research note

The structure-property relationship in a desulfurised and degassing hot work W500 tool steel

M. Kalantar[1]*, S.S. Ghasemi[2] and M.H. Saadati[3]
Department of Materials Engineering, Faculty of Mining and Metallurgy, Yazd University, Yazd, Iran

Abstract
The effect of secondary steelmaking processes such as desulphurization, removal of inclusions and vacuum degassing followed by hot forging and hardening heat treatment on the microstructure and mechanical properties of a hot-work w500 tool steel have been studied in details. In order to follow the progress of secondary steel making, the content of impurity elements such as S, P, O, H and N were measured. These elements influence the mechanical testing and the microstructure of the steel. The results show that desulfurization treatment can be accelerated at higher temperature of 1680 °C and 15 minutes holding time for silicon and aluminum with contents of 0.33% and 0.056% in the molten steel, respectively. In this condition, the removal percentage of sulfur has been reached to about 90% relative to the initial sulfur content. For the degassing sample A, the strength and the hardness, after hot working and quenching – tempering, have been increased from 976 to 2020 MPa and 29 to 52 R_C, respectively. Whereas for the normal sample B, the associated strength and the hardness have been changed from 870 to 1845 MPa and 21 to 55 R_C, respectively. The difference between mechanical properties of sample A and sample B cab be related to the presence of Al_2O_3 clusters, silicate inclusions, and a longer filamentary inclusion in the microstructure of sample B after hot-forging. Microstructural observations show that the morphology of pearlite in the forged sample A is more uniform and carbide particles are also much finer than these particles in the non-degassing forged sample B.

Keywords: secondary steel making, hot work W500 tool steel, heat treatment, mechanical properties, microstructure.

1. Introduction
Hot work tool steels are a group of heat treatable carbon and alloy steels with high stability of hot and cold mechanical working properties[1-5]. These types of steels, in general, are hypereutectoide ledburite containing carbides in the tempered martansitic matrix in which, hard carbide particles have been dispersed in the matrix in order to improve wear behavior of cold and hot working dies. The presence of strong carbide forming elements such as Cr, V, Nb, W and Mo along with high carbon content accelerate the formation of hard carbides in tool steels with a high hardness of 60-65 HRC. resulting a large fraction of the production cost for machining of dies[6-7]. Hot work tool steels are used for engineering applications including molds, punches, cutting and machining dies subjected to high thermal exposure as well as severe erosion conditions such as hot pressing, hot extrusion, hot forging, die casting and speed cutting tools[8-9]. These applications require high toughness, high resistance to thermo-mechanical stresses, good resistance to erosion and thermal fatigue[10-12]. Modern technology needs steels with a higher quality and the demands have been increased for steels with minimum inclusion, cavity, impurities such as phosphorous and sulfurous in which advanced physical and mechanical properties have been developed[13-16]. In this regard, the quality control of alloy steels has been improved by employing special techniques of secondary steelmaking treatments such as vacuum degassing, removing inclusion by blowing inert gas into the molten steel and deoxidizing by Al and Si with Ca-Si/Ca-Al agents. The purpose of this study is to investigate the effect of secondary steelmaking operations on the microstructure and mechanical properties of a developed hot work tool steel.

2. Materials and Experimental Methods
All experimental works related to the secondary steelmaking procedures were conducted in alloy steel company of Asfrayen. The samples of molten steel were taken from various stages of steel making process such as arc electric furnace, ladle furnace and vacuum degassers in different combination of melt chemical composition, holding temperature and time conditions. The chemical composition of the degassing (A) and the normal (B) steel samples were presented in Table 1.

** Corresponding author:*
Tel:+98 (913)3513418, TeleFax: +98(351)8210995
Email: Mkalantar@yazduni.ac.ir
Address: Department of Materials Engineering, Faculty of Mining and Metallurgy, Yazd University, Yazd, Iran.
1,2. Associate Professor
3. Alloy Steel Company of Sfrayen

Table 1. The Chemical composition of degasified (A) and non-degasified (B) W500 tool steel samples

Sample	C_{eq}	C	Si	Mn	P	S	Cr	Mo	Ni	V
A	1.15	0.58	0.26	0.74	0.01	0.01	1.18	0.47	1.48	0.066
B	1.13	0.57	0.28	0.73	0.023	0.029	1.16	0.48	1.46	0.06

The samples A and B were hot-worked in order to modify the size and the morphology of columnar grains with non-uniform inclusions as well as to reduce casting porositise and voids. The hot working process was carried out by radial and edge hot working treatment in two stages as schematically illustrated in Fig. 1 and detailed in Table 2.

