THE RELATIONSHIP BETWEEN HEAT-TREATED MICROSTRUCTURES AND MECHANICAL PROPERTIES IN CAST IRON-BASE ALLOY

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VZÁJOMNÝ VZŤAH MEDZI MIKROŠTRUKTÚROU A MECHANICKÝMI VLASTNOSŤAMI ODLIEVANEJ ZLIATINY NA BÁZE ŽELEZA PO TEPELNOM SPRACOVANÍ

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Abstrakt

Cieľom príspevku je analyzovať vplyv rôznych podmienok tepelného spracovania na vývoj mikroštruktúry a mechanické vlastnosti zliatiny Fe-30.8 Ni - 26.6 Cr, ktorá je spevnená precipitáciou karbidickej fázi. Východzí stav študovanej zliatiny bol systematicky podrobený starnutiu pri teplotách 800, 900, 1000 a 1100°C využijúc rôzne dlhé časy. Po procese starnutia bolo zistené, že sekundárne karbidy prednostne precipitovali v blízkosti sieťovia primárnych karbidov, ktoré sú na báze chrómu, prípadne komplexné na báze niób/titán. Precipitácia sekundárnych karbidov bola taktiež pozorovaná vo vnútri dendritov. Množstvo karbidov vylúčených vo forme tyčiniek a taktiež sekundárnych karbidov, narastá s predlžovaním času a zvyšovaním teploty starnutia. Hoci ako ukázali analýzy využijúc metódu EDS, chemické zloženie sekundárne vylúčených karbidov bolo takmer rovnaké ako chemické zloženie primárnych karbidov. Vo všeobecnosti je možné tvrdiť, že zvolené podmienky tepelného spracovania mali významný vplyv na tvar, veľkosť, rozloženie a miesto vylúčenia sekundárnych karbidov v mikroštruktúre vzoriek, čo malo následne vplyv na rozdielne mechanické vlastnosti, ako tvrdosť, medza klzu a medza pevnosti. V prípade starnutia vzoriek pri 800 a 900°C boli pozorované veľmi jemne precipitáty sekundárnych karbidov, ktoré boli vylúčené v oblastiach blízko pri primárnych karbidov. Ak bolo použité starnutie pri 1000 a 1100°C, tak hrubšie sekundárne karbidy boli vylúčené vo vnútri dendritov. Karbidy vo forme tyčiniek a filmu boli pozorované v mikroštruktúre vzoriek tepelne spracovaných pri 900, 1000 a 1100°C. Sekundárne karbidy, ktoré boli na báze chrómu, precipitovali po rôznych zvolených podmienkach tepelného spracovania, pričom chemické zloženie týchto karbidov je podobné chemickému zloženiu primárnych chrómových karbidov. Vo všeobecnosti je možné konštatovať, že rovnomerné vylúčenie jemných sekundárnych karbidov spôsobuje vyššie hodnoty medze pevnosti, medze

klzu, ako aj tvrdosti. Hoci je potrebné aj podotknúť, že v niektorých prípadoch výsledky testov mechanických vlastností neukazujú na výrazný vplyv podmienok starnutia, obzvlášť v prípade ťažnosti a „modulu húževnatosti“. Ako sa ukázalo, najvhodnejšími podmienkami tepelného spracovania na zvýšenie medze pevnosti je starnutie pri teplote 1100°C po dobu 10 hodín.

Abstract

This work has an aim to study and investigate the relationship between heat treatment conditions on microstructural evolution and mechanical properties in the iron-based alloy, Fe-30.8 Ni -26.6 Cr alloy, strengthened by carbide precipitation. Various aging temperatures (800, 900, 1000 and 1100°C) with various aging times are systematically introduced to the as-received alloy. After aging, it was found that the secondary carbides precipitated early near the primary carbides, which are chromium and niobium/titanium carbide networks. The secondary carbide precipitations were also found in the dendrite cores. The amounts of needle-like carbides and secondary carbide films increased with time and temperature of aging. However, by EDS analysis, the composition of secondary carbides was almost the same as that of primary carbides. It can be summarized that the heat treatment conditions have greatly effect on shape, size, dispersion and the location of secondary carbides in microstructure and result in the different mechanical properties such as hardness, yield strength and tensile strength. Aging at 800 and 900°C, the very fine precipitates of secondary carbide particles locate and concentrate in the area close to primary carbide. Aging at 1000 and 1100°C, the coarser secondary carbides disperse to the cores of dendrites. The needle-like and film carbides were found in heat-treated specimens at 900, 1000 and 1100°C. The precipitated secondary carbides precipitated after various heat treatment conditions are chromium carbide, which its chemical composition is similar to primary chromium carbide. It could be concluded that the uniform precipitation and dispersion of fine secondary carbides result in the higher ultimate tensile and yield strengths as well as hardness. However, the obtained result of some mechanical tests did not show any significant effect of aging conditions, especially in ductility and modulus of toughness. The most proper heat treatment condition to maximize tensile strength is aging at 1100°C for 10 hours.

