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THE INFLUENCE OF PRE – TEMPERING ON THE MECHANICAL PROPERTIES OF HS6-5-2 HIGH SPEED STEEL

WPŁYW WSTĘPNEGO ODPUSZCZANIA NA WŁASNOŚCI MECHANICZNE STALI SZYBKOTNĄCEJ HS6-5-2

Main objective of the research presented in this work was the evaluation of an influence of pre-tempering on the properties of HS6-5-2 steel after principal tempering. It was shown, that advance (by pre-tempering) of selected phase transformations during tempering may affect steel properties after principal tempering.

Samples of test steel, previously quenched in oil from the temperature of 1240°C have been pre-tempered at 20, 250, 450 and 550°C for 1.5 hour and subsequently cooled in air. Then principal tempering has been applied 3×580°C/1h. Due to high brittleness of test steel it was decided to test their strength with static bend test. The tests have been carried out on $\phi 5$ samples using an INSTRON testing machine. Evaluation of crack resistance of test steels has been made by means of linear elastic fracture mechanics method on the basis of the measurement of stress intensity factor K_{Ic}.

It has been found that, in test steel in which remains a lot of retained austenite after hardening, the pre-tempering within the range of cementite precipitation and transformation of retained austenite, adversely affects the properties after principal tempering. Pre-tempering of the investigated steel at the temperature insignificantly higher than precipitation of MC – type alloy carbides start temperature results in decrease in strength properties at insignificant increase of K_{Ic}.

Keywords: tool materials, high-speed steel, pre-tempering, mechanical properties, carbides, retained austenite.

W pracy przedstawiono wpływ wstępnego odpuszczania na własności stali HS6-5-2 po odpuszczaniu zasadniczym. Próbki badanej stali, uprzednio zahartowane od temperatury 1240°C (chłodzenie w oleju), odpuszczano wstępnie przy 20, 250, 450 i 550°C przez czas 1,5 godziny po czym chłodzono na powietrzu. Następnie zastosowano odpuszczanie zasadnicze 3×580°C/1h. Ze względu na to, iż badaniom podlegała stal szybkotnąca, zdecydowano się na badania wytrzymałości w statycznej próbie zginania. Badania wykonano przy użyciu maszyny wytrzymałościowej INSTRON na próbkach $\phi 5$. Ocenę odporności na pękanie badanych stali wykonano metodą liniowo-sprężystej mechaniki pękania w oparciu o pomiary współczynnika intensywności naprężeń K_{Ic}. Próbki zginano trójpunktowo na maszynie wytrzymałościowej INSTRON.

Stwierdzono, iż odpuszczanie wstępne w zakresie wydzielania cementytu i przemiany austenitu szczątkowego wpływa niekorzystnie na własności po odpuszczaniu zasadniczym. Wstępne odpuszczanie badanej stali przy temperaturze nieznacznie wyższej od temperatury początku wydzielania węglików stopowych typu MC powoduje spadek własności wytrzymałościowych przy nieznacznym wzroście K_{Ic}.

1. Introduction

During heating from the quenched state (tempering) of unalloyed, medium and high carbon steels, an occurrence of three principal transformations can be observed: precipitation of ε carbide, transformation of retained austenite into lower bainite and precipitation of cementite. In steels containing alloying elements inducing an effect of secondary hardening (V, Mo, W), a fourth transformation occurs: precipitation of MC and M_2C-type alloy carbides, that nucleate independently [1, 2].

During the first transformation in the temperature range of 100÷200°C, metastable ε carbide ($Fe_{2.4}C$) with hexagonal crystal structure precipitates from the supersaturated martensite [3]. Precipitation of highly dispersed ε carbide is believed to enhance strengthening in steel. However, the martensite with smaller carbon content is softer, what causes only a modest drop in strength and hardness of steel tempered in that temperature range [2].

Second transformation proceeding during tempering in the temperature range of 200÷320°C is a transformation of retained austenite. As a result of this transformation, a non-homogeneous mixture consisting of supersaturated ferrite and cementite, i.e. lower bainite forms. It should be noticed, that this transformation occurs only in

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steels containing more than 0.3%C because the amount of the austenite remaining in the steel after quenching strongly depends on carbon content. The austenite does not occur in steels containing less than 0.3%C [1, 2].