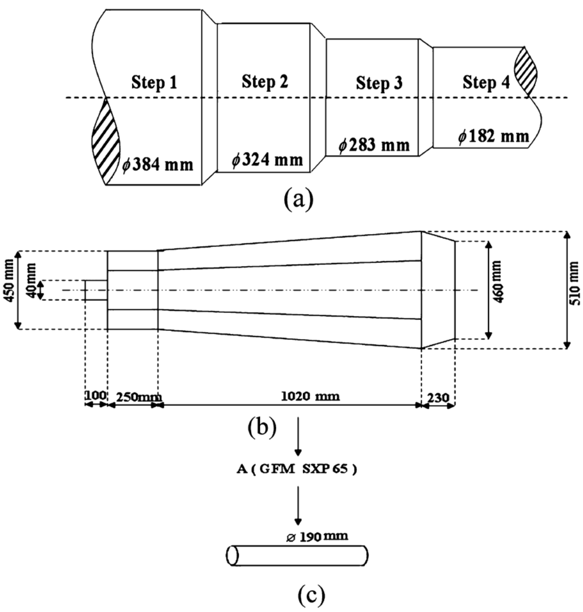


Fig. 1. The schematic representation of hot working process: (a) radial and edge hot- forging condition; (b)the initial sample; (c) the final hot-forged sample.

Table 2. Data related to Fig. 1(a).

Section	Diameter (mm)	Strain	RA%
1	384	0.71	51
2	324	1.05	65
3	283	1.35	74
4	182	2.23	84

A successive impact and homogeneous deformation were conducted during hot forging treatment. Before forging stage, the samples were homogenized according to the shawn cycle of Fig. 2.

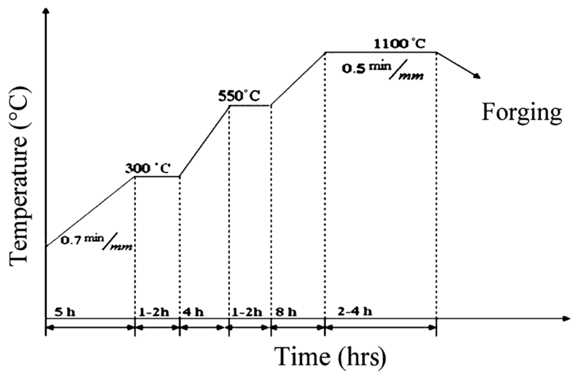


Fig. 2.The Heat treatment cycle of annealing before hot forging Stage.

The temperature of samples at the end of each forging stage should be at least 850 °C to avoid the formation of carbides continuously in grain boundaries. Because of low thermal conductivity, the hot forged samples were immediately placed in the annealing furnace to avoid initiate the thermal stress and micro-cracking on the subsequent cooling stage as shown schematically in Fig. 3.

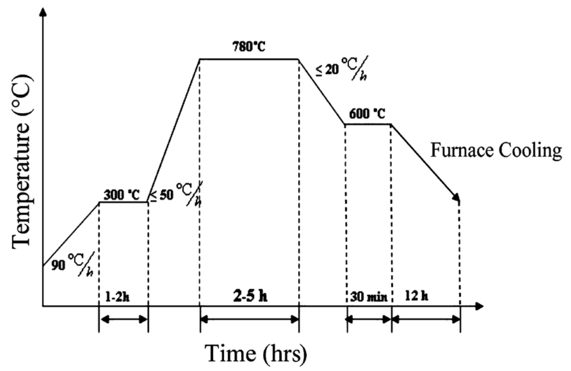


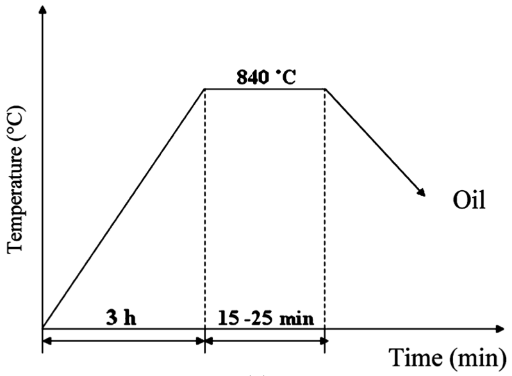
Fig. 3.The Heat treatment cycle of annealing after hot forging Stage.