Keywords: Iron-base alloy, Heat treatment, Aging, Carbide precipitation, Mechanical properties, microstructure

1. Introduction

The cast iron base alloys are widely used in the petrochemical industries, especially under conditions of long-term exposed at high temperatures in the range of 850 - 1150°C. The low to medium strength at high temperature is not the only one requirement but also the good resistance to surface degradation at such high temperatures such as hot oxidation and corrosion as well as good resistance to thermal fatigue. Most of the alloys in this Fe-Ni-Cr system contain chromium about 15 wt. % to improve surface degradation resistance at elevated temperatures as well as Nickel about 25 wt. % to stabilize the austenitic structure for good strength at high temperatures.

The Fe-30.8Ni-26.6Cr alloy, one of Iron-base alloy produced in the form of centrifugally cast tubes, is mainly used in petrochemical industry as material in reformer and pyrolysis furnaces. This alloy has been used instead of expensive superalloys with sufficient high temperature properties such as creep strength. Therefore, many microstructural features have been developed in order to increase high temperature strength in material. The designed

microstructure would contain a uniform coarsening grain size with beneficial segregation or particle dispersions such as fine and discontinuous carbides of the grain boundaries resulting in higher creep strength and crack growth resistance as well as for fatigue strength [1]. The Fe-30.8Ni-26.6Cr alloy is not a standard alloy, which can be simply classified in HP alloy group but it can be compared to another non-standard HP-35Ni-25Cr alloy. This non-standard iron alloy group consists of more additional elements. These additional elements can be defined as carbide forming elements: niobium, molybdenum, titanium, tungsten and zirconium as well as non-carbide forming elements: aluminum, copper and cobalt for an increase in strength and resistance to carburization. The addition of silicon in very high amount can improve carburization resistance but decreases creep and rupture strength. The carbon content must be carefully controlled to provide strength as carbide precipitation strengthening. However, too much carbon content would decrease the resistance to cyclic thermal shock [2].

Generally, such kind of nickel-rich iron-base alloy is much better in phase stability than other iron-base alloys, which contain lower nickel content. The alloy can be used at elevated temperatures without phase transformation to the brittle sigma (σ) phase during long-term service or processing. The HP iron-base alloy is austenitic structure at all temperatures thus it is not sensitive to σ phase transformation after long-term expose. The desired structure of the alloy could be achieved by heat treatment process by means of modified microstructures via a precipitation process of primary and secondary carbides under aging conditions. A number of studies were done subsequently to evaluate the phase changes during heat treatment process and the influence of its microstructure to mechanical strength at both room and elevated temperatures. An improved knowledge of this relationship leads to the development of new aging condition. The tube production using the centrifugal casting technique provides the higher creep properties through the morphological modifications in microstructure and the presence of more stable phases during long-term exposure. The primary eutectic-like carbide network appears to play an important role in resisting grain boundary sliding. Secondary precipitation of fine cube-shaped chromium carbides should act as barriers to dislocation movements.

However, there are still very few studies on the microstructural evolution in the Fe-38.8Ni-26.6Cr alloy to increase mechanical properties by various heat treatment conditions including the investigation of precipitation of secondary carbides and other phase transformations. Therefore, this research study provides an attempt to achieve the optimal microstructure characteristic and mechanical properties. The aging heat treatment programs were systematically performed in the as-received alloy after long-term use. The obtained specimens after various aging conditions were investigated and analyzed.

2. Material and Experimental Procedure

The Fe-38.8Ni-26.6Cr alloy has a chemical composition by wt. % as shown in Table 1. The as-received alloy was produced by the casting process. The initial alloy was still not proper microstructure and does not have good mechanical properties as desired after this manufacturing. Thus, the following heat treatment is necessary to fulfill the material requirements. Therefore, various conditions according to the tested program were carried out to the alloy as following:

1) Aging at 800°C for 1, 3, 10 and 24 hours;
2) Aging at 900°C for 1, 3, 10 and 24 hours;
3) Aging at 1000°C for 1, 3, 10 and 24 hours;
4) Aging at 1100°C for 1, 3, 10 and 24 hours.

Finally, all tested specimens were observed and analyzed by an optical microscope and scanning electron microscope. To find out the mechanical properties then hardness and tensile tests were carried out.

