MICROSTRUCTURAL DEVELOPMENT OF LEDEBURITIC TOOL STEELS DURING HEAT TREATMENT PROCEDURE

Peter JURČI

Faculty of Materials Sciences and Technology in Trnava, Bottova 25, Trnava, Slovakia, EU
p.jurci@seznam.cz

Abstract

Ledeburitic steels based on Cr- or Cr-V alloying belong to the most important cold-work tool materials for large series manufacturing. To enable high production stability, the tools must withstand various types of degradation processes. On the first side, it concerns the plastic deformation and wear. Therefore, the materials should have high hardness, tensile and/or compressive strength and wear resistance. Alternatively, the materials have to resist the brittle fracture, i.e. they must manifest sufficiently high toughness and fracture toughness. The review paper deals with the effect of heat treatment procedures on the microstructures of Cr- and Cr-V ledeburitic steels. Particular attention is paid to the effect of the austenitizing and quenching, tempering and sub-zero treatment on the main microstructural features of the steels, and their relation to the main mechanical properties.

Keywords: Ledeburitic steels, heat-treatment, martensite, retained austenite, carbides

1. INTRODUCTION

High carbon high chromium (and vanadium) ledeburitic steels have gained a wide popularity in tooling, due to their high wear resistance and compressive strength. These steels are used in manufacturing of automotive parts, powder compaction, stamping, paper cutting etc. In these applications, the tools made of Cr-V ledeburitic steels have to be heat treated. Proper heat treatment results in high hardness and strength, excellent wear resistance, but the materials frequently suffer from insufficient toughness and fracture toughness. The heat treatment is then always a compromise between these properties, and should be tailored to particular applications. The current review conference paper deals with the procedure which is commonly applied for the heat treatment of Cr-V ledeburitic steels.

2. AS - DELIVERED STATE

The bars, sheet metals and blocks made of Cr-V ledeburitic steels are distributed to the end-users in soft-annealed state. This is because these materials generally manifest poor machinability, and it is necessary to improve this “general” property to at least an acceptable level, thorough lowering their hardness and modification of carbide shape. Typical microstructure of ledeburitic steel Vanadis 6 after soft annealing is depicted in **Figure 1**. The material consists of alloyed pearlite, secondary (SCs) and eutectic carbides (ECs). It is worth noticing that the carbides are very fine and uniformly distributed throughout the matrix in this case as the steel is manufactured by powder metallurgy of rapidly solidified particles.

3. AUSTENITIZING AND QUENCHING

It is strongly required that the tools made of Cr-V ledeburitic steels must be heat treated before use. Proper heat treatment brings: high hardness of tools strength and wear resistance. Nevertheless, this procedure should be carried out carefully. Otherwise, undesirable changes in the steel microstructures (like grain coarsening and/or too high retained austenite amount) can occur.

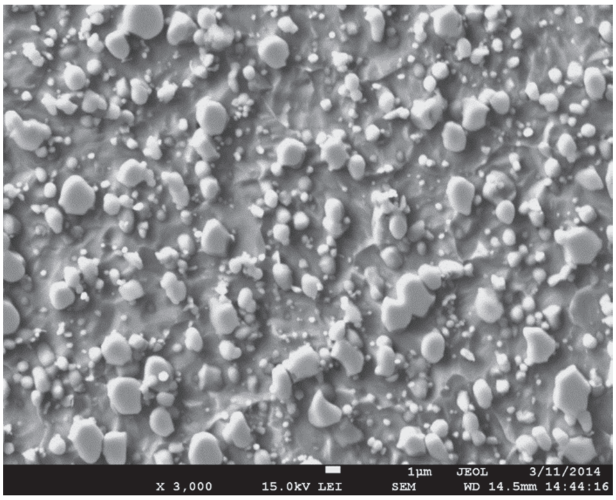

Figure 1 SEM micrograph showing as-annealed microstructure of Cr-V ledeburitic steel Vanadis 6

The first step in heat treatment is austenitizing. Because of poor thermal conductivity of the materials, the heating up to final temperature should be carried out gradually, in order to minimize thermal gradients between the surface and core and to prevent too large distortion of tools or their failure.