Based on the chemical analysis given in Table 1 and the empirical following formula, the martansite start temperature (Ms) is about 233 °C. Therefore, a minimum temperature of 250 °C is necessary to decompose the retained austenite as shown in Fig. 4(b).

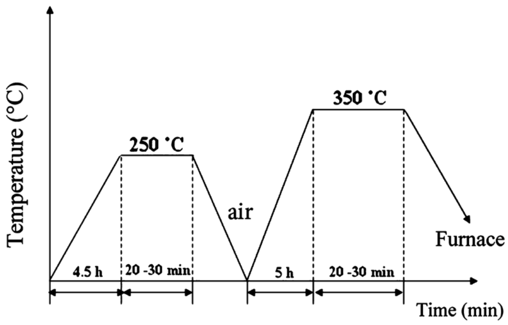
$$Ms\,(^{\circ}C) = 512 - 453\,C - 16/9\,Ni + 15\,Cr - 9.5\,Mo + 217\,(C)^2 - 71/5\,(C)(Mn) - 67/6\,(C)(Cr)$$

$$Ms\,(^{\circ}C) = 512 - 453\times(0.58) - 16/9 \times (1.48) + 15 \times (1.18) - 9.5 \times (0.47) + 217 \times (0.58)^2 - 71/5 \times (0.58) \times (0.74) - 67/6 \times (0.58) \times (1.18) = 233^{\circ}C$$

Hardening heat treatment was performed on the austenized sample at the temperature of 850 ºC by quenching in hot oil bath (Fig. 4a). In order to decompose the retained austenite completely and to improve the mechanical properties, a two–stage temper treatment was applied according to Fig. 4(b). The mechanical properties of samples A and B were determined after hot forging, annealing and heat treatments stages. Tension samples were prepared according to standard ASTMÁ 370 using a universal Zwick system. Impact samples were prepared according to standard ASTME23. The microstructure of samples studied by optical microscopy. The amount of inclusions and cavities were measured according to standard DIN 50602 and ASTME 45. All chemical compositions was analysed by a quantometry instrument. The amount of impurity elements of sulfur, carbon, hydrogen, oxygen and nitrogen was determined by the equipment of model Leco, Ghlaph Selax and Hydrix.



(a)



(b)

Fig. 4.Heat treatment cycles: (a) hardening; and (b) tempering treatments.

3. Results and Discussion

3.1. Secondary Steelmaking Process

In general, the desulfurization of molten steel can be facilitated by the presence of a basic slag including high content of CaO to minimize dissolved oxygen of the molten steel in making the ladle during secondary steel atmosphere at higher temperature. In order to decrease the solubility of oxygen, it is necessary to use strong oxide-forming elements such as Mn, Si, Al and Ca. In this investigation, with increasing of Al content from 12 Kg to 28Kg per 50 tons of molten tool steel, removal percentage of the sulfur has been changed from 36% to 88% after VDP[1] treatment (Fig. 5) by the following reaction:

$3(CaO)+ 2[Al]+ 3[S]= 3(CaS) + (Al_2O_3)$

The use of higher content of Ca-Si agent in the molten steel leads to lower content of the sulfur as a consequence of direct reaction between calcium and sulfur (CaS), and indirect reaction between silicon and oxygen (SiO_2) respectively. For example, increasing Si content from 0.2% to 0.33%, the amount of the sulfur content remains still approximately 45% of the initial sulfur content (Fig. 6). With increasing the temperature of the molten steel from 1635°C to 1685°C at the beginning of VD processing, the residual amount of the sulfur in the molten steel is about 50% of the original one (Fig. 7), depending on the progress of the endothermic desulfurization reaction.