Table 1 Chemical composition of the alloy (by wt.%; analyzed by Emission Spectroscopy)

C	Si	Mn	Cr	Ni	Cu	Co	Al	Nb	Ti	V	Pb	W	Fe
0.3	1.43	1.40	26.6	30.8	0.05	0.17	0.003	0.68	0.054	0.049	0.005	0.25	38.2

3. Results and Discussion

3.1 Microstructure investigation

3.1.1 Microstructure of as-received alloy

The received microstructure consists of primary carbide networks in austenitic matrix, as shown in Fig. 1. The dendrite structure indicates the characteristic of casting microstructure. However, no secondary carbide was detected in the microstructure, see Fig. 2. From SEM analysis, it was found that the primary carbide networks could be classified in two types as black and white phases, Fig. 3. Using EDS to analyze the chemical composition of each phase is concluded that the black phase consists of 70.59 % of chromium and white phase consists of 19.93 % titanium, 32.60 % niobium and 0.61 % chromium.

The matrix consists of 35.74 % iron, 31.59 % nickel, and 24.54 % chromium, see Table 2. The alloy consists of 30.8 % nickel, which is high enough to stabilize the austenitic matrix microstructure. The primary carbide networks could form during slow cooling of solidified alloy by the combination of carbon and chromium, niobium and titanium. Titanium and niobium would form as niobium-titanium carbide, which precipitated at higher temperature comparing to chromium carbide resulting in high ratio between Cr and C [3]. Therefore, the presence of primary carbide precipitation type is $M_{23}C_6$ [4]. They locate near austenitic grain boundary networks.

Fig.1 Microstructure of as-received material

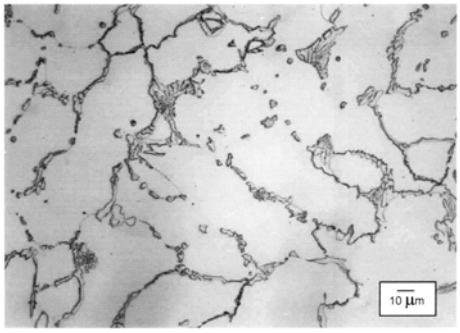

Fig.2 Microstructure of as-received material

Table 2 EDS analysis of as-received alloy (by wt.%)

Phase	Si	Ti	Cr	Fe	Ni	Nb	C, Co & W
White phase	1.7	19.93	0.61	23.20	21.88	32.60	0.07
TiC (2)	0.14	60.20	5.89	0.75	0.54	0.43	32.06
Black phase	0.85	-	70.59	15.03	7.7	2.29	1.36
Matrix	2.78	-	24.54	35.74	31.59	1.87	0.24

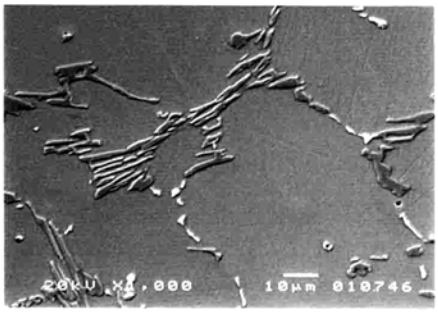

Fig.3 SEM micrograph of as-received material

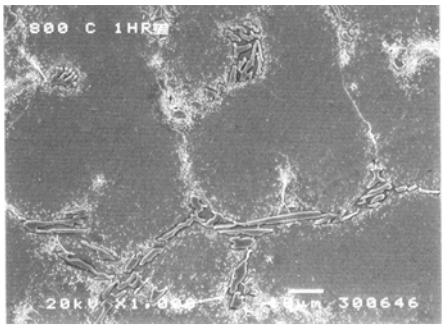

Fig.4 Microstructure of specimen after aging at 800°C for 1 hour

3.1.2 Microstructure of the alloy after heat treatment

From SEM micrographs, generally, all microstructures after various heat treatment conditions were found in similar manner. Most of microstructures consist of primary carbides as the as-received microstructure. However, very fine precipitations of secondary carbides were found locating in the matrix, usually, in areas close to primary carbides, Fig. 4. After aging at 800°C, the secondary carbide particles concentrate in the zones adjacent to the primary carbides. The amount of secondary carbide particles increase with time of heat treatment (10 and 24 hours), as shown in Fig. 5. Furthermore, the film and needle-like carbides were also observed. It should be noted that these secondary carbide particles precipitate in higher concentration near the primary carbide particles and more precipitation disperse toward the dendrite core when aging time increase.

After aging at 900°C for various aging time, the heat-treated microstructures are similar to those aged at 800°C but are different in amounts of secondary carbide precipitation. After short-term heat treatment (1 and 3 hours), secondary carbide are in high concentration near primary carbides. However, when aging time was increased up to 10 and 24 hours, the previous precipitation of secondary carbide particle would agglomerate to become in coarser sizes and there are more very fine secondary carbides in the center of dendrite core, Fig. 6. Coarsen needle-like and film carbides are found as well.