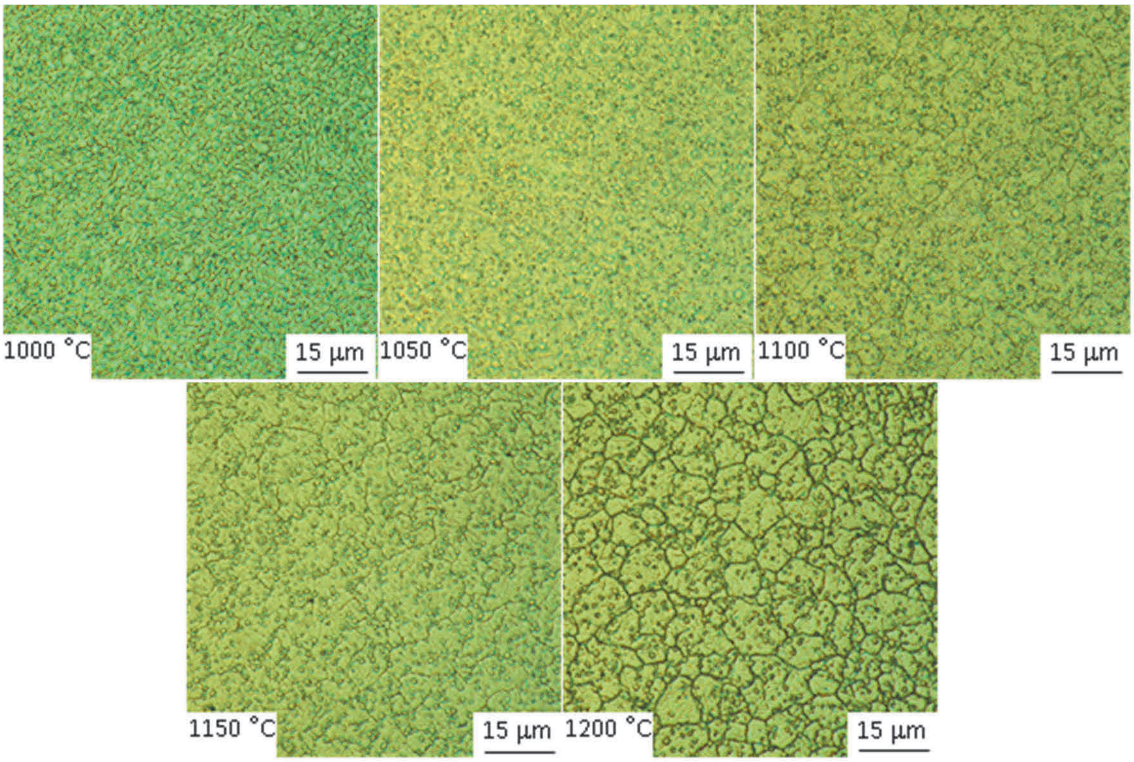

Figure 2 Light micrographs showing the microstructure of the Vanadis 6 ledeburitic steel after quenching from different austenitizing temperatures [1]

The transformation of pearlite into the austenite does not lead to sufficient saturation of the austenite since the alloying elements amount in pearlite is very low. This is why the Cr-V ledeburitic steels must be heated up to much higher temperature. During the heating from the A_1 temperature up to the final austenitizing, part of secondary carbides undergoes the dissolution. In recent study it has been reported on complete dissolution of secondary M_7C_3-carbides up to an austenitizing temperature of 1100 °C [1].

The upper limit of recommended austenitizing temperature is determined mainly by two phenomena: beginning of the austenitic grains coarsening and rapid increase of retained austenite amount in as-quenched steels. Also, an overall economy of the process should be taken into the account - the higher austenitizing temperature the increased costs are for realizing of the heat treatment. **Figure 2** shows light micrographs of as-quenched microstructures of Vanadis 6 steels. Measurement of austenitic grain size according to the ASTM-method revealed that after the quenching from lower temperature, it was 11.5 and it increased to 10 after quenching from a temperature of 1200 °C. Correspondingly, the retained austenite (γ_R) amounts were 10.1 ± 3, 12.1 ± 1, 20.2 ± 1.6, 22.7 ± 4.7 and 28.2 ± 3 vol. % after quenching from the temperatures 1000, 1025, 1050, 1075 and 1100 °C, respectively. Retained austenite is undesirable microstructural constituent in high strength steels. It is metastable at the room temperature, and can transform into brittle martensite when heavily loaded. Therefore, it is highly recommended to eliminate the γ_R before use of tools or components.

4. SUB-ZERO TREATMENT

Sub-zero treatment (SZT) is a supplementary process, which is inserted into the heat treatment schedule of steels in-between the quenching and tempering. In this process, the steels are cooled down slowly to the lowest temperature of the heat treatment cycle, held there for predetermined time, and re-heated slowly to the room temperature.

