EFFECT OF COOLING RATE ON THE MICROSTRUCTURES AND HARDNESS OF Fe-Ni-Cr SUPERALLOY

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ABSTRACT

Fe-Ni-Cr superalloy is a heat resistant material that shows good mechanical strength and resistance to oxidation and corrosion at high temperature. Heat treatment was conducted on Fe-Ni-Cr superalloy to observe its effect on the microstructures and mechanical property namely hardness. Solution treatment were carried out at 900ºC, 975ºC, 1050ºC, and 1125ºC followed by two cooling methods i.e quenching and air cool. The samples were analyzed by using materials characterization techniques such as SEM, XRD, and optical microscope. The Fe-Ni-Cr superalloy formed mainly 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. Different cooling rate after solution treatment shows that fast cooling produces fewer and finer chromium carbide ($Cr_{23}C_6$) precipitates, finer grain, and higher hardness as compared with slow cooling rate. Highest hardness value which is 213.5 Hv was recorded on sample solution treated at 900ºC followed by water quenched.

KEYWORDS: *Fe-Ni-Cr superalloy, Solution treatment, Cooling rate, Precipitation*

1.0   INTRODUCTION

Superalloy is a very useful invention in engineering world. The term 'super' is referred to its performance which is higher than usual material in the respective aspects, which is why superalloy also known as high-performance alloy. This material is commonly known for the excellent mechanical strength and creep resistance at high temperatures, good surface stability, and corrosion and oxidation resistance (Sims et.al, 1987).

Heat resistant material is one type of superalloy that extensively used in engineering design especially in high temperature application. 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 alloy (Fe-Ni-Cr) is one type of super alloy which is being used as heat resistant material. The combination of iron, nickel, and chromium provides a desired characteristic needed for this material to operate in high temperature environment.

This research is carried out to determine the effects of different cooling rate during heat treatment on the microstructures and hardness of Fe-Ni-Cr superalloy.

2.0   METHODOLOGY

All samples prepared for heat treatment was cut into 10 x 10 x 10 mm dimensions. The cutting process was conducted by using abrasive cutter with continuous supply of coolant in order to avoid microstructure change from excessive heat.

In order to study the effects of heat treatment on the microstructure and hardness of Fe-Ni-Cr superalloy, the types of heat treatments selected were solution treating followed by two different cooling method.

Table 2.1 shows the heat treatment temperature and time as well as cooling method conducted.

TABLE 1
Heat treatment

Solution Treating (^{o}C) for 2 hour	Cooling Method	
900	Air cool	Water quench
975	Air cool	Water quench
1050	Air cool	Water quench
1125	Air cool	Water quench

The heat treated samples were analyzed by several materials characterization techniques which are optical microscope, scanning electron microscope (SEM), energy dispersive x-ray spectroscopy (EDX), x-ray diffraction (XRD), volume fraction analysis and hardness test.

As for metallurgical investigation, the specimen was mounted by using hot mounting process. The mounted specimen then undergoes surface finishing process before being inspected under image analyzer and Scanning Electron Microscopy. The surface finishing processed includes grinding by using abrasive silicon carbide paper with increasing number sequence, started

from 240, 320, 400, 600, 800, 1200, and 2400. Once grinding was completed where only soft scratch was detected on the surface, the specimen undergone polishing processed with alumina solution in order to produce deformation free surface which are flat, scratch free, and mirror appearance. Finally, etchant was applied to the specimen surface to reveal the microstructure of specimen. The etchant used was combination of 45 ml glycerol, 30 ml hydrochloric acid, and 15 ml nitric acid.

Hardness test was conducted by using Vickers Hardness machine on specimen before and after heat treatment process. The load used was 30 kgf and indentation duration was 15 seconds. In order to obtain an accurate and reliable result, 5 indentations placed on each specimen and average reading was taken.

Grain size is the diameter of individual grain on microstructure of material. However, Fe-Ni-Cr superalloy has a dendritic structure, thus, the grain size was measured according to Dendritic Arm Spacing (DAS). Technically, the measurement will be conducted perpendicularly along the dendrite and DAS is calculated by division of total length with number of dendrite intercepted. Average value was calculated from 15 measurements on different dendrites.