Cementite is formed during the third transformation occurring during tempering in the temperature range of 200÷420°C. This results in further decarbonization of the matrix and dissolution of metastable carbides allowing for recovery of the steel matrix. The mechanism of the nucleation of cementite is, however, not fully understood yet. According to Ref. [4] precipitating cementite nucleates independently or „in situ" on ε carbide particles. Whereas according to Ref. [5] cementite nucleates independently, mainly on grain boundaries of former austenite or on subgrain boundaries of newly formed cell structure.

Above 400°C, diffusion of alloying elements such as V, Mo and W takes place. Then, the cementite gradually dissolves to make the nucleation of MC and M_2C carbides coherent with the alloy matrix possible. This leads to an increase of hardness of tempered steel and is thus referred to as secondary hardening [2].

High speed steels owe its name to ability of fast machining and cutting of various materials including the alloys with iron matrix. They are characterized by high content of carbon and other alloying elements, mainly carbide forming, such as W, Mo, V and Cr. Some types of high speed steels contain also Co [1,2,6].

One of more important attributes of these steels are their cutting properties dependent on wear resistance, impact resistance and the resistance to heat tempering. Wear resistance depends on the type, content and form of primary carbides (MC, M_6C) and the matrix hardness. The impact resistance is determined by the state of tempered matrix, grain size of former austenite, spatial arrangement and the size distribution of primary carbides. The matrix of high speed steels consists of well tempered martensite and the carbides inducing secondary hardness. [1,6].

Microstructure of correctly heat treated high speed steel should consist of a hard and homogeneous matrix with a high volume fraction of fine and uniformly distributed carbides both the undissolved during austenitizing and being formed during tempering.

Among numerous research conducted in recent years on high speed steels one may distinguish the research on modification of chemical composition [7÷12], phase transformations during tempering [13÷16] and the surface properties of tools made of high speed steels [17,18].

Main objective of the research presented in this work was the evaluation of the influence of pre-tempering on the properties of HS6-5-2 steel after principal tempering.

2. Material

The research was conducted on HS6-5-2 high speed steel with chemical composition given in Table 1.

TABLE 1

Chemical composition for the investigated steel

Grade	mass %						
	C	Mn	Si	Cr	Mo	W	V
HS6-5-2	0.85	0.30	0.31	4.14	4.61	6.55	1.94

Prior to testing, the samples of the investigated steel were soft annealed at 840°C/4 hours and successively cooled to 600°C at the rate of 6°C/hour. After that the samples were cooled to room temperature in the furnace.

3. Experimental procedure

Samples of test steel, previously quenched from the temperature of 1240° (austenitizing times were 5 min for samples for flexural strength testing, 7 min for samples for crack resistance testing, quenching in oil in both cases), have been pre-tempered at 20, 250, 450 and 550°C for 1.5 hour and subsequently cooled in air. After that the principal tempering has been applied 3 × 580°C/1 h.

Due to high brittleness of the test steel it was decided to test their strength by a static bend test. The tests have been carried out on Ø5 samples using an INSTRON testing machine.

Evaluation of crack resistance of the test steel has been made by means of linear elastic fracture mechanics method on the basis of the measurement of stress intensity factor K_{Ic}, in a static bend test. The samples with dimensions of 9x18x90 mm have been used for the testing. The samples with a 2 mm deep fatigue cracks on the bottom of mechanically cut notch have been three-point bent on INSTRON testing machine.

Hardness measurements have been made with Vickers method using a HPO 250 apparatus.

The microstructure of investigated steels were examined by a light microscope Axiovert 200 MAT and a scanning electron microscope Hitachi 3500N

Fractographic study has been performed on fractures of samples used for K_{Ic} factor determination. The fracture surfaces have been examined using also a Hitachi 3500N type SEM analyzing microscope.

4. Results and discussion

In as-quenched state the structure of HS6-5-2 steel (**Fig. 1**) consists of partially twinned martensite, retained austenite in amount of 27,9% and carbides undissolved during austenitizing, mainly M_6C and occasionally MC [13].