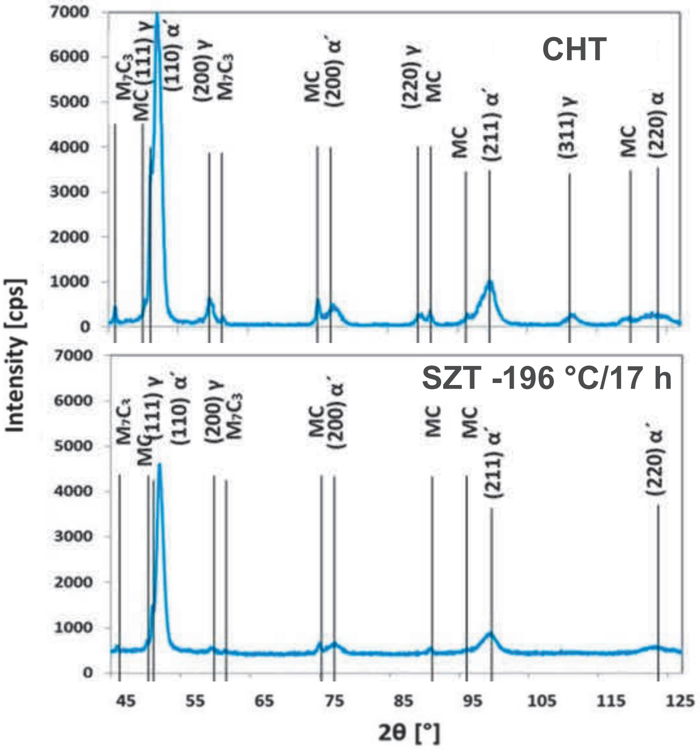

Figure 3 Typical X-ray diffraction profiles of conventionally quenched (CHT) and sub-zero treated Vanadis 6 steel

One of the benefits of the SZT is substantial reduction of γ_R amount. Many researchers really evidenced this fact, and their findings differ one from each others only in the extent of the γ_R reduction, from either “almost complete removal” [2-7] or “elimination” [8-12] to „considerable reduction“ [13-15]. An example of X-ray diffraction profiles of conventionally quenched and SZT (-196 °C/17 h) Vanadis 6 steel is in **Figure 3.** Note that the retained austenite amount was 20 vol. % after conventional quenching but it was reduced to 2.1 vol. % after SZT.

The second advantage of the SZT is that the martensite manifests clearly evident refinement compared to that developed by conventional quenching [13,15]. An example is depicted in **Figure 4.** Refinement of the martensite may have a beneficial effect on both the strength and toughness of the materials as suggested recently [16,17]. An explanation of the martensite refinement due to the SZT was first suggested in the paper [15] where the aggregate γ_R to α´ transformation was divided into two components: i) diffusion-less (athermal) component, which takes place during conventional room temperature quenching, and ii) time-dependent isothermal component, which is active during hold of the materials at a temperature of SZT. The second component is accompanied with extensive plastic deformation of newly formed "virgin" martensite, which results in capture of carbon atoms by moving dislocations, and their clustering [13,15,18,19]. These phenomena are associated with certain mass transfer, and might be responsible for controlling of the growth of martensitic domains, which would make them finer compared to those formed in athermal transformation. Also, it is logical that martensitic domains grow relatively freely during room temperature quenching as there is much space for their growth available within original austenitic grains but the growth of isothermally formed martensite is limited because of the retained austenite (after room temperature quenching) is encapsulated between athermally grown domains of martensite.

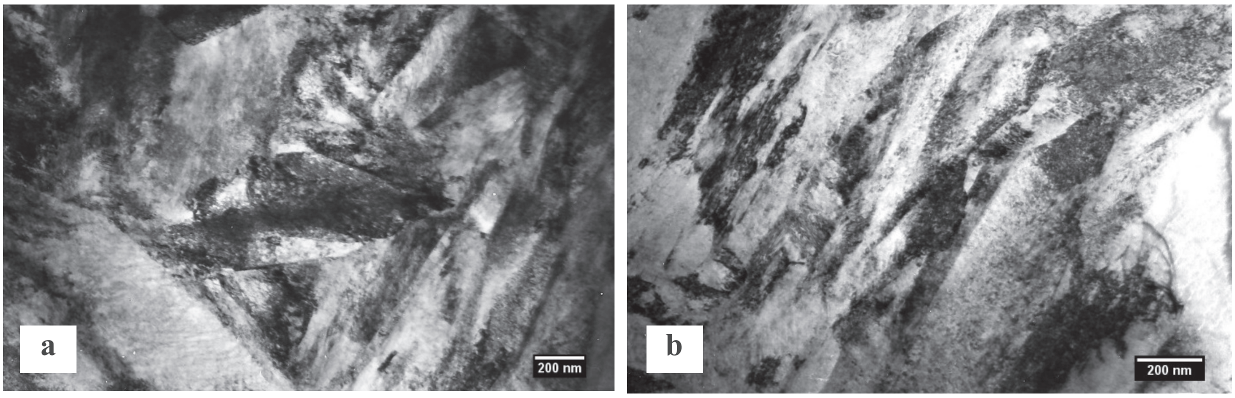

Figure 4 TEM micrographs showing the matrix microstructure of a) conventionally quenched (CHT), b) sub-zero treated Vanadis 6 steel