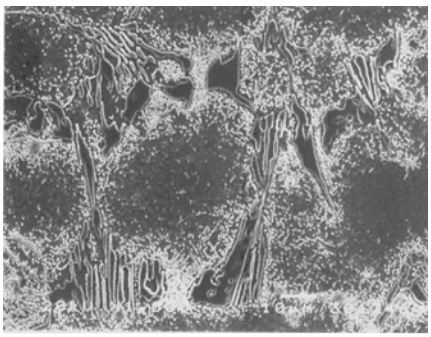

Fig.5 Microstructure of specimen after aging at 800°C for 24 hours

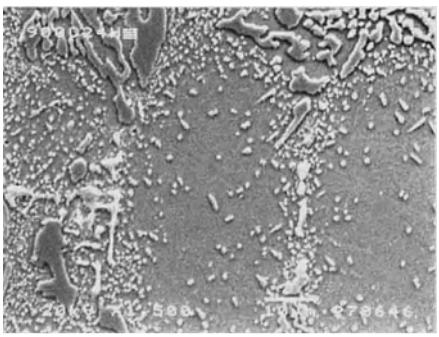

Fig.6 Microstructure of specimen after aging at 00°C for 24 hours

The microstructures, after aging at 1000 and 1100°C for various aging time, are quite similar to those aged at 800 and 900°C, Figs. 7 and 8. The secondary carbides are in round shape

and precipitate toward the dendrite core. The secondary carbides are in high concentration to the primary carbides in case of exposed time of 1, 3 and 10 hours. For aged microstructure for 24 hours, very fine particles of secondary carbide would agglomerate as coarsening size. However, using SEM investigation in all cases, precipitates free zones (PFZ) were found close to primary carbides because of low chromium content in these areas, where chromium precipitated during previous secondary carbide precipitation.

Fig.7 SEM micrograph of specimen after aging at 1000°C for 10 hours

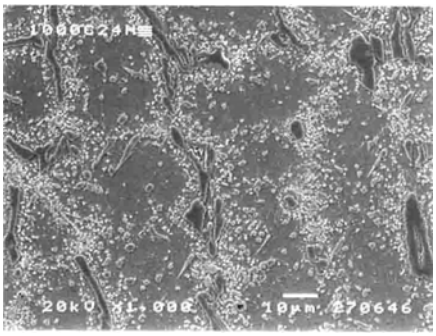

Fig.8 SEM micrograph of specimen after aging at 1000°C for 24 hours

From EDS analysis of primary and secondary carbides as well as in matrix, it is summarized that the amounts of chromium in the matrix decreases after all heat treatments as chromium forms the secondary carbides. However, amounts of other elements in matrix are quite constant, see Table 3. Furthermore, it was also found that the amount of chromium (70.72 %) in secondary carbides is very close to that of primary carbide (the black phase). This could imply that no significant phase change in primary carbide after all heat treatment but only loses some amount by the decomposition of partial primary carbides to secondary carbides. For the needlelike secondary carbide, its chemical composition is as following: 37.86 % Fe, 32.33 % Ni, and 27.92 % Cr, as can be seen in Table 3.

Table 3 EDS analysis of individual phases after aging at 1000°C for 24 hours (by wt.%)

Elements	Si	Cr	Mn	Fe	Ni	Nb	C, Co & W
Secondary carbide	0.85	70.72	-	14.86	11.03	0.87	1.64
Needle-like carbide	1.69	27.92	-	37.86	32.33	-	0.04
Matrix	5.46	29.07	1.46	35.36	29.73	1.11	0.10

After all heat treatments, the chemical composition of primary carbide is almost constant due to no phase transformation, see Tables 2 and 3. However, the another type of primary carbide (the white phase), which consists of 32.6 % Nb, 0.61 % Ti, 1.7 % Si, and 21.88 % Ni, the amount of niobium has a trend to decrease while the amounts of nickel and silicon increase after aging at 800, 900, 1000, and 1100°C for 24 hours. This might be due to the occurrence of phase instability during long-term aging at high temperatures. Finally, it is summarized that temperature and time of aging have significant effect on size, shape, and dispersion of secondary carbides. The most precipitated secondary carbides are the same type as primary carbide, $M_{23}C_6$ [2 - 12]. Furthermore, niobium and chromium also influence the shape of secondary carbides as well [8, 11]. High amount of niobium and chromium in iron base alloy

induces to needle-like secondary carbide formation at long-term exposed conditions. Therefore, the control of niobium and chromium is very important. The proper and careful control of amount of both elements reduces the tendency of needle-like carbide formation, which causes the brittle fracture later.

