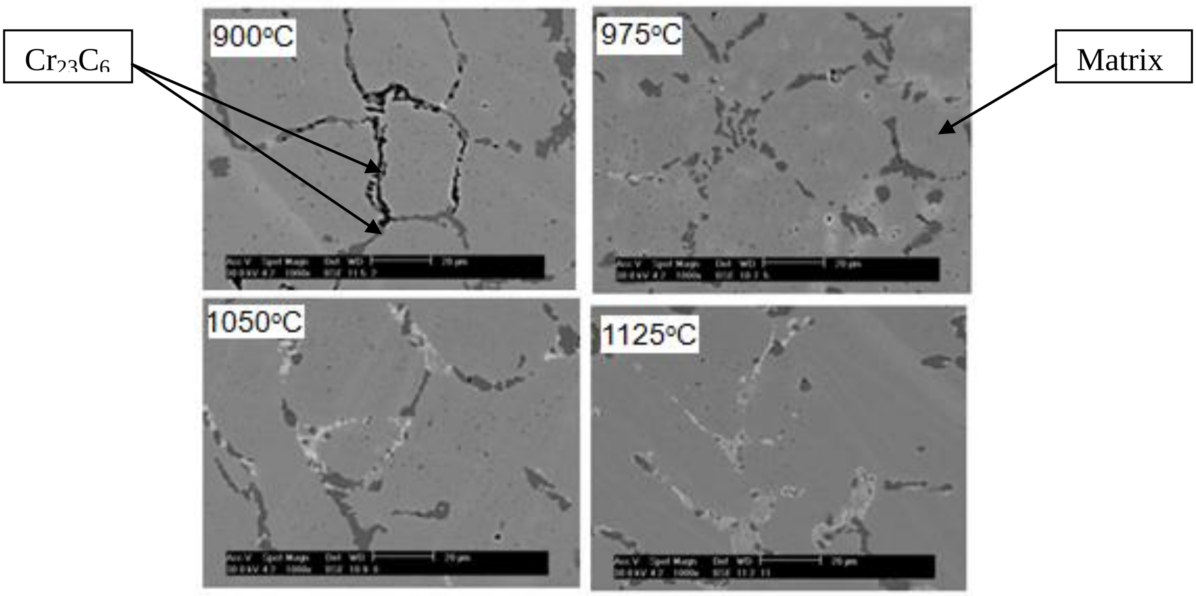


Figure 5. SEM micrographs of water quenched samples.

As shown in Figures 5 and 6, $Cr_{23}C_6$ compound was observed on the microstructure for both water quenched and aged samples. This compound was identified as the dark phase on the micrograph, which is mainly concentrated in the grain boundary area. Furthermore, area fraction analysis was conducted using an image analyzer to obtain an accurate volume fraction of the carbide phase by using Eq. (1). The amount of carbide phase observed for the water quenched samples was decreased (dissolved from the grain boundary) as the solution temperature increased. This finding is similar to that observed by Jacuinde et al. (2003). The reason for this behavior is that at higher temperatures (above 1000^{o}C), austenite can dissolve higher carbon contents, so that the amount of secondary carbides is also minor (Jacuinde et al., 2003). However, the amount of carbide observed for aged samples differed slightly, and at a temperature of 1125^{o}C, the quantity of carbide phase observed is higher than at 1050^{o}C. This might be due to precipitation of carbide during aging after being dissolved extensively at high temperature (1125^{o}C). According to Davis (1997), after the carbide is dissolved, the matrix becomes a supersaturated solid solution, thus, during aging the precipitation of this phase will occur.