Volume fraction of carbide was measured by using image analyzer software, Omnimet. SEM micrograph was analyzed by measuring the area fraction of carbide on microstructure. Area fraction is the fraction of carbide area with respect to the matrix area.

According to ASTM E1245-03, the relationship between volume fraction and area fraction are as follows;

$$V_V = A_A = \frac{A_i}{A_T}$$

(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)

3.0   RESULTS AND DISCUSSION

3.1   Optical Micrograph Observation

Optical micrograph of heat treated samples for all condition shows that the microstructure remain as austenitic dendritic structure. However, grain coarsening was observed as the solution treatment temperature increased. Figure 3.1 and 3.2 shows the optical micrograph for both water quenched and air cool samples.
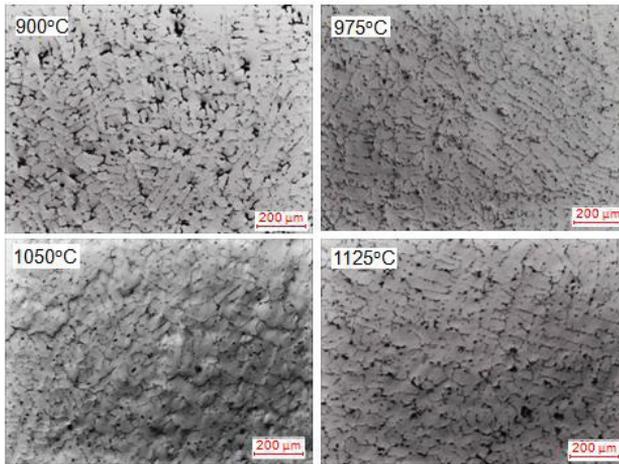
Figure 3.1 : Optical micrograph of water quenched samples
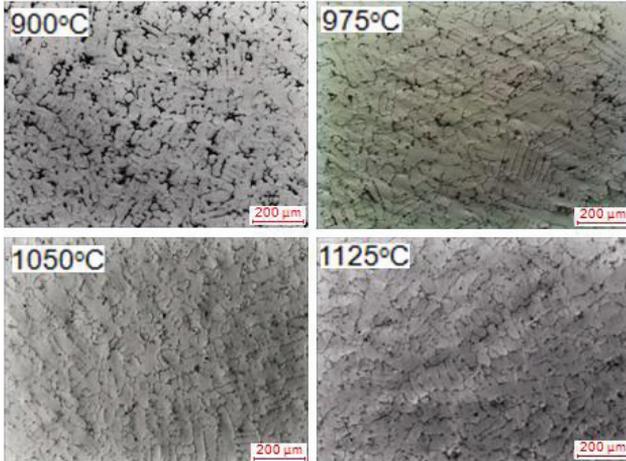
FIGURE 3.2
Optical micrograph of air cooled sample

Due to its dendritic structure, the grain size measurement was conducted by measuring the dendritic arm spacing (DAS). Figure 3.3 shows the graph plotted on the DAS of heat treated samples. All samples show an increasing trend of DAS as temperature increase. This is because grain size is controlled by thermally activated process through the migration of grain boundary where the free energy of the boundaries acts as the major driving force for grain growth (Koul, 1982). Thus, an increase in solution temperature provides more free energy to the boundary which promote grain growth and results to grain coarsening.