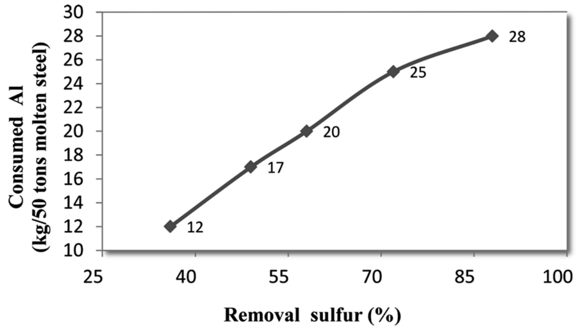


Fig. 5.The Effect of aluminum on the desulfurised treatment.

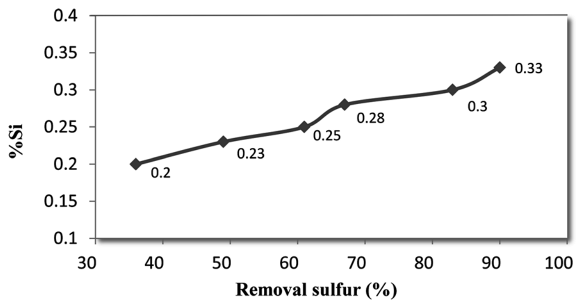


Fig. 6. The Effect of Si in the molten steel on the removal sulphur at 1635°C.

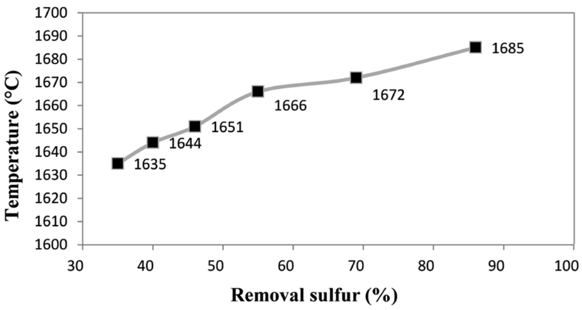


Fig. 7. The Effect of molten metal temperature on the removal sulfur.

[1] *Vacuum Degassing Process*

In the subsequent increase in holding time to more than 15 minutes at 1685 °C during VD processing, the amount of oxygen in the molten steel was increased along with the erosion and the solubility of refractory oxide lining, resulting a reduction in desulfurization rate (Fig. 8).

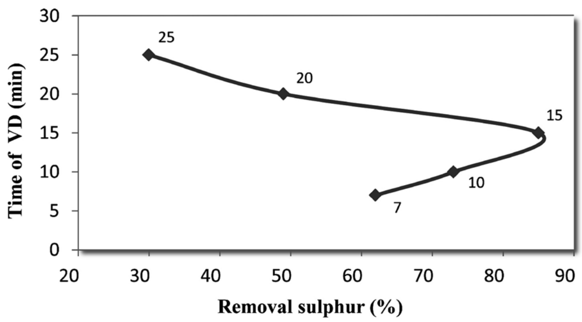


Fig. 8. The Effect of VD time on the desulfurisation at 1685 °C.

The amount of nitrogen in the sample B was determined about 71 ppm (Table 3) due to the formation of stable nitrides; whereas, this content for the degassed sample has been reached to 27.3 ppm. The solubility of oxygen in the molten steel in the electric arc furnace after the deoxidation stage is approximately 50 ppm which corresponding to oxygen content of the sample B (56 ppm in Table 3). For the degassed sample, the amount of soluble oxygen has been reached to 12.27 ppm. These observations are related to the mechanical properties developed in the samples A and B. As cleared in Table 4, there is a significant reduced value in tension strength and impact energy of the sample B in compared with the sample A. These results indicate that the mechanical properties of W500 tool steel is very sensitive to the solubility of hydrogen, oxygen and nitrogen. The presence of only 6.3 ppm soluble hydrogen that is almost five times higher than soluble hydrogen content in degassed sample A (Table 3) could be more effective in the brittle fracture.

Table 3. The Content of soluble gases (H, O and N) in samples A and B.