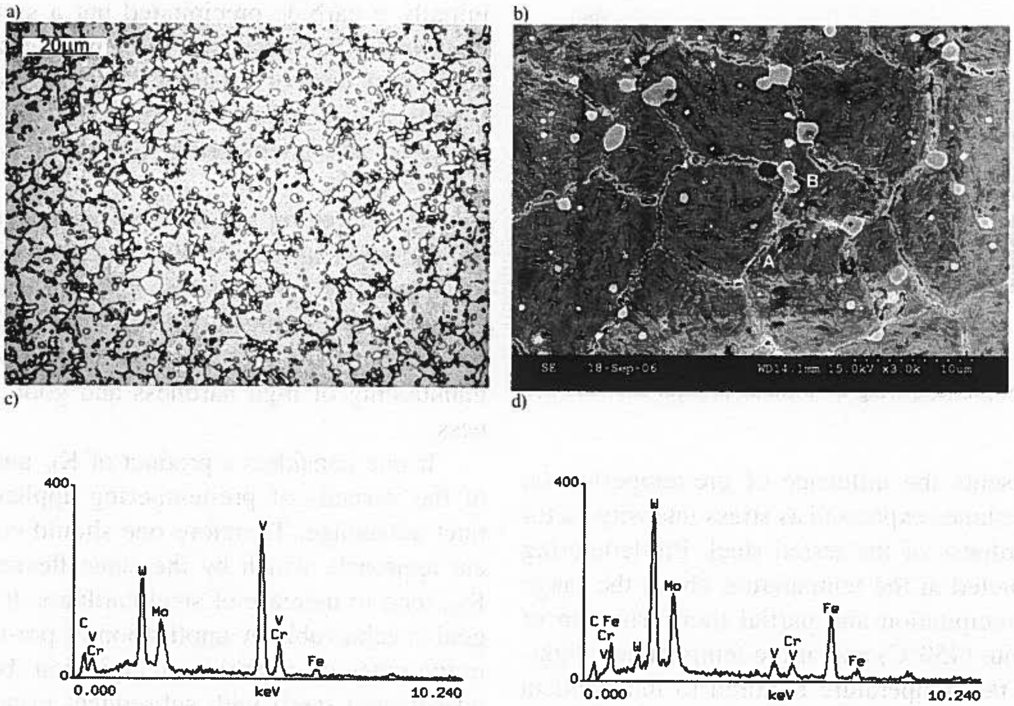


Fig. 1. Microstructure of tested steel samples after quenching from 1240°C: (a) light microscope, nital etched (b) scanning microscope, nital etched, (c) EDS spectrogram of MC carbides (place A), (d) EDS spectrogram of M_6C carbides (place B)

Fig. 2 presents the influence of pre-tempering on flexural strength and hardness of the steel after principal tempering. The temperatures and duration (1.5 h) of principal tempering have been selected on the basis of previous research [13]. For testing of the high speed steel a pre-tempering at the following temperatures has been selected: 250°C – i.e. slightly above the temperature of ε carbide precipitation end and start of cementite precipitation, 450°C – i.e. after the precipitation of cementite and the part of transformation of retained austenite are finised, 550°C – i.e. slightly above the temperature of precipitation start of independently nucleating carbides of the MC – type.

During pre-tempering at 250°C mainly the precipitation of ε carbide has been occurred. Negligible cementite precipitates on a boundary between martensite and austenite have not decreased of carbon content in austenite and, in result of it, during actual tempering the austenite behaved like at the absence of pre-tempering. That is why the flexural strength had not changed. However, the precipitation of ε carbides (mainly) has resulted in more uniform distribution of carbon after their solubilization inhibiting precipitation of cementite and, by it, creation of more favourable conditions for independent precipitation within whole volume of steel. Hence, the hardness of samples after the pre-tempering at 250°C is higher by about 25HV30. It confirms the above stated suggestion that the ε carbide precipitation (exclusively) may increase the hardness of steel after actual tempering.

Similarly, the values of flexural strength and hardness after pre-tempering at 450°C may be explained. They are almost identical as the ones after pre-tempering at 250°C. Most probably it is a result of higher stabilization of retained austenite and distinct decrease of advance of cementite precipitation in the HS6-5-2 steel if compared to the HS18-0-1 steel [13].

