

FRACTURE TOUGHNESS OF LEDEBURITIC VANADIS 6 STEEL AFTER SUB-ZERO TREATMENT FOR 17 H AND DOUBLE TEMPERING

LOMNA ŽILAVOST LEDEBURITNEGA JEKLA VANADIS 6 PO TOPLOTNI OBDELAVI S 17-URNIM PODHLAJEVANJEM IN DVOJNIM POPUŠČANJEM

Jana Ptačinová¹, Peter Jurčí¹, Ivo Dlouhý²

¹Institute of Materials Science, Faculty of Materials Science and Technology Trnava, Jána Bottu 25, 917 24 Trnava, Slovakia

²Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Žitkova 22, 61662 Brno, Czech Republic
jana.ptacinova@stuba.sk

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The influence of sub-zero treatment on the fracture toughness of Cr-V ledeburitic steel Vanadis 6 was examined, in comparison with the same material processed without the sub-zero period. The microstructure of the material consists of the matrix and several types of carbides – eutectic carbides (ECs), secondary carbides (SCs) and small globular carbides (SGCs). Small amounts of retained austenite were also present in the microstructure, but only in the untempered or low-temperature tempered steel. Sub-zero treatment increases the amount of small globular carbides. On the other hand, tempering results in a decrease in the population density of these particles. The fracture toughness of conventionally heat-treated steel firstly increases with the tempering, but then it decreases rapidly when the steel is tempered at the temperature of secondary hardening. In the case of the sub-zero treated material, the fracture toughness is correspondingly lower when the material is tempered at low temperatures, but it becomes slightly higher in the temperature range normally used for secondary hardening. Generally, one can say that the fracture toughness follows well the values of the hardness of the material, except in the narrow temperature range in the case of the sub-zero treated steel, where a "window" for a simultaneous enhancement of hardness and toughness exists.

Keywords: ledeburitic steel, sub-zero treatment, fracture toughness, carbides, fracture surface

Avtorji so preučevali vpliv standardne toplotne obdelave v kombinaciji s podhlajevanjem na lomno žilavost Cr-V ledeburitnega jekla Vanadis 6 v primerjavi z enakim materialom brez podhlajevanja. Mikrostruktura materiala sestoji iz matrice in več vrst karbidov – eutektičnih karbidov (angl. ECs), sekundarnih karbidov (angl. SCs), in manjših globularnih karbidov (angl. SGCs). Majhne količine zaostalega avstenita so bile prisotne v mikrostrukturi, vendar le v nepopuščnem ali v nizkotemperaturno-popuščnem jeklu. Podhlajevanje povečuje količino manjših globularnih karbidov. Po drugi strani pa se s popuščanjem zmanjšuje populacijska gostota teh delcev. Lomna žilavost konvencionalno toplotno obdelanega jekla se najprej povečuje s poviševanjem temperature popuščanja vendar se prične hitro zmanjševati (zniževati), ko je prekoračena temperatura sekundarnega utrjevanja. Pri toplotni obdelavi materiala s podhlajevanjem, se lomna žilavost posledično znižuje, ko je le-to popuščeno na nižjih temperaturah, vendar se rahlo povečuje v območju temperature, ki se navadno uporablja za sekundarno utrjevanje. V splošnem je možno reči, da lomna žilavost sledi vrednostim trdote materiala, razen v primeru toplotno obdelanega jekla v kombinaciji s podhlajevanjem, kjer v zelo ozkem temperaturnem intervalu obstaja "temperaturno okno". Tam dosežemo istočasno povečanje žilavosti in trdote.

Ključne besede: ledeburitno jeklo, podhlajevanje, lomna žilavost, karbidi, lomna površina

1 INTRODUCTION

In modern tooling, it is necessary that the tool materials have a good wear performance and high hardness, accompanied with at least acceptable toughness. Otherwise the tools might fail very early, before any wear damage can occur.

The toughness of steels quantifies their resistance to the initiation of brittle fracture under given conditions. For real tools made of Cr- and Cr-V ledeburitic steels, the toughness determines their capability to resist either the chipping or a total failure. The toughness, being expressed by the three-point bending strength, σ_b , of brittle ledeburitic steels is the highest when the material is soft-annealed and it decreases as a consequence of the application of heat treatment. However, the higher the

austenitizing temperature (T_A), the lower is the toughness and, at a constant T_A , the tempering first results in an increase in the toughness, which is followed by its decrease at the maximum secondary hardening.¹ Hence, the application of proper heat treatment is a compromise between the requirements for the maximum hardness and at least acceptable toughness.