Despite the fact that the opinions on the changes in precipitation kinetics of carbides manifest clear contradictions the latest experimental results indicate that the precipitation rate of transient carbides on Cr- and Cr-V ledeburitic steels is rather accelerated due to the application of SZT. For instance, transient nano-sized particles have been found in SZT Vanadis 6 steel already prior tempering while no precipitates occurred in conventionally quenched steel [20]. Theoretically, the explanation is based on the fact that the strain of the martensitic transformation is accommodated in the martensite and not in the retained austenite. This accommodation is realized by extensive plastic deformation of relatively ductile "virgin" martensite [21,22], which generates an increased number of carbon clusters at crystal defects. These clusters can act as nuclei for further precipitation of carbides.

While the precipitation rate of transient carbides can be considered as accelerated in low temperature tempering range, the precipitation of alloy carbides at temperatures commonly used for secondary hardening is suppressed [18,20,23]. This may be due to the fact that the formation of these carbides requires extra carbon atoms, but the martensite is depleted by carbon as a result of the formation of small globular carbides (SGCs).

The presence of enhanced number and population density of SGCs (size usually 100 - 500 nm) in ledeburitic tool steels is the last microstructural change induced by the SZT, **Figure 5**. The explanation of the formation of SGCs is based on generally accepted fact that martensitic transformation is associated with 3 - 4% volume expansion. The volume change is associated with high compressive stresses in the retained austenite [24,25]. This is why the martensitic transformation is never completed despite the temperature of SZT lies well below the characteristic M_f temperature of alloys. On the other hand, low processing temperature is a strong driving force for further progress of the transformation. This progress is, however, only possible when the compressive stresses in the γ_R would be relieved. At very low temperatures the only possible way how to relief the stresses is a deformation - induced formation of phase with lower specific volume. The obtained experimental data in the most recent investigations infer that [15,25]:

- The percentage reduction of γ_R corresponds well to the estimated area fraction of sites with presence of SGCs
- The increase of amount and population density of these carbides is time - dependent, and follows the decrease in the γ_R amount
- The nature of the SGCs was determined as cementite, i.e. as phase with lower specific volume than that of both the martensite and the austenite
- The indications on plastic deformation of cementite particles were evidenced. It is expected that newly formed SGCs co-deform with the matrix during the isothermal part of the martensitic transformation.