Pre-tempering at 550°C has started the transformation of retained austenite and has more advanced the precipitation of cementite, also initializing the precipitation of MC carbides. Most probably it has postponed the precipitation of MC carbides during actual tempering resulting in distinctly higher hardness of the steel if compared to standard tempering. Whereas a decrease of strength is most probably connected with transformation of part of retained austenite during pre-tempering.

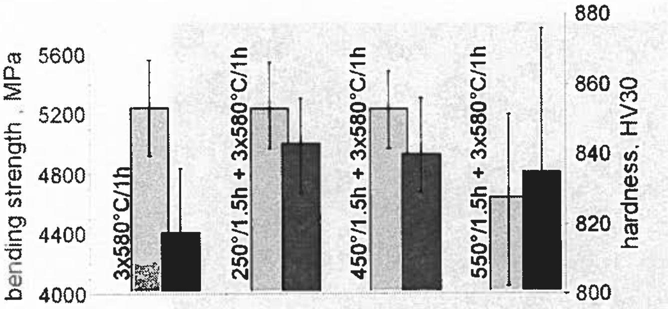


Fig. 2. Influence of pre-tempering on flexural strength and hardness of test steel

Fig. 3 presents the influence of pre-tempering on the fracture resistance expressed as stress intensity factor K_{Ic} and on hardness of the tested steel. Pre-tempering has been conducted at the temperature above the range of cementite precipitation and partial transformation of retained austenite (450°C) and at the temperature slightly higher than the temperature required to independent nucleation of MC – type carbides (550°C). Compared to the results of hardness measurements performed on samples after a bent test the results obtained are insignificantly higher what most probably is a result of a longer austenitizing time during hardening of the samples for the K_{Ic} test. Nevertheless the type of hardness changes is compatible for both tests.

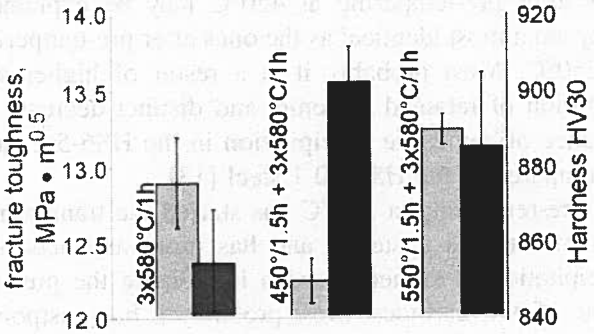


Fig. 3. Influence of pre-tempering on stress intensity factor K_{Ic} and on hardness of test steel

During pre-tempering of the HS6-5-2 steel at 450°C initially ε carbide precipitated but a significant part of them had solubilized due to cementite precipitation. In addition a part of retained austenite had transformed. It caused a significant increase (by 47HV30) of hardness after actual tempering at a little decrease of K_{Ic} (by 0.7MPa $m^{0.5}$).

Pre-tempering at 550°C has started the transformation of larger amount of retained austenite and increase of advance of cementite precipitation and thus the process of alloy carbides precipitation of MC – type during actual tempering had been postponed what resulted in maintaining of high hardness and good fracture toughness.

If one considers a product of K_{Ic} and hardness, any of the variants of pre-tempering applied brings a distinct advantage. Therefore one should consider a different approach, which by the same flexural strength and K_{Ic}, tend to increase of steel hardness. It seems that this goal is achievable by application of pre-tempering within the range of ε carbide precipitation (below 200°C for investigated steel) with subsequent principal tempering 3 × 580°C/1 h applying fast heating (eg. in salts) to this temperature.

Fig. 4×6 presents the fractures of the steel after crack resistance test. One may notice that the appearance of the fractures is similar. In all variants the significant influence on flexural strength and K_{Ic} has had a content and distribution of undissolved carbides during hardening process.