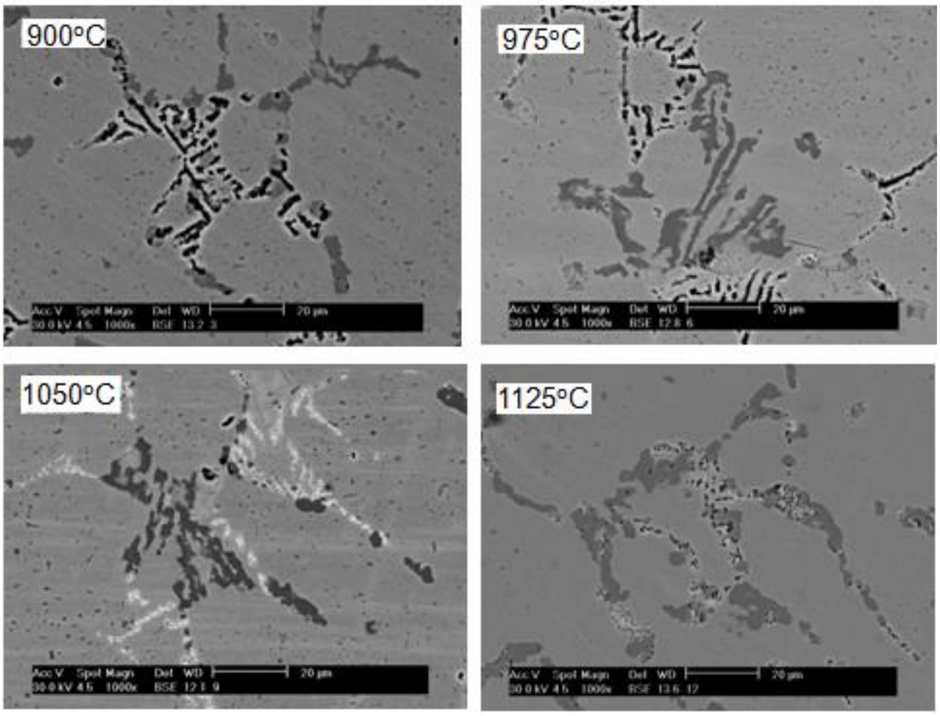
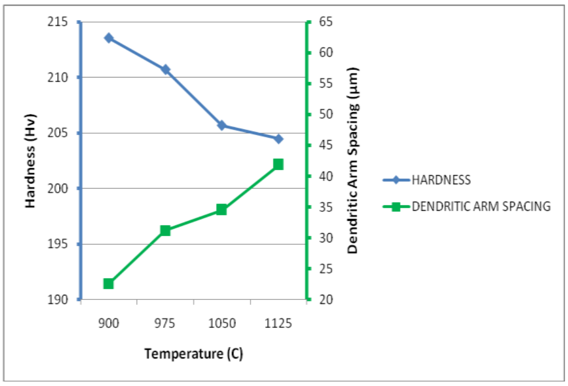


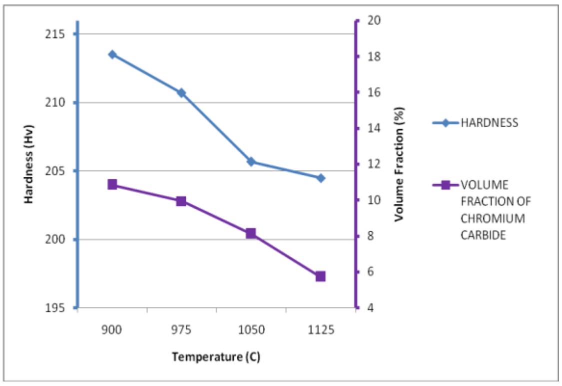
Figure 6. SEM micrographs of aged samples.