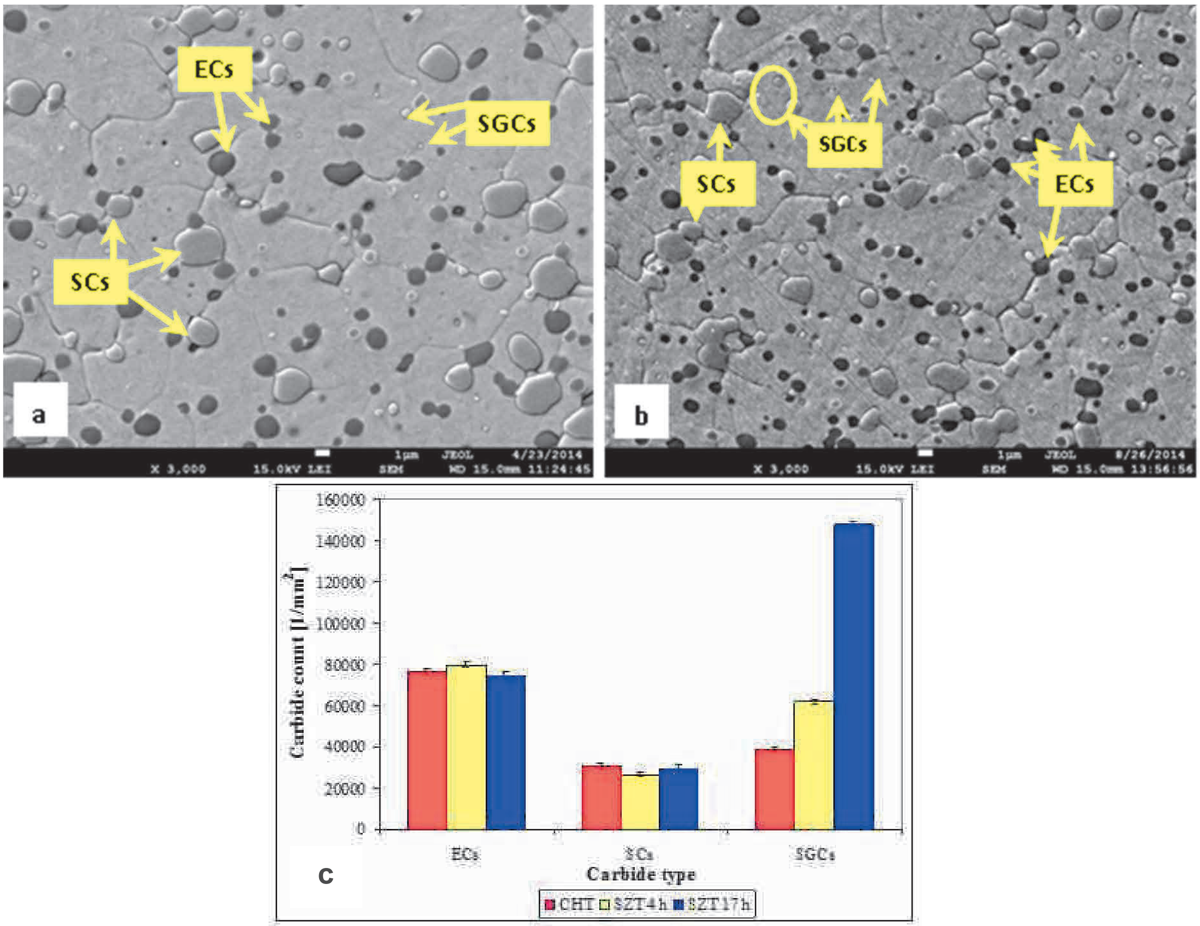

Figure 5 SEM micrographs showing the microstructure of a) conventionally quenched (CHT), b) SZT Vanadis 6 steel, carbide count per mm^2 of ECs, SCs and SGCs for differently heat treated specimens

Based on the summary above the following concept describing the formation of SGCs can be adopted: the SGCs could be considered as a “by-product” of the isothermal component of martensitic transformation, which takes place at the temperature of SZT. The reaction can generally be described using the formula:

$$\gamma_R \rightarrow \alpha' + SGCs(carbide) \tag{1}$$

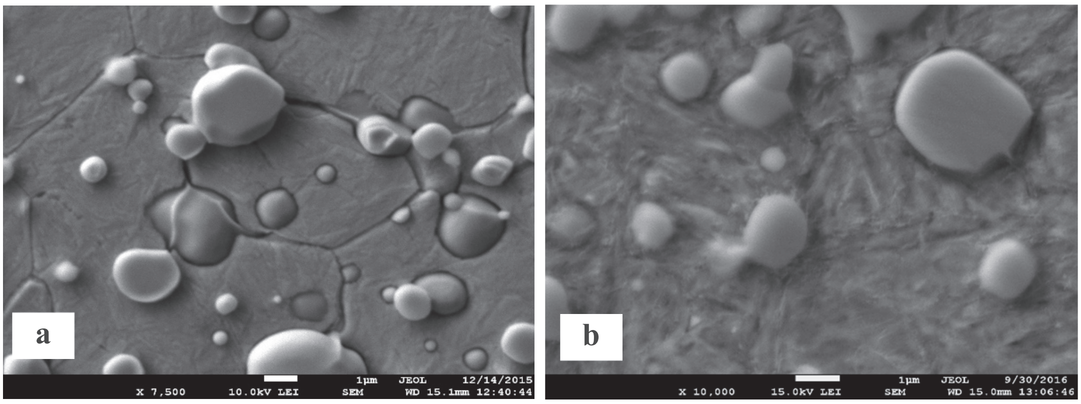

Figure 6 ¨0 6 steel

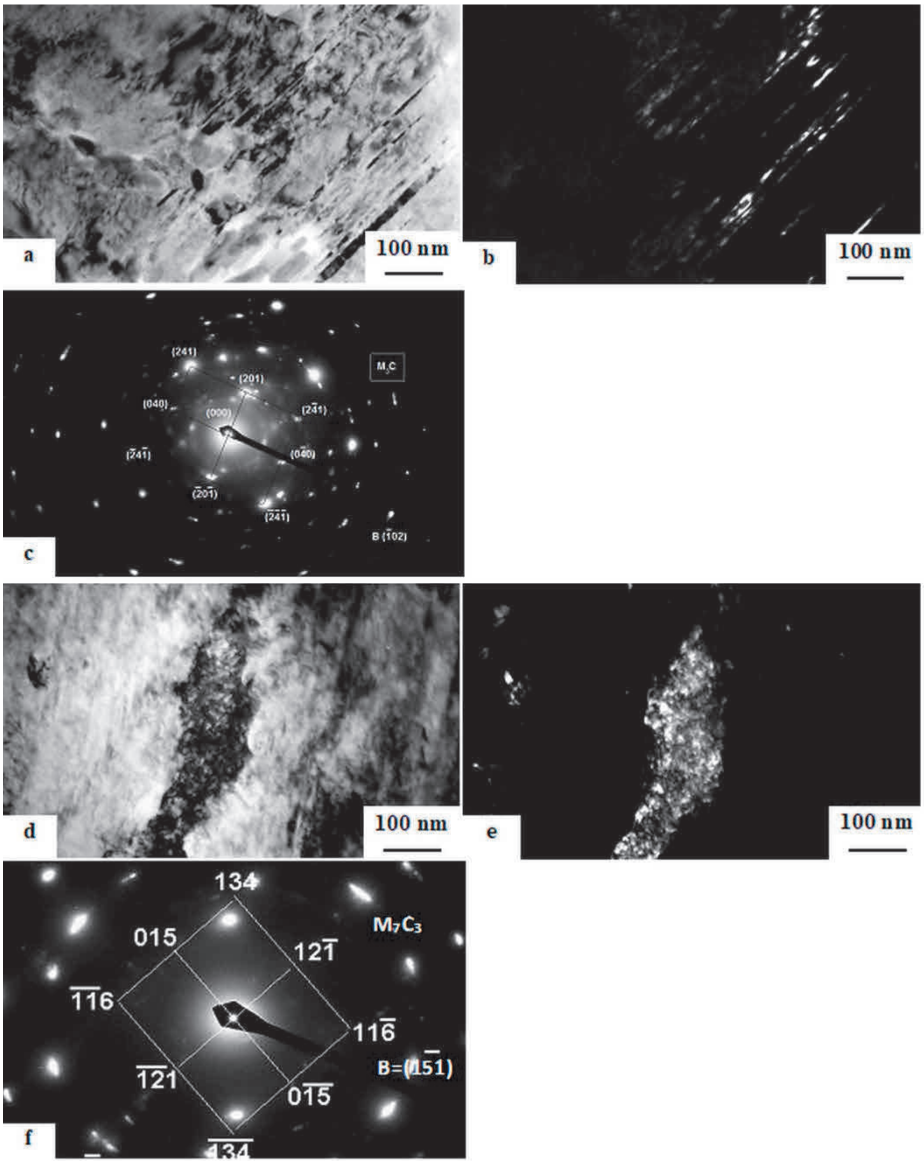

Figure 7 TEM micrographs showing the sub-structure of conventionally quenched Vanadis 6 steel after tempering at 530 °C. a, d - bright-field images, b, e - corresponding dark-field images, c, f - diffraction patterns of precipitates [20].

5. TEMPERING

Tempering should follow the quenching and/or sub-zero treatment as soon as possible. Otherwise, the retained austenite could have been stabilized. For a more complete transformation of γ_R to martensite, as well as for tempering of the alloying elements and carbon newly formed martensite, it is necessary to temper at least twice. During the tempering, diffuse out as from solid solutions forming precipitates responsible for secondary hardening effect, **Figures 6, 7.** Cooling down from the tempering temperature induces decomposition of γ_R, **Figure 8** [20]. .

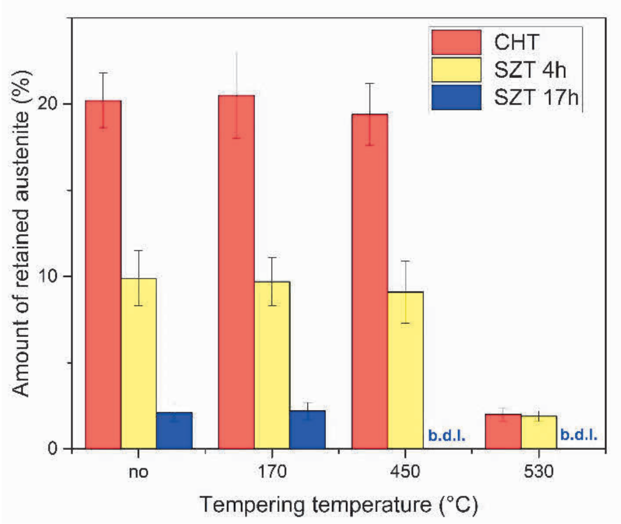

Figure 8 Retained austenite amount as a function of tempering temperature for differently heat treated Vanadis 6 steel. Note that "b.d.l." means that the amount is below the detection limit of X-ray diffraction [20].