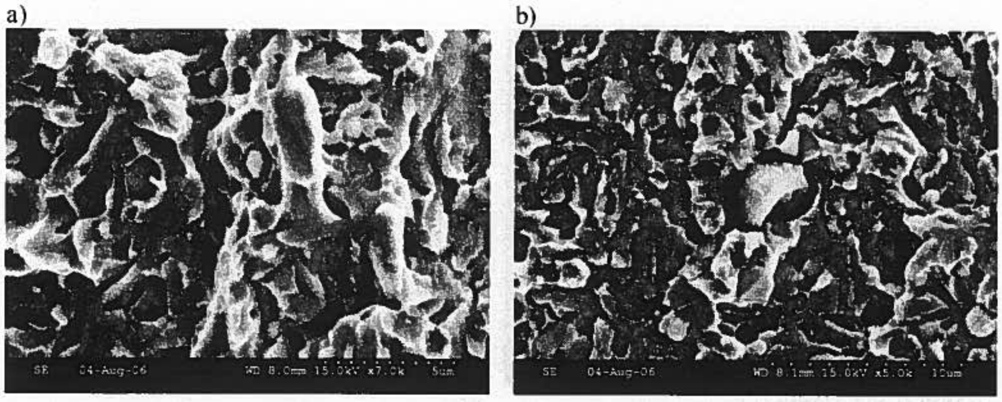


Fig. 4. Fractures of HS6-5-2 steel samples after crack resistance test, tempered 3×580°C/1 h. SEM

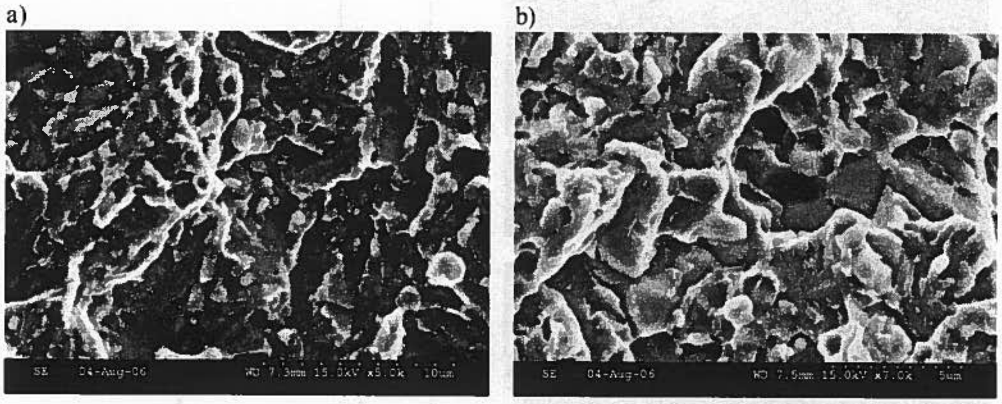


Fig. 5. Fractures of HS6-5-2 steel samples after crack resistance test, tempered at 450°C/1.5 + 3×580°C/1 h. SEM

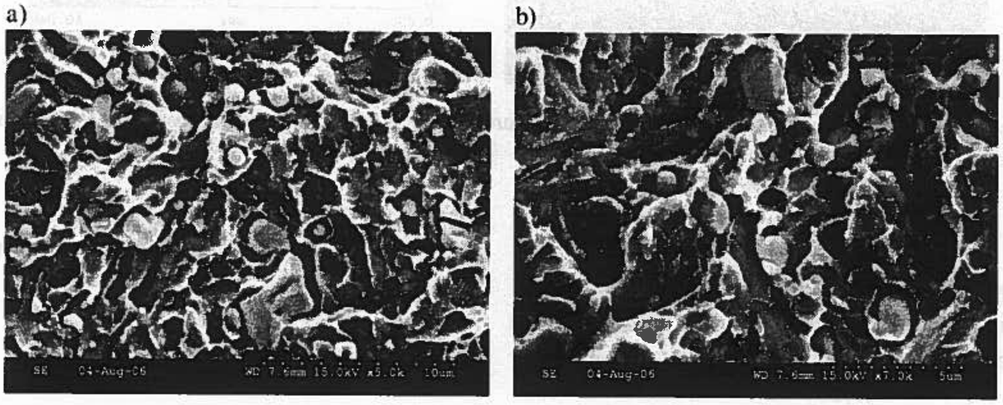
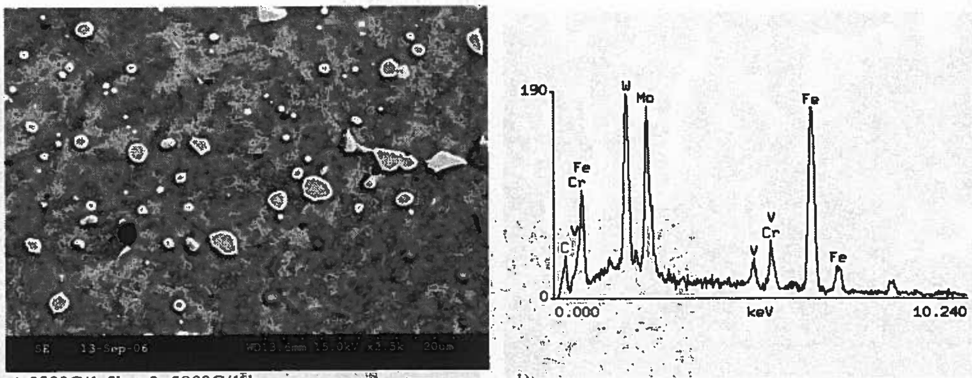