Hardness

The hardness results of the water quenched samples are shown in Figure 7. Generally, the hardness value decreased as the solution temperature increased. This finding is similar to that observed by Wang (2009). Figure 7(a) shows the effects of grain size on the hardness, where grain coarsening due to increases in temperature lowers the hardness of the material. This is because grain coarsening reduces the grain boundary strengthening effect. This finding is similar to that of Zhang et al. (2008). Meanwhile, as the grain size decreases, there is more build-up of dislocation at the grain edge. Since it requires a lot of energy to move dislocation to other grains, this dislocation will build up along the boundary, which increases the strength of the alloy (Zhang et al., 2008). This strengthening theory is also known as Hall-Petch strengthening. Thus, Hall-Petch strengthening is reduced as the grain size increases. On the other hand, carbide content also affects the hardness of the material. Figure 7(b) shows the effect of carbide on the hardness. The curves show that decrease in carbide content causes a decrease in the hardness of the material. This finding is similar to that observed by Jacuinde et al. (2003). The reason for this behavior is the presence of carbide precipitates, which increase the yield strength of the material by blocking the dislocation motion (Jacuinde et al., 2003), since dislocation can only move through this particle by either shearing or climbing. Thus, this interruption in dislocation motion increases the strength of the material.



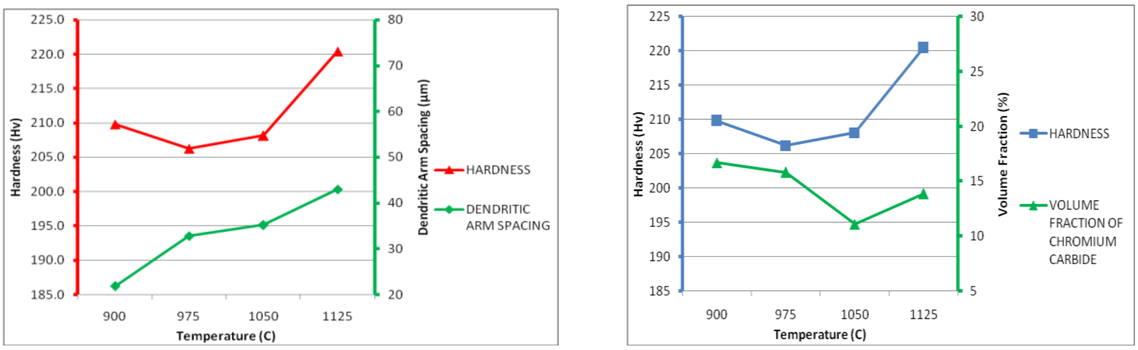
(a)



(b)

Figure 7. Effect of temperature on (a) hardness and DAS of water quenched samples; (b) hardness and volume fraction of chromium carbide ($Cr_{23}C_6$) of water quenched samples.

Hardness curves of the aged samples are shown in Figure 8. The curve shows a different trend from the water quenched samples. As the temperature increases from 900^{o}C to 975^{o}C, the hardness value slightly decreases. However, as the temperature increases to 1050^{o}C, the hardness value increases. Then, as the temperature reaches 1125^{o}C, the hardness value increases markedly, reaching 220.4 Hv. The mechanical strength achieved at this temperature is the highest value obtained as compared with the other heat treatment. Figure 8(a) shows the effect of grain size on the hardness of the material. It can be seen that the grain coarsening characteristic did not reflect the unique increases in the hardness behavior, so the effect of carbide content on the hardness of aged samples was analyzed. Figure 8(b) shows the effect of carbide content on the hardness of the material. It shows that decrease in carbide content from 900^{o}C to 975^{o}C produces a slight decrease in hardness. However, a slight increase in hardness was observed as the carbide content continued to decrease. As the temperature increased from 1050^{o}C to 1125^{o}C, an increase in carbide content, followed by a remarkable increase in hardness, was observed. The increase in hardness at a temperature of 1125^{o}C can be related to the increase in carbide volume fraction prior to the precipitation hardening process. Even though the carbide content was slightly lower compared to the sample heated at 900^{o}C, the carbide precipitates at this temperature were finer. The reason for this behavior is that a large amount of carbide had been dissolved as the heating temperature reached 1125^{o}C, then the aging process promoted the carbide to precipitate but in a controlled atmosphere, which results in finely dispersed particles of carbide precipitates in the matrix. Stallybrass et al. (2004) explained that this form of carbide can give a better strengthening effect than coarser carbide. In addition, the increment in grain size from 1050°C to 1125°C was only around 4µm, which did not have a significant effect in reducing its strength. However, at a temperature of 1050°C, a decrease in carbide content still caused an increase in the hardness value. It should be noted that the sample heated at 1050°C also shows a certain amount of carbide dissolution. Thus, prior to aging, this sample also experienced fine carbide precipitation, which increased the strength regardless of the lower amount of carbide. This is because the carbide morphology plays an important role in terms of the strengthening effect, as stated previously by Stallybrass et al. (2004). Meanwhile, at temperatures of 900°C and 975°C, carbide dissolution was hardly observed in the microstructure. Thus, the precipitation strengthening effect did not have a significant effect at these two temperatures. The hardness value tends to decrease prior to the decrease in carbide content and increase in grain size.