Aging time and aging temperature, especially in the range of 800 - 1000°C, can decrease amount of niobium in niobium/titanium carbide while increase amount of nickel and silicon, Table 4. In this temperature range of 800 - 1000°C, niobium/titanium carbide is not stable and susceptible to the transformation of G-phase or nickel-niobium-silicide ($Ni_{16}Nb_7Si_{16}$) according to previous study [13]. However, the G-phase transformation would rarely occur partially because titanium could inhibit the phase transformation. The G-phase probably is considered as the weak point for creep-rupture strength.

Table 4 EDS analysis of primary carbides after aging for various aging time (by wt.%)

Specimen	Si	Cr	Ti	Fe	Ni	Nb	C, Co & W
As -received	1.7	19.93	0.61	23.2	21.88	32.6	0.07
800°C/ 24 hrs.	2.14	22.25	0.61	25.97	24.63	24.17	0.09
900°C/ 24 hrs.	5.56	19.94	0.74	12.89	29.96	30.65	0.22
1000°C/ 24 hrs.	3.23	21.63	0.68	21.94	22.78	29.6	0.15
1100°C/ 24 hrs.	1.29	16.99	0.81	24.36	21.16	35.32	0.08

3.2 Hardness tests

From results of hardness tests, it is summarized that the hardness of heat-treated specimens is higher than that of as-received one, Fig. 9. In most cases, there is an increase in hardness reaching to the peak value brought about by increased amounts of precipitates and changes in precipitate morphology, as mention previously. After the hardness peak is reached, the hardness slightly decreases with continuing particle growth of previously existing secondary carbides for longer aging time (10 and 24 hours). Therefore, it is concluded that the hardness strongly relates to morphology of secondary carbide. For heat treatment in the temperature range of 800 - 900°C, there are secondary carbides in finer size and fewer amounts than those in the temperature range of 1000 - 1100°C. Hence, the hardness results after the lower temperature aging are less than those of higher ones.

Furthermore, the micro-hardness tests of individual phases (matrix and primary carbide) were performed as seen in Tables 5 and 6 and Figs. 9 - 13. Figure 9 shows the effect of aging treatment in an increase of hardness of the material. No significant difference in micro hardness of primary carbide was observed between as received (350.6 HV25g) and heat-treated specimens, Figs. 10 and 11. This might be due to that the investigated areas for hardness tests are very small and no phase transformation of primary carbide occurs. However, it can be concluded that aging at 1000°C for 1 hour and aging at 1100°C for both 10 and 24 hours provided the highest micro hardness of primary carbide in this study. Furthermore, the amount, size and shape of precipitated secondary carbides have also very strong influence to the micro hardness of primary carbides too. Therefore, each aging temperature condition, which provides its own morphology of precipitated secondary carbides including its precipitation and agglomeration rates during aging, results in different values of micro hardness of primary carbides. Figures 12 and 13, the micro hardness of matrix slightly increases with aging times due to the more precipitation of very fine secondary carbides in matrix. Figure 12 shows that 24-hours aging time, no matter what the aging temperature, provides the stable and similar of micro

hardness results, which are very close to 200 HV 25g. The aging temperature of 1100°C provides the highest micro hardness of each aging temperature.

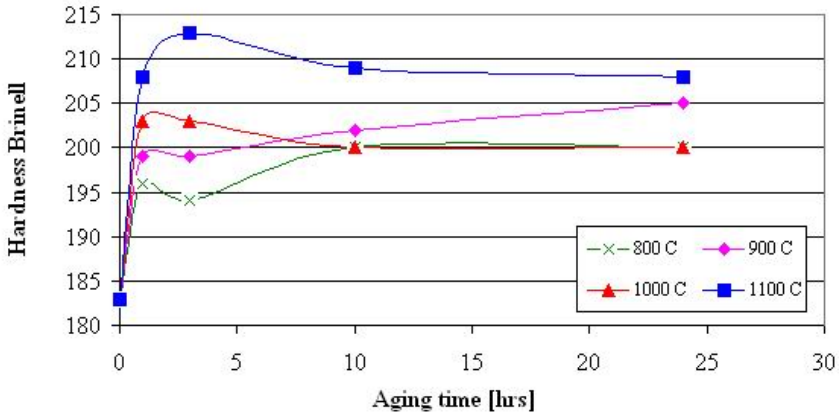

Fig.9 The relationship between hardness and aging time

Table 5 Results of micro hardness tests (HV25g) of primary carbide

Aging temperature [°C]	Aging time [hr.]			
	1	3	10	24
800	340	360	327	372
900	348	356	364	364
1000	378	358	351	343
1100	366	365	378	377

Table 6 Results of micro hardness tests (HV25g) of matrix

Aging temperature [°C]	Aging time [hr.]			
	1	3	10	24
800	164	169	189	181
900	167	172	181	180
1000	200	198	191	190
1100	226	215	215	195