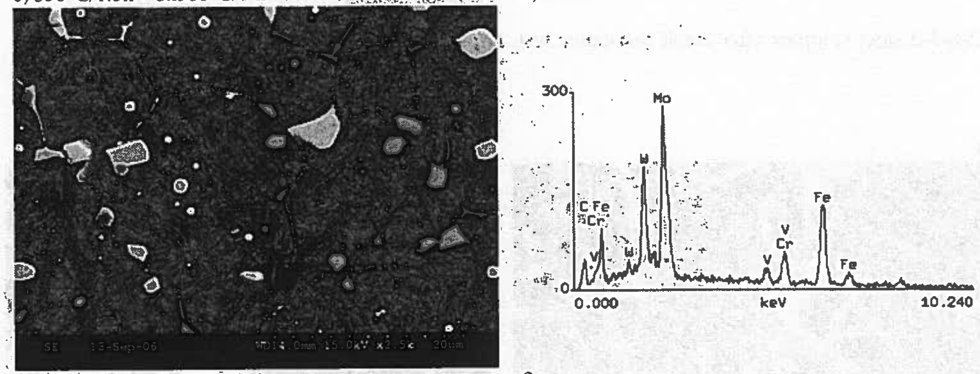
Fig. 6. Fractures of HS6-5-2 steel samples after crack resistance test, tempered at 550°C/1.5 + 3×580°C/1 h. SEM

Microstructures of samples after different variants of tempering are presented in Fig. 7. There appeared a few precipitates of $M_{23}C_6$ carbide in the structure of the tested steel after tempering at 3×580°C. Pre-tempering at 450 and 550°C, due to previously discussed phenomena, has intensify this process. After pre-tempering at 550°C a network of precipitates of these carbides has been already created along the boundaries of former austenite grains. It confirms a fact that Mo is susceptible to segregation towards grain boundaries.



c) 550°C/1.5h + 3x580°C/1h

d)



e) 550°C/1.5h + 3x580°C/1 h

f)

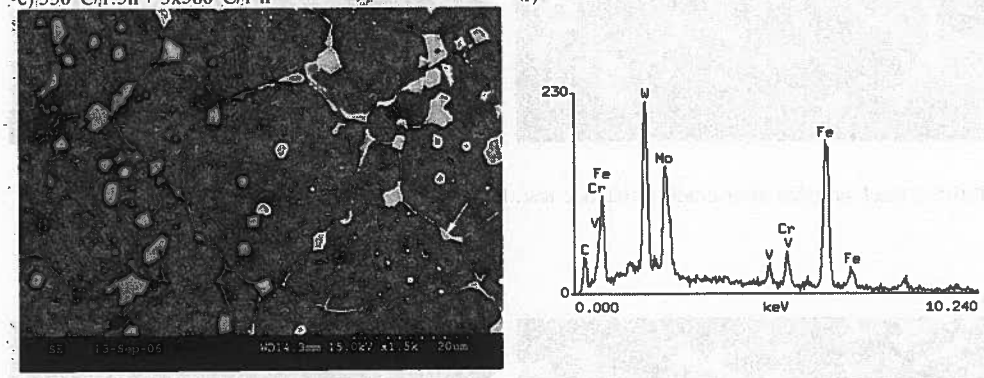


Fig. 7. Morphology and identification of carbides: a,c,e) microstructures of tested steel after applied heat treatments, scanning microscope, 2% nital etched; b,d,f) suitable EDS spectrograms of $M_{23}C_6$ carbides (marked places)