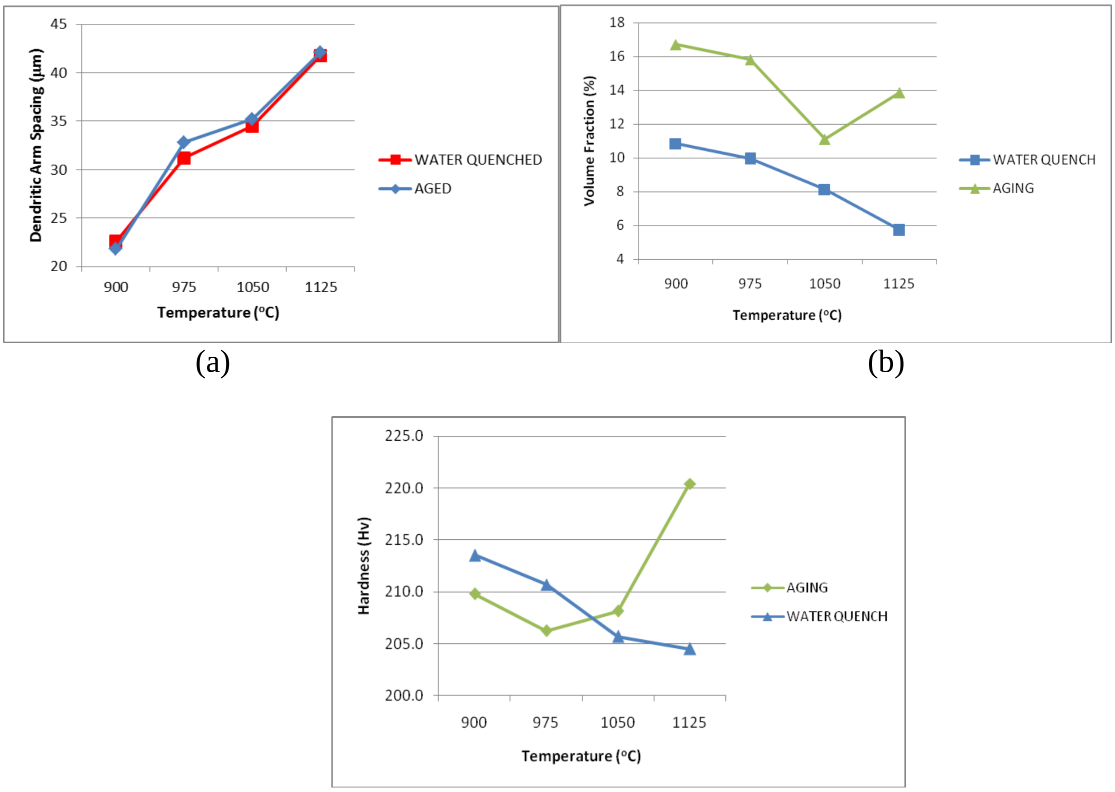


(a) (b)