Sample	ppm(H)	ppm(O)	ppm(N)
A	1.53	12.27	27.3
B	6.3	56	71

3.2. Mechanical Properties and Microstructures

In Table 4, mechanical properties of the sample A has been compared with the sample B after hot working, annealing, quenching, and tempering heat treatments. Obviously, the difference in mechanical properties of samples A and B are very high, so these observations can be related to differences in secondary steel making operations. For the degassing sample (A), because of the purification and removal of impurity elements such as sulfur and phosphorous, the level of inclusions and cavities have been decreased (Fig 9d). Consequently, the hot working operation is more effective in which a higher mechanical property has been developed in comparison with the forged sample B (Table 4). For the sample B, the clusters of Al_2O_3 and silicate inclusions can be formed as the long filamentary inclusions after hot-forging operation (Figs 9a,b,c) due to the low plasticity of inclusions; As a result, the micro-cracks can be initiated around these particles. Therefore, the derived results of tension strengths, hardness and impact toughness for the sample B are considerably less than these results for sample A (Table 4) which can be result of distributing non-uniform inclusions and blow-holes interaction in the matrix of tool steel.

Table 4. The mechanical properties of samples A and B after hot working, annealing, quenching and tempering heat treatments.

Condition	Tension Test				Impact test	Hardness test	Sample
Mechanical Properties	Ultimate Strength (MPa)	Yield Strength (MPa)	ε %	(A%)	Absorbed energy, J (25 °C)	HRC	
Hot working	976	878	16.6	31.54	39	29	A
	870.2	475.4	14	26	35	21	B
Annealing	1198	1080	14	19	32	24	A
	995	756	10	17.79	23	26	B
Quenching and Tempering	2026	1800	6.6	6.5	11	55	A
	1845	1720	5.9	5.7	9	52	B

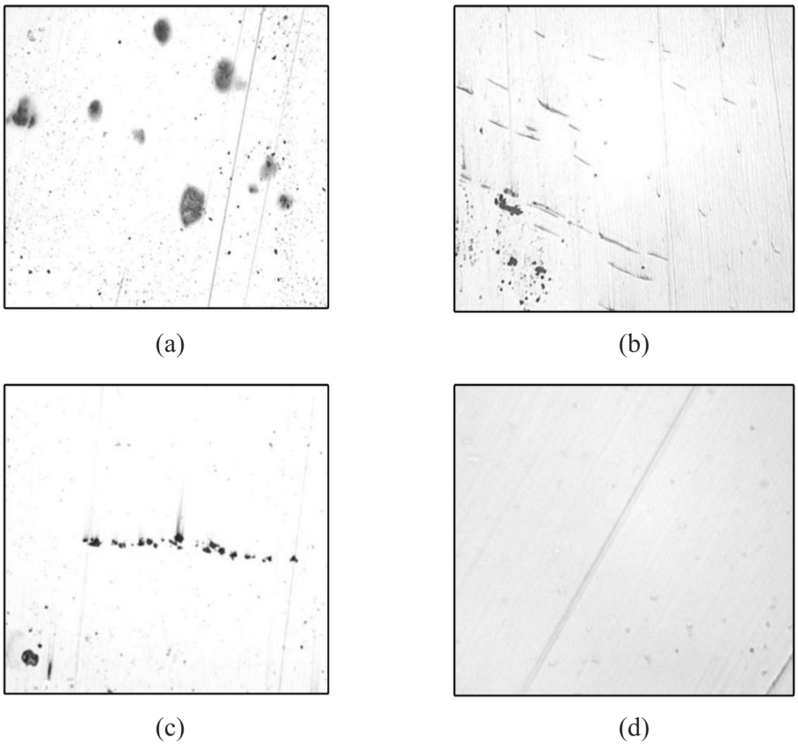


Fig. 9. The Optical microstructure of: (a, b, c) sample B; and (d) sample A (100X).