In terms of cooling rate, Figure 3.3 shows that water quenched samples show finer grain size compare to air cooled sample. This behavior could be related to principle of grain refinement by accelerated cooling. Grain size is determined by the number of nuclei until transformation is complete. Thus, large number of nuclei will caused the grain size become finer; meanwhile, small number of

nucleation will caused the grain become coarser. During continuous cooling, transformation occurred at certain temperature range. This range decreases with an increase in cooling rate. Thus, when cooling rate increase, grain size will decreased due to the increase in nucleation rate and decrease in growth rate. As cooling rate increases, the growth kinetic also become sluggish which causes the growth rate to decrease. This theory explained the reason of fine grain after accelerated cooling. Furthermore, as cooling rate decrease, the grain has sufficient time to grow and produces growth that is larger than fast cooling rate. As a result, the grain size of slower cooling rate samples is larger as compare to fast cooling rate.
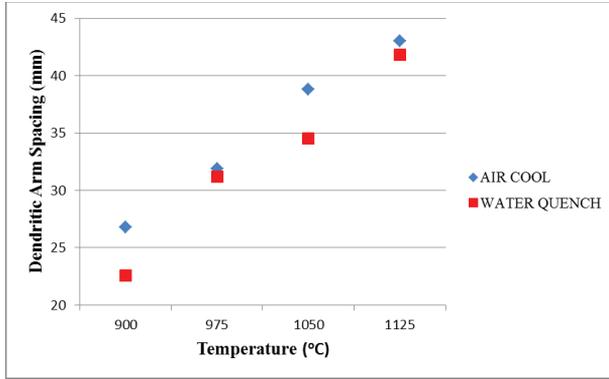
FIGURE 3.3
Effect of cooling rates on Dendritic Arm Spacing (DAS)

SEM micrograph of all samples shows presence of chromium carbide, $Cr_{23}C_6$ which determined by EDX analysis. Figure 3.4 shows the EDX analysis of $Cr_{23}C_6$. SEM micrograph of heat treated samples was shown in figures 3.5 and 3.6.
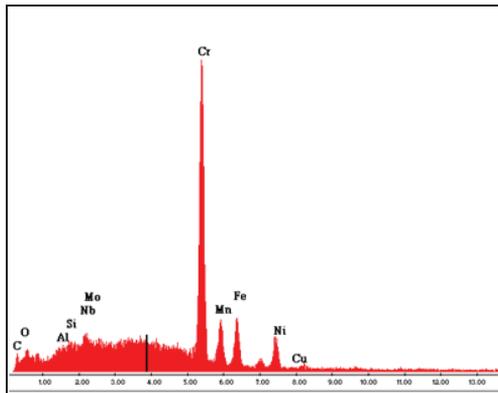
FIGURE 3.4
EDX analysis of $Cr_{23}C_6$
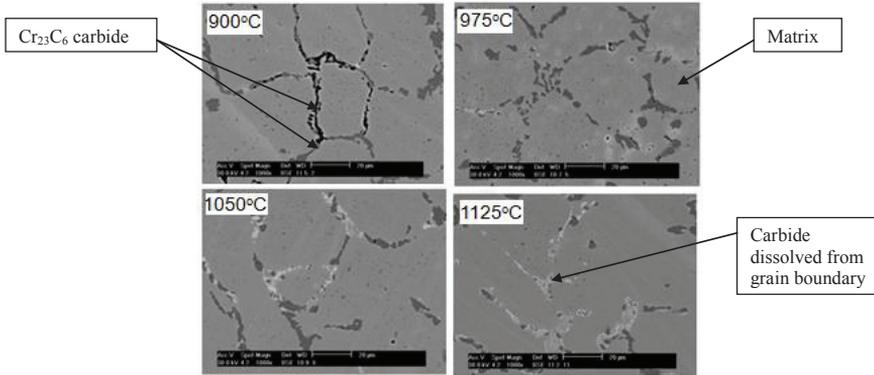
FIGURE 3.5
SEM micrograph of water quenched samples
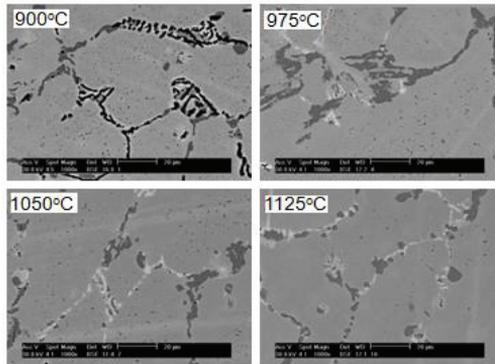
FIGURE 3.6
SEM micrograph of air cooled samples