Figure 8. Effect of temperature on (a) hardness and DAS of aged samples, and (b) hardness and volume fraction of chromium carbide ($Cr_{23}C_6$) of aged samples

Effect of Aging on Quenched Samples

Discussing the effects of aging on quenched samples after solution treatment, a critical comparison is made between water quenched and aged samples. Figure 9 shows the effect of aging on the grain size, carbide content, and hardness. As shown in Figure 9(a), there is no apparent difference in grain size after the aging process. This is because during aging, the energy supplied might not be sufficient to promote migration of the grain boundary to cause grain growth (Civjan et al., 1972). On the other hand, grain growth is also promoted by dissolution of pinning particles from the grain boundary, which is unlikely to occur during aging; instead, carbide precipitation will take place (Civjan et al., 1972). Thus, even if grain growth takes place during aging, which might be due to extremely long heating time, the amount will be so little that it will be hardly noticeable in the microstructure analysis. Figure 9(b) shows that aged samples showed a consistently higher amount of chromium carbide than water quenched samples. This shows that the aging process can markedly increase the amount of chromium carbide. However, as discussed previously, at a temperature of 1125°C the aged sample showed a unique increase in carbide content compared to the 1050°C sample. Here, the reason can be analyzed in further detail as a reflection of the amount of carbide dissolved right after the solution treatment process and before aging is conducted. As shown in the water quenched samples, at a temperature of 1125°C, the amount of carbide was very low at about 5.7% compared to the as-received alloy which had about 12.6% carbide content. This means that about 6.9% of the carbide dissolved during the solution treatment at this temperature. Thus, the percentage of carbide dissolution at this temperature was higher than at other temperatures. Meanwhile, the high amount of metastable dissolved carbide in the water quenched condition had precipitated well after aging. This might be the possible cause of the increased carbide content in the aged sample at 1125°C solution temperature. However, it can also be seen that at temperatures of 900°C and 975°C, the increase in carbide content was quite high compared to 1050°C, even though the carbide dissolution was lower due to insufficient heat. The reason for this behavior was that the increase in carbide content was actually due to the growth of pre-existing carbide after aging, which was undesirable in terms of mechanical properties (Stallybrass et al., 2004). However, as the temperature increased to 1050°C, more carbide started to dissolve, as shown in the SEM micrograph of the water quenched sample. According to Ridhwan et al. (2013), increase in solution temperature could lead to higher carbide dissolution from the matrix. Thus, the increase in carbide content at a temperature of 1050°C was mostly due to carbide precipitation rather than carbide growth, which explained the small increase in carbide content. Nevertheless, the presence of this newly precipitated carbide is in the form of fine and dispersed carbide, which is desirable in terms of mechanical properties.



(a) (b)

(c)

Figure 9. Effect of aging on (a) grain size, (b) carbide content, and (c) hardness of Fe-Ni-Cr superalloy

In terms of hardness, Figure 9(c) shows that, as the temperature increases from 900°C to 975°C, the hardness value of water quenched samples is higher than the aged samples. However, as the temperature increases to 1050°C and 1125°C, the aged samples showed higher hardness than the water quenched samples which are in the solution heat-treated state. At temperatures below 1050°C, the higher hardness of the water quenched samples can be attributed to the finer precipitate of pre-existing carbide compared to the aged samples, which have a coarser carbide morphology. Meanwhile, at temperatures of 1050°C and 1125°C, the lower hardness value of the water quenched samples is due to high carbide dissolution which causes extremely low content of carbide precipitate. At the same time, the increase in hardness in the aged samples is due to the presence of finely dispersed particles of carbide precipitate which further increase the hardness of the samples.

XRD Analysis

On the other hand, XRD analyses were also conducted to identify the phase transformation after aging. Figure 10 shows the XRD patterns of aged samples and water quenched samples. No new peak is observed due to the aging treatment. At the same time, the pre-existing peak can still be observed after the aging process. This shows that no new phase was being formed and no pre-existing phase was transformed prior to the aging treatment. This is because the as-received material had shown austenitic structure before the aging process, which has high stability and is not likely to transform into another phase under the aging treatment. This behavior is similar to that found by Gheno et al. (2011).