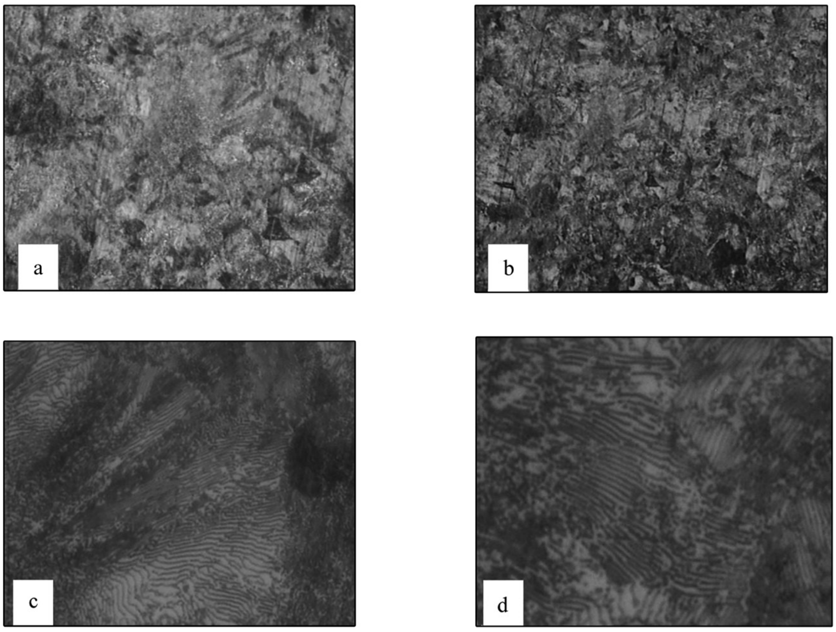


Fig. 10. The Optical microstructure after hot forging stage: (a) sample A (b) sample B (100X); (c) sample A; and (d) sample B (500 X).

The alumina inclusions can be converted to the calcium aluminates by adding Ca-Si agent to the molten steel, which reduce their negative effect on mechanical testing.

The microstructure of hot forged samples A and B are shown in Fig. 10. As is shown at higher magnifications in Figs. 10(c) and (d), there are fine Pearlite with the dispersed carbid particles in both microstructure of samples A and B. It is obvious that the morphology of the pearlite in the hot-forged sample A is more uniform and carbide particles are much finer than these particles in the normal forged sample B.

Both hot-forged samples A and B after annealing give the more uniform microstructure in compared to the only hot-forged samples (Fig. 11).