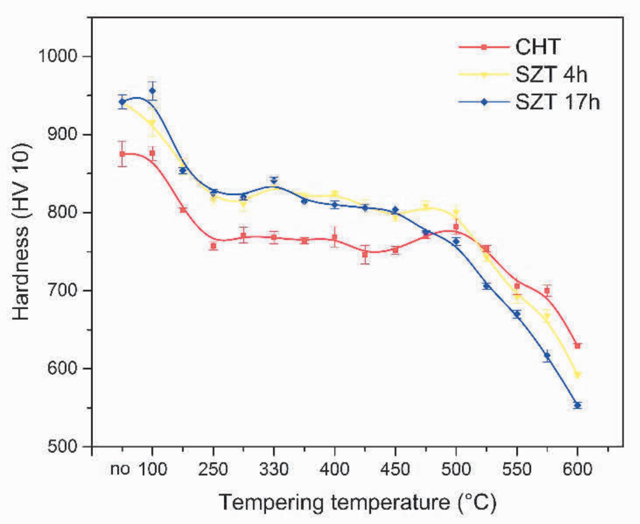

Figure 9 Tempering charts of differently heat treated SZT Vanadis 6 steel

Cr- and Cr-V ledeburitic steels manifest so-called "secondary hardening", i.e. the hardness increases with increasing the tempering temperature within a certain temperature range. This is due to complementary effect of transformation of γ_R, precipitation of carbides and tempering of the martensite. **Figure 9** shows tempering diagrams of the Vanadis 6 steel after conventional quenching, and after SZT. It is shown that there is secondary hardening effect in the case of conventionally quenched steel while this effect disappears completely for SZT material. Detailed description of this fact has been published recently [20], but it can be summarized briefly a follows: The contribution of retained austenite transformation is reduced because SZT steel contain considerably lowered γ_R amount. Also, the precipitation of special carbides is suppressed since the matrix is depleted by carbon as a result of the formation of SGCs during the sub-zero treatment

6. CONCLUSION

The conference paper describes the heat treatment schedule, which is applied in manufacturing route of cold work tool steels made of Cr- and Cr-V ledeburitic steels. It is reported that the microstructure changes from the carbide/pearlite, which is typical for as-annealed materials, to that consisting of the martensite, retained austenite and undissolved carbides after austenitizing and quenching. An attention is paid to the changes due to the sub-zero treatment, which comprise reduction of the retained austenite amount, formation of extra carbides, overall refinement of the microstructure, and alterations in precipitation behaviour. Finally, tempering of the materials is described, with particular attention to transformation of retained austenite and precipitation of carbides.

ACKNOWLEDGEMENTS

The authors acknowledge that the paper is a result of experiments realized within the project VEGA 1/0264/17. In addition, this publication is the result of the project implementation "Centre for Development and Application of Advanced Diagnostic Methods in Processing of Metallic and Non-Metallic Materials - APRODIMET", ITMS: 26220120014, supported by the Research & Development Operational Programme funded by the ERDF.

REFERENCES

[1] JURČI, P. Cr-V Ledeburitic Cold-Work Tool Steels. *Materials and Technology,* 2011, vol. 45, pp. 383 - 394.

[2] DAS, D. DUTTA, A.K., RAY, K.K. Sub-zero treatments of AISI D2 steel: Part II. Wear behaviour, *Materials Science and Engineering,* 2010, vol. A527, pp. 2194 - 2206.

[3] DAS, D. DUTTA, A.K., RAY, K.K. Influence of varied cryotreatment on the wear behaviour of AISI D2 steel. *Wear,* 2009, vol. 266, pp. 297 - 309.

[4] DAS, D., SARKAR, R., DUTTA, A.K., RAY, K.K. Influence of sub-zero treatments on fracture toughness of AISI D2 steel. *Materials Science and Engineering,* 2010, vol. A528, pp. 589 - 603.

[5] DAS, D. DUTTA, A.K., RAY, K.K. Optimization of the duration of cryogenic processing to maximize wear resistance of AISI D2 steel. *Cryogenics,* 2009, vol. 49, pp. 176 - 184.

[6] DAS, D., DUTTA, A.K., TOPPO, V., RAY, K.K. Effect of deep cryogenic treatment on the carbide precipitation and tribological behavior of D2 steel. *Materials and Manufacturing Processes*. 2007, vol. 22, pp. 474 - 480.

[7] DAS, D., DUTTA, A.K., RAY, K.K. Sub-zero treatments of AISI D2 steel: Part I. Microstructure and hardness. *Materials Science and Engineering,* 2010, vol. A527, pp. 2182 - 2193.

[8] AMINI, K., AKHBARIZADEH, A., JAVADPOUR, S. Effect of Carbide Distribution on Corrosion Behavior of the Deep Cryogenically Treated 1.2080 Steel. *Journal of Materials Engineering and Performance,* 2016, vol. 25, pp. 365 - 373.

[9] AKHBARIZADEH, A., JAVADPOUR, S., AMINI, K. Investigating the effect of electric current flow on the wear behaviour of 1.2080 tool steel during the deep cryogenic heat treatment. *Materials and Design,* 2013. vol. 45, pp. 103 - 109.