Volume fraction analysis was conducted by using image analyzer to obtain an accurate volume of the carbide phase. The amount of carbide phase observed for water quenched and air cooled samples was decreased (dissolved from grain boundary) as solution temperature increased, this finding is similar as being observed by Jacuinde et.al. (2003).The reason for this behavior is at higher temperatures (above 1000°C), austenite tend to dissolve higher carbon contents, thus, the amount of secondary carbides retained is also minor (Jacuinde et.al, 2003). Meanwhile, in terms of different cooling rate, a curve is plotted to study the effect of cooling rate on precipitation of chromium carbide.

Figure 3.7 shows the effect of different cooling rates on volume fraction of retained carbide.
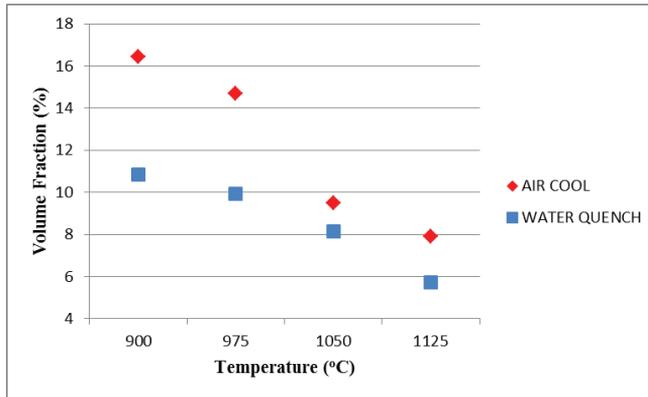
FIGURE 3.7
Effect of cooling rates on volume fraction of chromium carbide, $Cr_{23}C_6$

The result shown in Figure 3.7 illustrates higher carbide content in air cooled sample than water quenched sample. Larger differences of carbide content are noticed at temperatures 900°C and 975°C where the differences become smaller as the temperature increases.

Fast cooling rate from water quench produced lower carbide content because during fast cooling from solutioning temperature, the carbide did not have enough time to form and grow completely. This was due to suppression process that prevented the dissolved carbide to precipitate again when subjected to fast cooling. In fact, one of the main reasons to apply quenching was to minimize precipitation effects during cooling.

Meanwhile, slower cooling rate produced higher amount of carbide content because during cooling the carbide had enough time to form and grow after being dissolved during solutioning. However, in certain cases, this method is not preferable since the precipitation process cannot be controlled and might produced uneven precipitation.

However, it should be noted that chromium carbide is a very hard phase and would not easily dissolved due to heating, especially at low temperature. Thus, at lower heating temperature, the carbide dissolution was not complete and thus some carbide remain in the material which during cooling will continue to grow provided significant time is given. This could be the reason of large differences between air cool and water quenched sample at lower solution temperature as compare to high temperature since for water quenched sample, the carbide did not have enough time to precipitate compare to air cooled sample.

Hardness results of water quenched and air cooled samples were shown in figure 3.8 and 3.9 respectively. Generally, for both water quenched and air cooled samples, the hardness value was decreased as solution temperature increase. This finding is similar as being observed by Wang (2009).
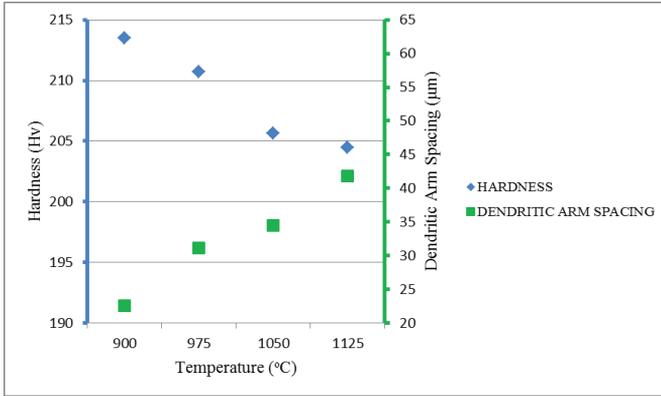
(a)
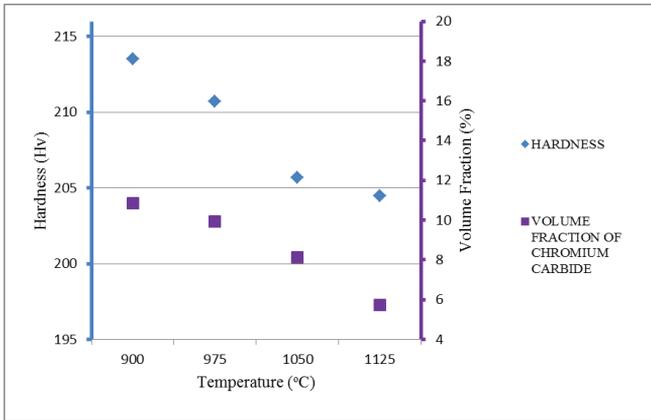
(b)