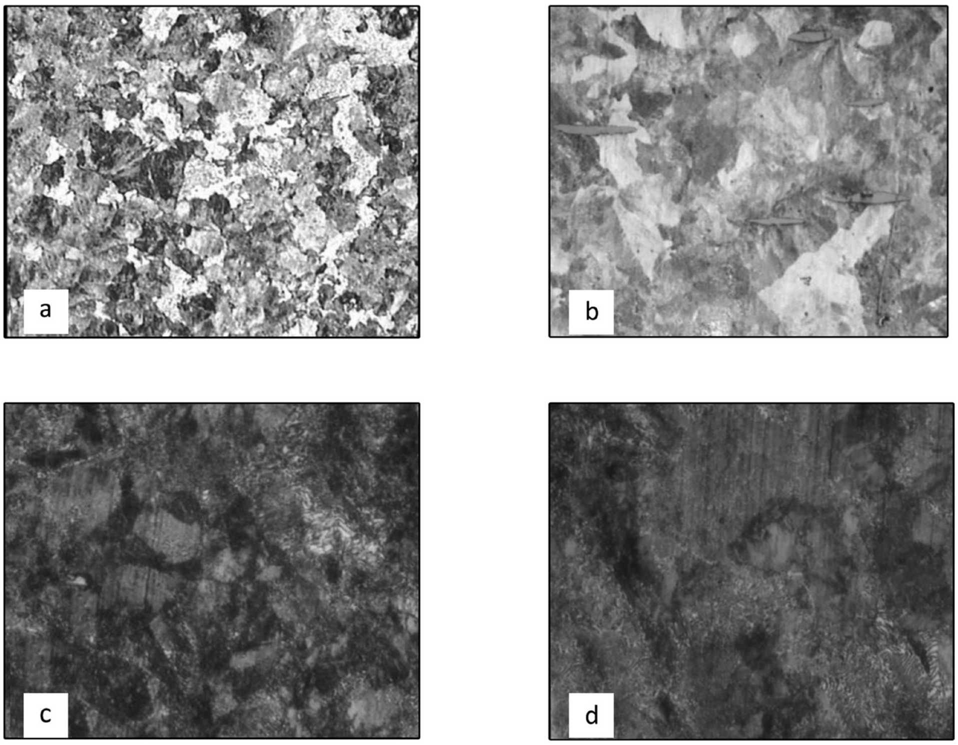


Fig. 11. The Optical microstructure after annealing treatment: (a) sample A; (b) sample B (100X); (c) sample A; and (d) sample B (500 X).

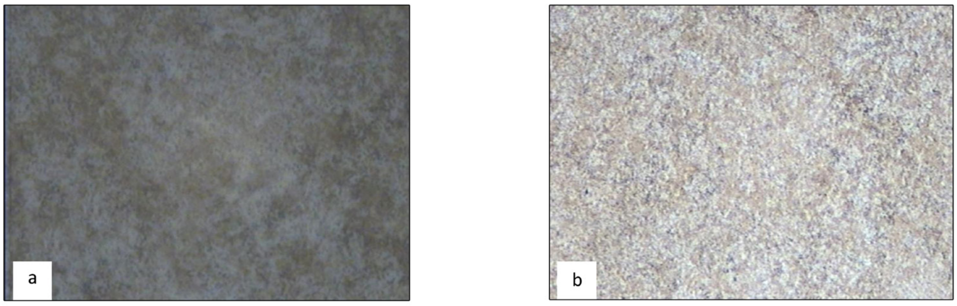


Fig. 12. The Optical microstructure after quenching and tempering Preocesses: (a) sample A; (b) sample B (500 X).

After quenching and tempering processes, it occurs the most difference between mechanical properties of samples A and B (Table 4). The formation of micro-crack around the inclusions in the sample B has been increased during the quenching stage as a consequence of interaction between hard martensitic structures and high solubility of hydrogen (6.3 ppm); so it reduces considerably mechanical properties in this sample. Also the high content of hydrogen (Table 3) is another reason which decreases mechanical properties of the sample B after hot forging, annealing, quenching and tempering treatments respectively. In the condition by higher hydrogen content than 6 ppm (the limited solubility), a hydride phase or a filamentary cavity can be formed in the microstructure which is more sensitive brittle behavior. It decreases impact toughness of steel (Table 4). The presence of cavities and gas blows within the sample B can be lead to the reduction of the effective section size against the applied force; thus, stress concentration has been increased around the inclusions and so the localized stress initiates the formation and growth of micro-cracks during mechanical testing. As a result, necking phenomena in the sample B can be occurred at much less applied stress than the same one for the sample A (Table 4).

It has been formed fine martensite with carbide particles in the microstructure of both samples A and B after the quenching and tempering treatments (Fig. 12). Therefore, the tensile and yield strengths, hardness and impact energy of both samples, particularly the sample A, were significantly improved after quenching and tempering treatments compared to hot forging and annealing processes (Table 4).

4. Conclusions

Various secondary steelmaking processes have been studied for the hot worked W500 tool steel. The main derived conclusions are as follows:

● 90% removal of sulfur in the molten W 500 tool steel can be achieved in the secondary steelmaking by conditions of 15 minutes holding time at 1685 °C in the vacuum degassing chamber, and using a high content of 0.056%Al and 0.33%Si.

● Vacuum degassing process reduces the amount of soluble H, O and N from 6.3 to 1.53 ppm, 56 to 12.27 ppm and 71 to 27.3 ppm, respectively. The degassing samples are more homogeneity in the microstructure and mechanical properties.

● The significant difference between mechanical properties of degassing samples and normal ones , especially, after quenching and tempering processes due to the presence of 6.3 ppm content dissolved hydrogen in the hard martensitic structure which is more sensitive to hydrogenous brittleness, the formation and growth of micro-cracks during loading test.

● After any case of hot working, annealing, quenching and tempering heat treatments, the tensile and yield strengths of both degassing sample and normal one were improved because of the fine structure associated with smaller carbide particles and non-metallic inclusions.

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