5. Summary

Advance (by pre-tempering) of selected phase transformations during tempering affects steel properties after principal tempering. It has been found that, in the test steel, in which a lot of retained austenite after remains hardening (~27%), the pre-tempering within the range of cementite precipitation and transformation of retained austenite, adversely affects the properties after principal tempering. Pre-tempering of the investigated steel at the temperature insignificantly higher than the start temperature of precipitation of MC – type alloy carbides results in decrease of strength properties at insignificant increase of K_{Ic}.

After pre-tempering at 550°C a network of precipitates of $M_{23}C_6$ carbides has been created along the boundaries of former austenite grains. It confirms a fact that Mo is susceptible to segregation towards grain boundaries.

REFERENCES

[1] R. W. K. Honeycombe, H. K. D. H. Bhadeshia, Steels. Microstructure and properties, 2nd ed. London: Edward Arnold (1995).

[2] A. K. Sinha, Physical metallurgy handbook, The McGraw-Hill Companies, Inc. (2003).

[3] R. Padmanabhan, W. E. Wood: Precipitation of ε carbide in martensite. Materials Science and Engineering, **65**, 289-297 (1984).

[4] R. C. Thomson, M. K. M. Miller, Acta Matall, **46**, 2203-2213 (1998).

[5] J. Pacyna, B. Pawłowski, Effect of tempering temperature on 30HGSNA steel toughness. Metallurgy and Foundry Engineering, **10**, **4**, 409-421 (1984).

[6] G. Krauss, Steels: Processing, Structure and Performance, ASM Int. (2005).

[7] L. A. Dobrzański, W. Kasprzak, J. Mazurkiewicz, The structure and properties W-Mo-V-Co high-speed steel of the type 11-2-2-5 after heat treatment, Proceedings of the 4th International Scientific Conference „Achievements in Mechanical and Materials Engineering" AMME'95, Gliwice-Wisła, 83-86 (1995).

[8] L. A. Dobrzański, A. Zarychta, Journal of Materials Processing Technology, **77**, 180-193 (1998).

[9] F. Pan, Ma Hirohashi, Y. Lu, P. Ding, A. Tang, D. V. Edmonds, Metallurgical and Materials Transactions, **35A**, 2757-2766 (2004).

[10] L. A. Dobrzański, M. Ligraski, Proceedings of the 4th International Scientific Conference „Achievements in Mechanical and Materials Engineering" AMME'95, Gliwice-Wisła, 87-90 (1995).

[11] M. Wang, Y. Wang, F. Sun, Materials Science and Engineering A, **438**, 1139-1142 (2006).

[12] L. Xu, J. Xing, S. Wei, Y. Zhang, R. Long, Wear **262**, 253-261(2007).

[13] P. Bała, The kinetics of phase transformations during tempering and its influence on the mechanical properties, PhD thesis, AGH University of Science and Technology, Cracow 2007. Promotor J. Pacyna (in Polish).

[14] P. Bała, J. Pacyna, J. Krawczyk, Journal of Achievements in Materials and Manufacturing Engineering **19**, 1, 19-25 (2006).

[15] P. Bała, J. Pacyna, J. Krawczyk, Journal of Achievements in Materials and Manufacturing Engineering **18**, 47-50 (2006).

[16] T. H. Yu, C. Y. Chen, J. R. Yang, Journal of Materials Engineering and Performance **16**, 102-108 (2007).

[17] L. A. Dobrzański, M. Adamiak, W. Kasprzak, Proceedings of the 7th International Scientific Conference „Achievements in Mechanical and Materials Engineering" AMME'98, Gliwice-Zakopane, 107-110 (1998).

[18] T. Babul, N. Kucharieva, A. Nakonieczny, J. Senatorski, Journal of Materials Engineering and Performance **12**, 696-700 (2003).

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