There is a shortage of literature pertaining to the effect of the sub-zero treatment (SZT) on the toughness. D. N. Collins and J. Dormer reported that the toughness of the AISI D2 steel decreased with an application of the SZT up to the temperature of -70 °C, which was followed by a moderate increase in the toughness when a lower processing temperature was used for the SZT.^{2,3} It should be noticed here that the samples were low-temperature tempered at 200 °C.

The fracture toughness (K_{IC}) (the resistance against the crack propagation) of the ledeburitic steels can be quantified using either pre-cracked (bend) specimens or chevron-notch technique.^{4–6} The fracture toughness of ledeburitic steels is very low, whereas the K_{IC} values usually follow the three-point bending strength.

The effect of the SZT on the fracture toughness was reported by several groups of investigators. D. Das et al., for instance, pointed out that the SZT carried out at temperatures of (–75, –125 and –196) °C led to a decrease in the fracture toughness compared to conventionally heat-treated samples when low-temperature tempered at 210 °C for 2 h.⁶ The variations in the fracture toughness were attributed to the reduction of the retained-austenite amount and to the increase in the population density of SGCs in the microstructure after the SZT. In our recent paper, it was demonstrated that the effect of the SZT on the toughness is marginal, but the effect of this processing on K_{IC} is rather positive when the material is tempered at the temperature normally used for secondary hardening.⁷

The main goal of the current investigations is to characterize the variations in the toughness and fracture toughness of powder-metallurgy (PM) ledeburitic steel Vanadis 6, as a result of different heat-treatment schedules (austenitizing, sub-zero treatment, tempering) and to relate them to the microstructural alterations, due to these treatments, like the reduction of the retained-austenite amount, increase in the carbide count and others.^{8–10}

2 MATERIAL AND EXPERIMENTAL METHODS

2.1 Material and processing

The experimental material was tool steel Vanadis 6 with nominally 2.1 % C, 1.0 % Si, 0.4 % Mn, 6.8 % Cr, 1.5 % Mo, 5.4 % V and Fe as balance, made with PM. The conventional heat treatment (CHT) consisted of the following steps: heating up the material to the desired T_A (1050 °C) in a vacuum furnace, holding it at the temperature for 30 min and nitrogen gas quenching (5 bar). One set of samples was processed without the inclusion of the SZT between quenching and tempering, while the other samples were subjected to the SZT carried out at the temperature of –196 °C for 17 h. Double tempering (2 h + 2 h) was performed at temperatures in the range of 100–600 °C.

2.2 Experimental methods

Metallographic samples were prepared in the standard way and etched with a picric-acid reagent for the SEM observation. The microstructure was recorded using a JEOL JSM 7600 F device equipped with an EDS detector (Oxford Instruments), at an acceleration voltage of 15 kV. Details of the categorization of the carbides have been published recently.^{10,11} Macro-hardness measurements were completed using the Vickers (HV10) method. Five measurements were made on the metallo-

graphic specimens processed with any combination of heat-treatment parameters, and both the mean values and the standard deviations were then calculated. For the fracture-toughness determination, pre-cracked specimens predetermined for bending, with dimensions of 10 mm × 10 mm × 55 mm were used. For the pre-cracking of the samples, four bending fixtures and a resonance frequency machine (Cracktronic 8024) were used, which allowed the fatigue-crack initiation and further propagation through the actual frequency of the cycling to be controlled.

In addition, the crack development was checked on both sides of a sample using digital long-distance microscopic methods. Both the pre-crack preparation and bend tests were carried out at room temperature according to the ISO12137 standard.¹² For the test, an Instron 8862 machine was used and a loading rate of 0.1 mm/min was applied. Specimen deflection was measured by means of an inductive transducer integrated directly into the loading axis. In total, five samples were tested for each investigated condition. Fracture-surface morphology was investigated with a scanning electron microscope JEOL JSM 7600F with an EDS detector (Oxford Instruments). The topography of fracture surfaces was studied with a confocal laser scanning microscope Zeiss LSM 700. Three-dimensional topographical resolution was achieved using the ZEN 2009 software.