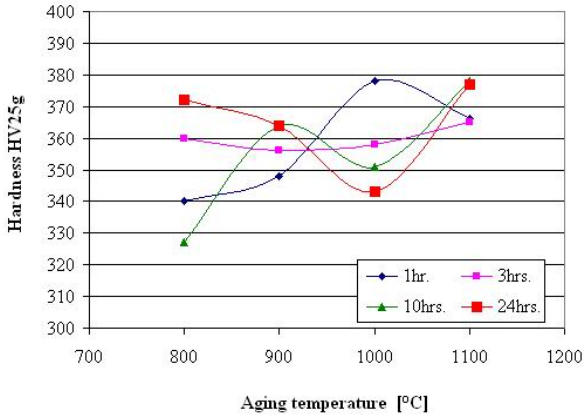

Fig.10 The relationship between micro hardness tests (HV25g) of primary carbide and aging temperature

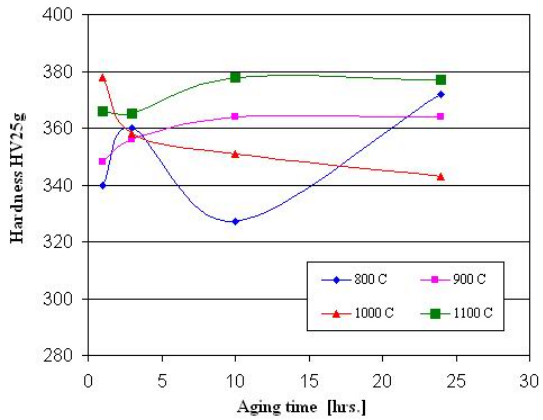

Fig.11 The relationship between micro hardness tests (HV25g) of primary carbide and aging time

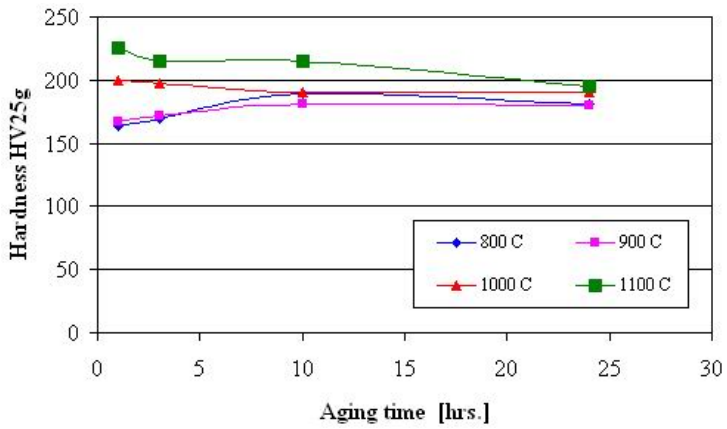

Fig.12 The relationship between micro hardness tests (HV25g) of matrix and aging time

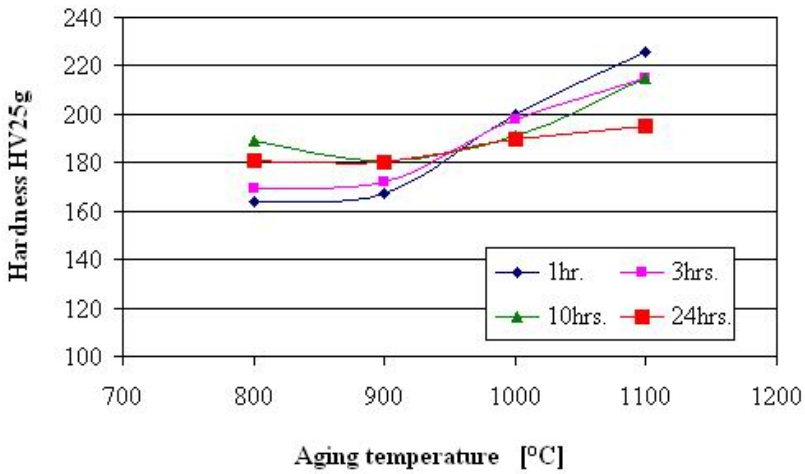

Fig.13 The relationship between micro hardness tests (HV25g) of matrix and aging temperature

3.3 Other mechanical tests

From the tensile test results in Fig. 14, the ultimate tensile strengths of specimens after all heat treatment are similar and are higher than that of the as-received one. The ultimate tensile strength of specimen aged at 800°C for 1 hour is the lowest due to fewer amounts of finer secondary carbide precipitated particles. Usually, the ultimate tensile strength slightly increases as time of aging increased at the same temperature. The uniform dispersion of fine secondary carbide particles has high efficiency pinning the movement of dislocations resulted in higher strength. In the case of aging at 900°C, ultimate tensile strength does not increase with aging time because of the higher precipitation of brittle needle-like carbides resulted in lower ultimate tensile strength than those of aging at 800°C. The ultimate tensile strength after aging at 1000 and 1100°C for 1 and 3 hours are higher than those after aging specimen at 800 and 900°C due to more dispersion and precipitation of secondary carbides. However, after longer aging time, the ultimate tensile strength slightly decreases due to the occurrence of coarser secondary carbide particles and films. It was also found that the ultimate tensile strength behavior has similar manner as the hardness behavior. Considering the yield strength in Fig. 15, it is seen that the yield strength has the same trend as the ultimate tensile strength.