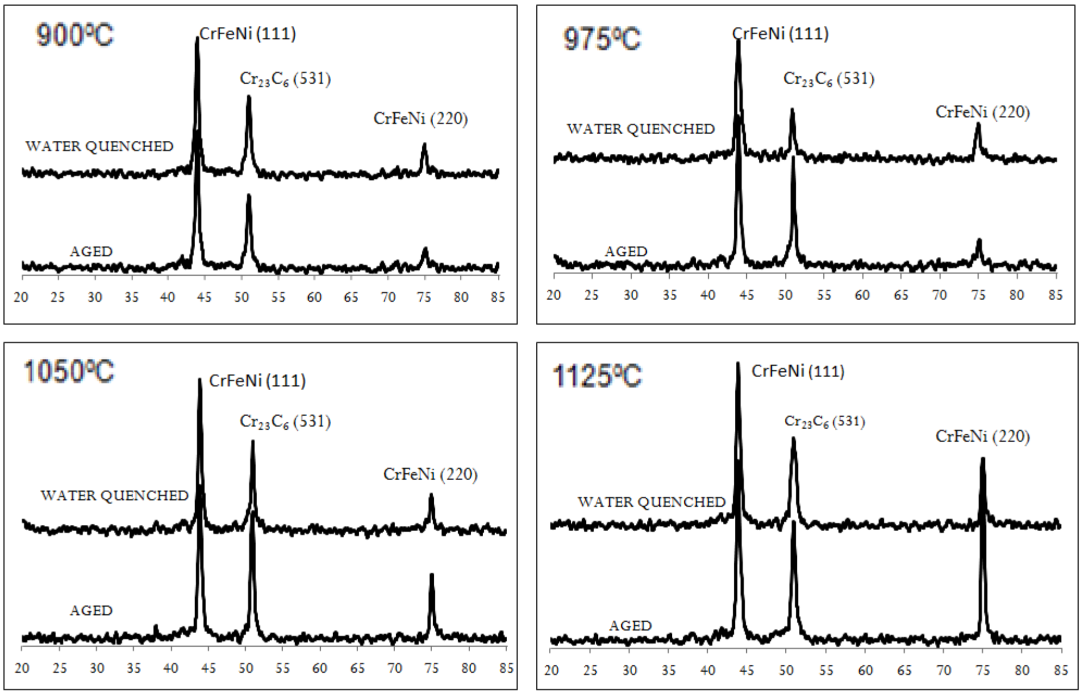


Figure 10. XRD pattern of water quenched and aged samples.

CONCLUSIONS

The effects of aging treatment on the microstructure and hardness of Fe-Ni-Cr superalloy have been investigated. Microstructure investigation and hardness tests have been conducted as well. Analysis of the results leads to a few major conclusions. In terms of solution treatment, increase in temperature leads to increase in grain size but reduces the chromium carbide, $Cr_{23}C_6$ content and the hardness value. In addition, for non-aged samples, grain coarsening behavior reduces the hardness value due to its low grain boundary strengthening effect. Higher amounts of fine chromium carbide in microstructure increase the hardness value due to the precipitation strengthening effect, while lower amounts of fine chromium carbide reduce the hardness value. Aging treatment increases the amount of chromium carbide, which further increases the hardness value of the alloy by precipitation strengthening. The highest average hardness value of 220.4 Hv and tensile strength 743.1 MPa was recorded on the sample solution treated at 1125^{o}C for 2 hours (water quenched) and aged at 720^{o}C (air cooled) then 650^{o}C (air cooled) for 4 hours and 12 hours respectively. The XRD pattern shows that neither phase formation nor phase transformation was observed during all the series of heat treatments conducted.

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