3 RESULTS AND DISCUSSION

3.1 Microstructure

SEM micrographs, **Figure 1**, show an example of the microstructure evolution of the material after the sub-zero treatment in liquid nitrogen (for 17 h) and tempering at different temperatures. The microstructure after the sub-zero treatment consists of a matrix made of martensite, a small amount of retained austenite and carbides (**Figure 1a**). As expected, the character of the matrix microstructure changes with increasing tempering temperature (**Figures 1b to 1f**). Due to the tempering, the martensite becomes more sensitive to the etching agent (the so-called tempered martensite). This is a commonly known fact and it is related to the precipitation of carbides during tempering. Retained austenite is transformed into martensite. The portion of carbides is also changed due to the tempering. The volume fractions of both eutectic carbides and secondary carbides are invariant over the range of the heat-treatment parameters used in the experiments. The population density of the small globular carbides increased with the application of the sub-zero treatment, but rapidly decreased with the increasing temperature of the tempering (**Figure 2**).

3.2 Hardness characteristics

The bulk hardness of the non-SZT and SZT Vanadis 6 steel, as a function of the tempering temperature, is

shown in **Table 1**. The as-quenched hardness of the conventionally heat-treated steel that was not tempered was 838 ± 4.33 HV10. The hardness of the SZT steel soaked in liquid nitrogen for 17 h and not tempered was correspondingly higher, e.g., 917 ± 7.58 HV10. These results show that the as-quenched bulk hardness of the Vanadis 6 steel was improved due to the sub-zero treatment. The hardness of all the samples then decreases with the increasing tempering temperature. Here it is interesting that the hardness of the SZT steel remains higher up to the tempering temperature of 450 °C and then it drops more intensely than that of the conventionally quenched samples. As a result, the hardness of the specimens tempered in the range of the temperatures commonly used for secondary hardening is lower for the SZT steel than for the steel achieved after conventional quenching and tempering.

3.3 Evaluation of the SZT effect on the fracture toughness

The fracture toughness vs. heat treatment schedules are presented in **Figure 3a** (CHT) and **Figure 3b** (SZT). In the case of the CHT, the fracture toughness of the untempered material was measured to be $16.4 \text{ MPa m}^{1/2}$ and it firstly increased, due to the tempering, to $19.6 \text{ MPa m}^{1/2}$ with a subsequent slight decrease; however, the decrease in the fracture toughness markedly accelerated, at the temperature normally used for secondary hardening, to a value of $14.8 \text{ MPa m}^{1/2}$. For the material subjected to the SZT in liquid nitrogen for 17 h, the fracture toughness before tempering was $13.3 \text{ MPa m}^{1/2}$

(i.e., lower than that of the CHT steel). Then, the fracture toughness increased with the tempering temperature to more than $15 \text{ MPa m}^{1/2}$ and just slightly decreased at a tempering temperature of 450 °C. It is interesting that the K_{IC} values were higher for the SZT steel than for the CHT steel at the temperature of secondary hardening (530 °C).

Table 1: Hardness of the Vanadis 6 ledeburitic steel

Tempering of samples	CHT		SZT	
	HV10	±	HV10	±
untempered	838	4.3	917	7.6
tempered at 170 °C	784	3.7	870	5.4
tempered at 330 °C	736	7	851	15.7
tempered at 450 °C	724	3	816	7.8
tempered at 530 °C	758	5.9	701	3

The behaviour of fracture toughness vs. heat treatment schedules can be classified as logical because one can expect higher K_{IC} at lower hardness and lower K_{IC} at elevated hardness. In other words, the application of the SZT decreases K_{IC} when the steel is low-temperature tempered. This fact should be considered by toolmakers and users of tools in all the cases when they expect an increase in the wear resistance due to a high hardness resulting from the sub-zero treatment. A very interesting fact was found for the material tempered at the temperature of secondary hardening – K_{IC} was higher for the SZT material. This is in good agreement with the recently published results where a similar tendency was found for the same SZT steel treated for 4 h and 10 h.⁷ One can say that this kind of K_{IC} behaviour can be expected because of the lower hardness of the SZT steel when tempered in this temperature range; however, in the mentioned paper, it was also found that the increase of K_{IC} is accompanied with a better wear performance of the material, due to the presence of a higher amount of carbides. The enhanced number of carbides, compared to the material after the conventional quenching, was also identified in the current work. Hence, one can expect that a sub-zero treated material would have a better wear performance, too. These results are also interesting from