FIGURE 3.8
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
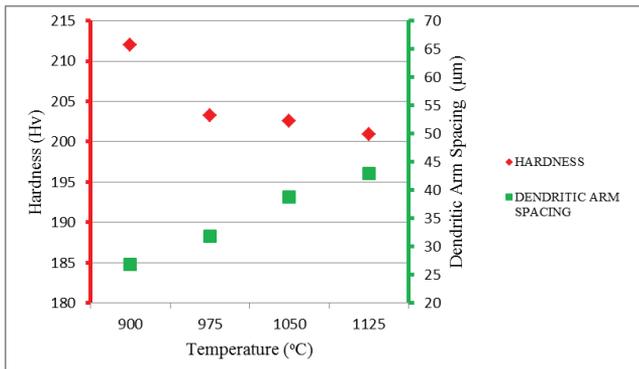
(a)
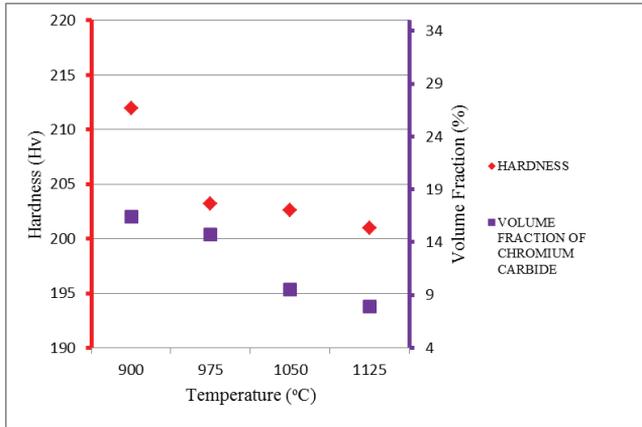
(b)

FIGURE 3.9

Effect of temperature on (a) hardness and DAS of air cooled samples (b) hardness and volume fraction of chromium carbide ($Cr_{23}C_6$) of air cooled samples

As shown in Figures 3.8(a) and 3.9(a), the increment in grain size of both water quenched and air cooled samples is inversely proprtional to the hardness value. This shows that grain coarsening occured due to increases in temperature had lower the hardness of material. This is because increase in grain size had reduced the grain boundary strengthening effect (Koul, 1982). Meanwhile, finer grain causes more build up of dislocation at the grain edge. Since it requires a lot of energy to move dislocation to another grains, this dislocation will build up along the boundary which increase strength of alloy (Zhang et.al, 2008). This strengthening theory also known as Hall-Petch strengthening. Thus, Hall-Petch strengthening effect is reduced as grain size increased.

On the other hand, carbide content also affects the hardness of material. Figure 3.8 (b) and 3.9 (b) shows that hardness of heat treated sample was decreases as carbide content decrease. This finding is similar as being observed by Jacuinde et.al, 2003. This is due to presence of carbide precipitates will increased yield strength of alloy by blocking dislocation within the crystal (Orlova, 1981). Since dislocation can only move through this particle either by shearing or climbing, thus, this interruption in dislocation motion increase the strength of material.