Figure 16 shows the relationship between % ductility of tensile tests and aging time. The figure expresses that the % ductility of specimens after all aging temperatures is very similar in each the same aging time, which are about nearly to 10 %. The aging time, no matter how long is it, did not provide any significant difference of the results. It should be noted that the obtained results of % ductility did not change greatly comparing to that of the as-received specimen. After aging, the modulus of toughness (in the range of 30 to 50 J) is all lower than that of the as-received specimen (about 60 J), as shown in Fig. 17. The aging time did not importantly result in the difference of modulus of toughness of each the same aging temperature. Furthermore, it can be seen that aging at 1100°C evidently resulted in the highest value of modulus of toughness. The modulus of toughness of specimens after aging at 1000°C is clearly higher than those of aging at 800°C and 900°C, which are very similar. Figure 18 shows that % volume fraction of secondary carbides could not provide any significant effect on hardness and especially, on tensile strength.

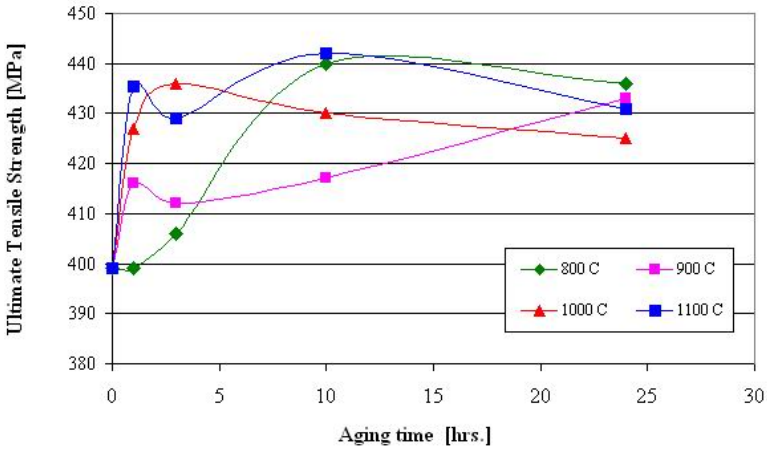

Fig.14 The relationship between ultimate tensile strength and aging time

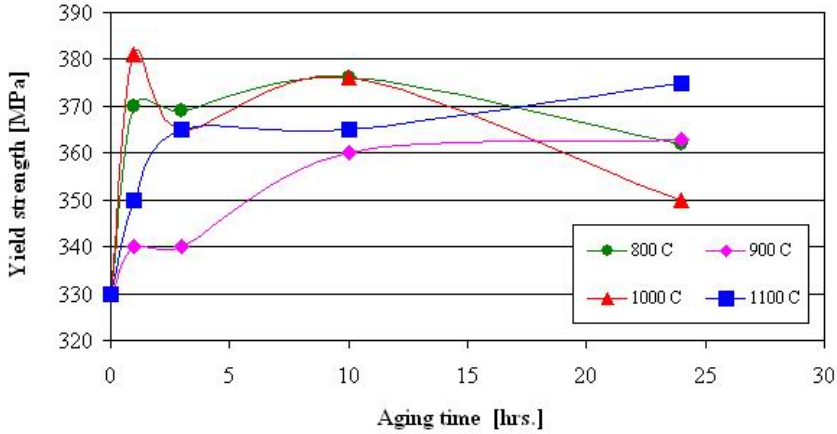

Fig.15 The relationship between yield strength and aging time