[10] AKHBARIZADEH, A., JAVADPOUR, S., AMINI, K., YAGHTIN, A.H. Investigating the effect of ball milling during the deep cryogenic heat treatment of the 1.2080 tool steel. *Vacuum,* 2013, vol. 90, pp. 70 - 74.

[11] AMINI, K., AKHBARIZADEH, A., JAVADPOUR, S. Investigating the effect of holding duration on the microstructure of 1.2080 tool steel during the deep cryogenic treatment. *Vacuum,* 2012, vol. 86, pp. 1534 - 1540.

[12] DAS, D., RAY, K.K., DUTTA, A.K. Influence of temperature of sub-zero treatments on the wear behaviour of die steel. *Wear*. 2009. vol. 267, pp. 1361 - 1370.

[13] TYSHCHENKO, A.I., THEISEN, W., OPPENKOWSKI, A., SIEBERT, S., RAZUMOV, O.N., SKOBLIK, A.P., SIROSH, V.A., PETROV, J.N., GAVRILJIK, V.G. Low-temperature martensitic transformation and deep cryogenic treatment of a tool steel. *Materials Science and Engineering,* 2010, vol. A527, pp. 7027 - 7039.

[14] SURBERG, C.H., STRATTON, P. LINGEHOELE, K. The effect of some heat treatment parameters on the dimensional stability of AISI D2. *Cryogenics,* 2002, vol. 48, pp. 42 - 47.

[15] JURČI, P., DOMÁNKOVÁ, M., ČAPLOVIČ, L., PTAČINOVÁ, J., SOBOTOVÁ, J., SALABOVÁ, P., PRIKNER, O., ŠUŠTARŠIČ, B., JENKO, D. Microstructure and hardness of sub-zero treated and no tempered P/M Vanadis 6 ledeburitic tool steel. *Vacuum*, 2015, vol. 111, pp. 92 - 101.

[16] GHASEMI-NANESA, H., JAHAZI, M. Simultaneous enhancement of strength and ductility in cryogenically treated AISI D2 steel. *Materials Science Engineering,* 2014, vol. A598, pp. 413 - 419.

[17] PTAČINOVÁ, J., SEDLICKÁ, V., HUDÁKOVÁ, M., DLOUHÝ, I., JURČI, P. Microstructure Toughness relationships in sub-zero treated and tempered Vanadis 6 steel compared to conventional treatment. *Materials Science and Engineering,* 2017, vol. A702, pp. 241-258.

[18] GAVRILJUK, V.G., THEISEN, W., SIROSH, V.A., POLSHIN, E.V., KORTMANN, A., MOGILNY, G.S., PETROV, Yu. N., TARUSIN, Y.V. Low-temperature martensitic transformation in tool steels in relation to their deep cryogenic treatment. *Acta Materialia,* 2013, vol. 61, pp. 1705 - 1715.

[19] OPPENKOWSKI, A., WEBER, S., THEISEN, W. Evaluation of factors influencing deep cryogenic treatment that affect the properties of tool steels. *Journal of Materials Processing Technology,* 2010, vol. 210, pp. 1949 - 1955.

[20] JURČI, P., DOMÁNKOVÁ, M., HUDÁKOVÁ, M., PTAČINOVÁ, J., PAŠÁK, M., PALČEK, P. Characterization of microstructure and tempering response of conventionally quenched, short- and long-time sub-zero treated PM Vanadis 6 ledeburitic tool steel. *Materials Characterization,* 2017, vol. 134, pp. 398-415

[21] McEVILY, A.J., KU, R.C., JOHNSTON, T.L. The Source of Martensite Strength, *Transactions of Metallurgical Society AIM,* 1966, vol. 236, pp. 108 - 114.

[22] PIETIKAINEN, J. Effects of the aging of martensite on its deformation characteristics and on fracture in Fe - Ni - Si - C steel. *Journal of Iron and Steel Institute,* 1968, vol. 1, pp. 74 - 78.

[23] GAVRILJUK, V.G., SIROSH, V.A., PETROV, Yu. N., TYSHCHENKO, A.I., THEISEN, W., KORTMANN, A. Carbide precipitation during tempering of a tool steel subjected to deep cryogenic treatment. *Metallurgical and Materials Transactions,* 2014, vol. 45A, pp. 2453 - 2465.

[24] VILLA, M., PANTLEON, K., SOMERS, M.A.J. Evolution of compressive strains in retained austenite during sub-zero Celsius martensite formation and tempering. *Acta Materialia,* 2014, vol. 65, pp. 383-392.

[25] DURICA, J., PTACINOVA, J., HUDAKOVA, M., KUSÝ, M., JURČI, P. Microstructure and Hardness of Cold Work Vanadis 6 Steel after Subzero Treatment at-140 degrees C. *Advances in Materials Science and Engineering,* 2018, Article Number: 6537509.