In terms of different cooling rate effects, Figures 3.10(a), 3.10(b), and 3.10(c) shows the effect of cooling rate on the grain size, carbide content, and hardness of material respectively. Figure 3.10(a) shows that there is a slight increase in grain size of air cooled sample as compare to water quenched sample. In other word, it can be said that slower cooling rate after solution treatment allowed more grain coarsening to take place as compared to fast cooling rate. This behavior could be related to principle of grain refinement

by accelerated cooling. Grain size is determined by the number of nuclei until transformation is complete. Thus, large number of nuclei will caused the grain size become finer; meanwhile, small number of nucleation will caused the grain become coarser. During continuous cooling, transformation occurred at certain temperature range. This range decreases with an increase in cooling rate. Thus, when cooling rate increase, grain size will decreased due to the increase in nucleation rate and decrease in growth rate. As cooling rate increases, the growth kinetic also become sluggish which causes the growth rate to decrease. This theory explained the reason of fine grain after accelerated cooling. Furthermore, as cooling rate decrease, the grain has time to grow and produces growth that is larger compare to fast cooling rate. As a result, the grain size of slower cooling rate is larger as compare to fast cooling rate.

Figure 3.10(b) illustrates higher carbide content in air cooled sample than water quenched sample. Larger differences of carbide content are at temperatures 900°C and 975°C. As the temperature increases, the differences become smaller. Fast cooling rate from water quench produced lower carbide content because during fast cooling from solutioning temperature, the carbide did not have enough time to form and grow completely (Stallybrass et.al, 2004). This was due to suppression process that prevented the dissolved carbide to precipitate again when subjected to fast cooling. In fact, one of the main reasons to apply quenching was to minimize precipitation effects during cooling.

Meanwhile, slower cooling rate produced higher amount of carbide content because during cooling the carbide had enough time to form and grow after being dissolved during solutioning (Das et.al, 2003). However, in certain cases, this method is not preferable since the precipitation process cannot be controlled and might produce uneven precipitation. However, it should be noted that chromium carbide is a very hard phase and would not easily dissolved due to heating, especially at low temperature. Thus, at lower heating temperature, the carbide dissolution was not complete and thus some carbide remain in the material which during cooling will continue to grow provided significant time is given. This could be the reason of large differences between air cool and water quenched sample at lower solution temperature as compare to high temperature since for water quenched sample, the carbide did not have enough time to precipitate compare to air cooled sample.

Figure 3.10(c) shows hardness of alloy undergone water quench is slightly higher compared to air cool. Highest hardness value which is 213.5 Hv was recorded at 900°C. This is due to the effect of finer grain size for water quenched sample. Thus, water quenched sample possess higher grain boundary strengthening effect compare to air cooled sample. In addition, precipitation strengthening effect also plays a significant role here. Even though water quenched sample contained lower amount of carbide precipitates, the carbide formed on the microstructure is finer as compared to air cooled sample (Lu et.al, 1995). This is because, during fast cooling, the precipitate did not have enough time to grow as compared to slower cooling rate where the precipitate will grow as there was enough time for the process. This fine carbide precipitates have