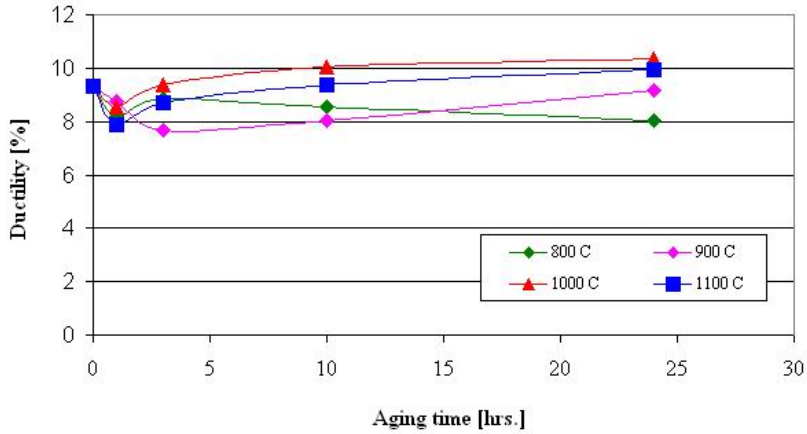

Fig.16 The relationship between % ductility and aging time

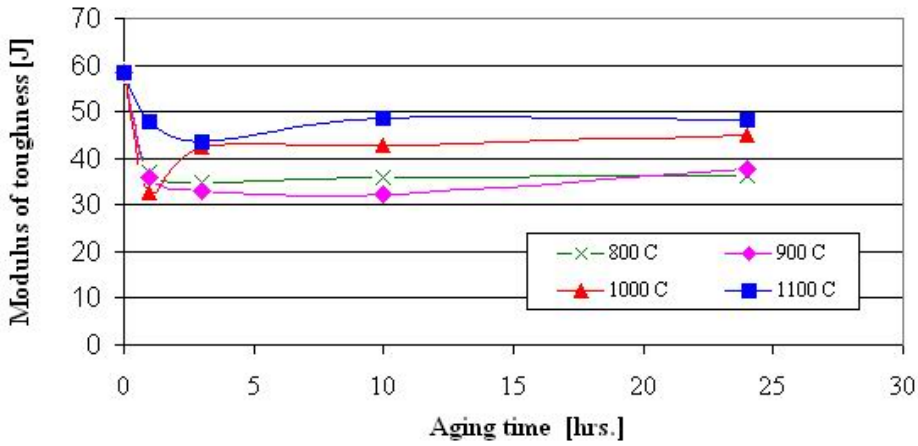

Fig.17 The relationship between modulus of toughness (J) and aging time

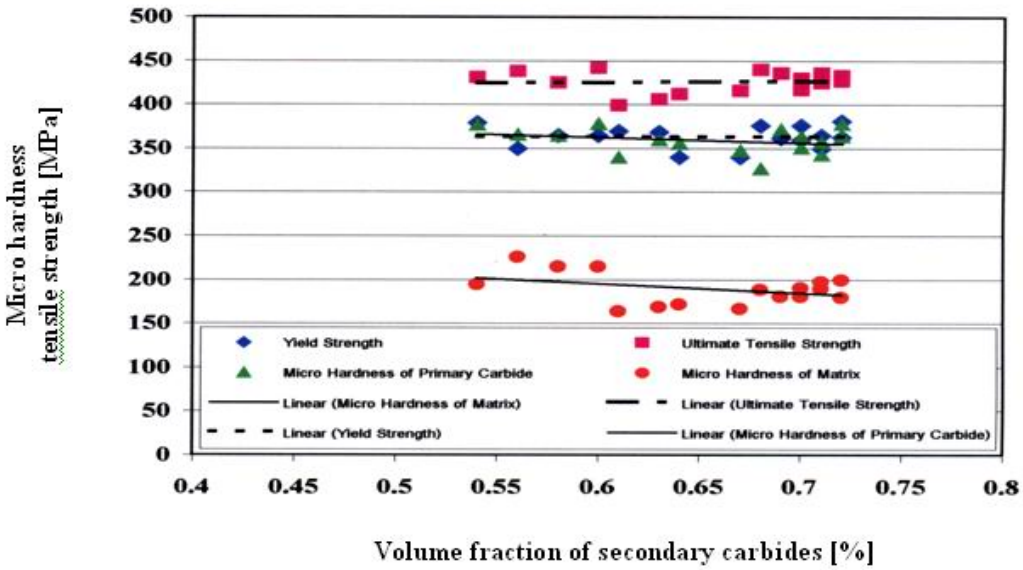

Fig.18 The relationship among volume fraction of secondary carbides, tensile strength and micro hardness

4. Conclusions

1. The secondary carbides precipitated after various heat treatment conditions are chromium carbide, which its chemical composition is similar to primary chromium carbide.
2. Size, shape and dispersion of secondary carbides depend on aging times and aging temperatures as follows:
   - 2.1 Aging at 800 and 900°C, the very fine precipitates of secondary carbide particles locate and concentrate in the area close to primary carbides.
   - 2.2 Aging at 1000 and 1100°C, the coarser secondary carbides disperse to the cores of dendrites.
   - 2.3 The needle-like and film carbides were found in heat-treated specimens at 900, 1000 and 1100°C.
3. The precipitation and dispersion of fine secondary carbides result in the higher ultimate tensile and yield strengths as well as hardness.
4. The most proper heat treatment condition to maximize tensile strength is aging at 1100°C for 10 hours.

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