better strengthening effect compare to coarse precipitate. These two theories explained the reason of higher hardness and strength of fast cooling rate as compare to slow cooling rate samples.
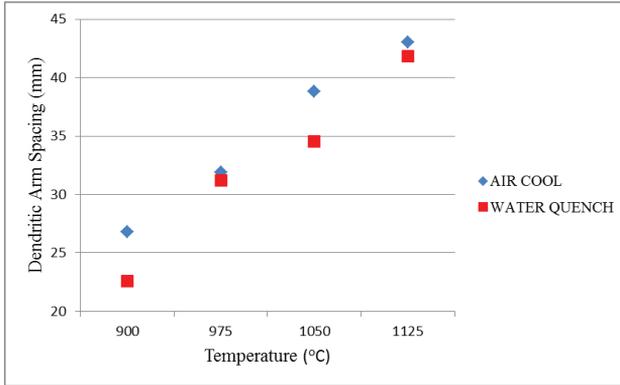
(a)
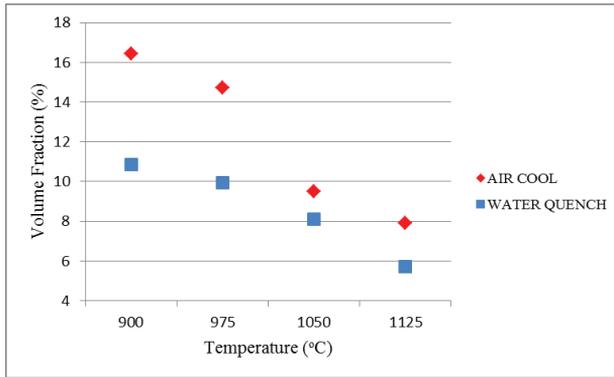
(b)
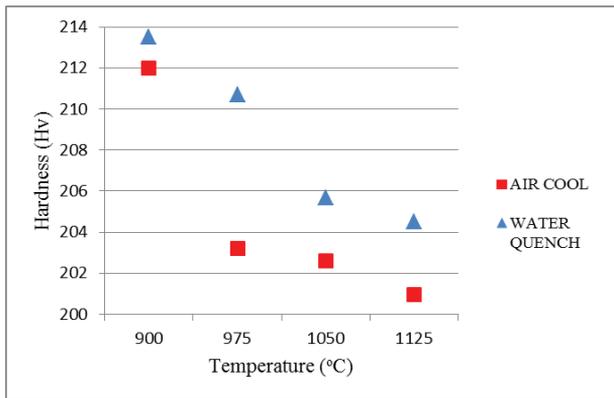
(c)

FIGURE 3.10
Effect of different cooling rate on (a) grain size (b) carbide content (c) hardness of Fe-Ni-Cr superalloy

In addition, XRD analysis shows that there is no phase formation or phase transformation occurred during heat treatment with different cooling rate. Figure 3.11 shows the XRD pattern of water quenched and air cooled samples.
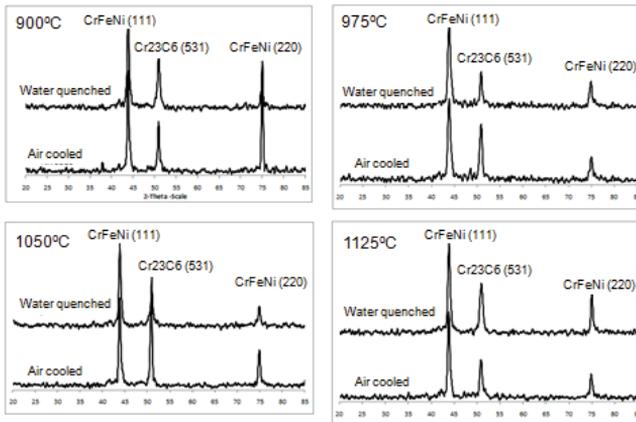
FIGURE 3.11
XRD pattern of water quenched and aged sample

4.0   CONCLUSION

The effects of cooling rate during heat treatment on microstructure and hardness of Fe-Ni-Cr superlloy have been investigated. Analysis of the results leads to few major findings. In terms of solution treatment, increase in temperature leads to increase in grain size but reduce the chromium carbide, $Cr_{23}C_6$ content and hardness value. In addition, grain coarsening behavior lower the hardness value due to its low grain boundary strengthening effect. Higher amount of fine chromium carbide in microstructure increase the hardness value due to precipitation strengthening effect while reduce amount of fine chromium carbide lower the hardness value. In terms of cooling rate, fast cooling rate produces slightly finer grain as compare to slow cooling which result to higher hardness. In addition, fast cooling rate also reduce the amount of chromium carbide precipitate by suppression due to heating. Highest hardness value which is 213.5 Hv was recorded on sample solution treated at 900oC followed by water quenched. XRD pattern shows that there is no phase formation nor phase transformation during all series of heat treatment.

5.0   ACKNOWLEDGEMENTS

6.0   REFERENCES

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