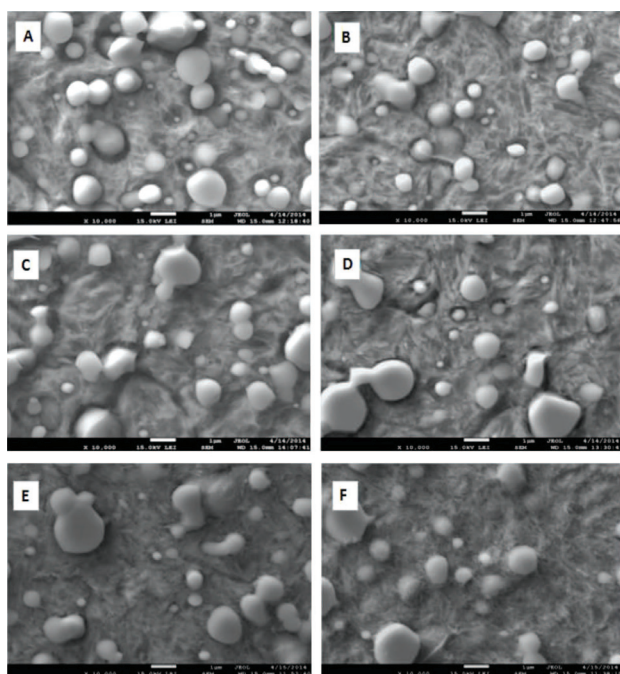


Figure 1: SEM micrographs showing the microstructure of Vanadis 6 ledeburitic steel: a) after quenching, subsequent SZT and b) tempering at 170 °C, c) 330 °C, d) 450 °C, e) 530 °C, f) 600 °C

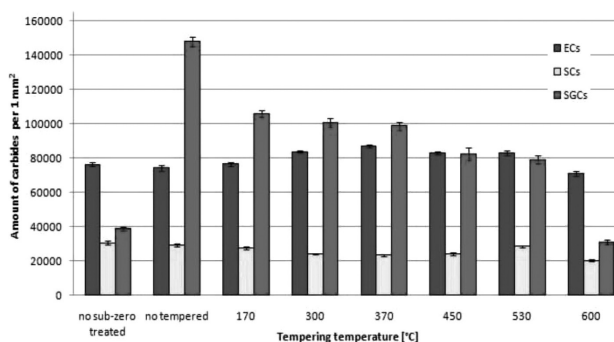


Figure 2: Amount of carbides in the Vanadis 6 steel after conventional quenching, SZT for 17 h and SZT and subsequent tempering at different temperatures

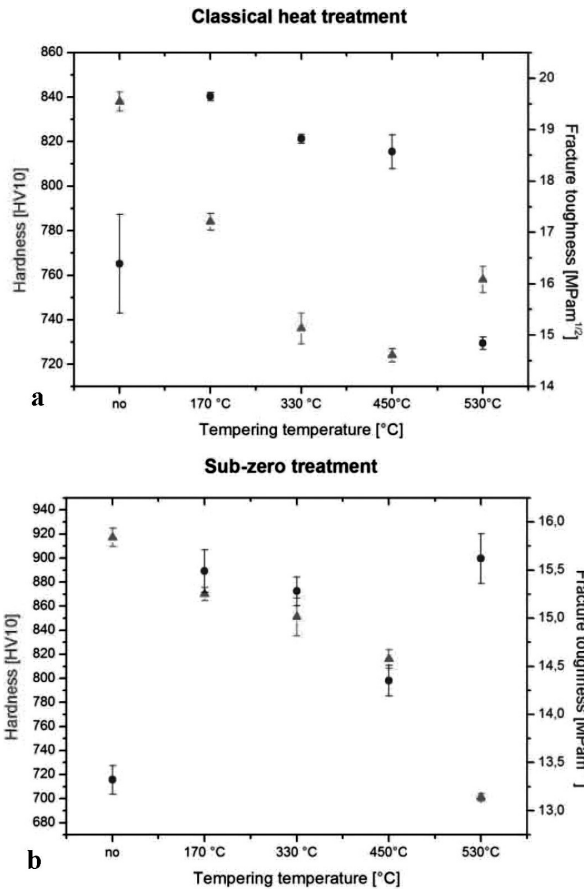


Figure 3: Hardness HV10 and fracture toughness K_{IC} of Vanadis 6 steel in dependence on the heat treatment applied: a) conventional quenching, b) sub-zero treatment

the point of view of industrial practice. They indicate that it is possible to increase the wear performance, along the toughness, of the material in a certain tempering-temperature range.

3.4 Fracture-surface morphology

The micro-mechanics of fracture propagation is demonstrated through representative SEM micrographs showing how the fractures appear in the cases of the steel that was sub-zero treated at -196 °C for 17 h and not

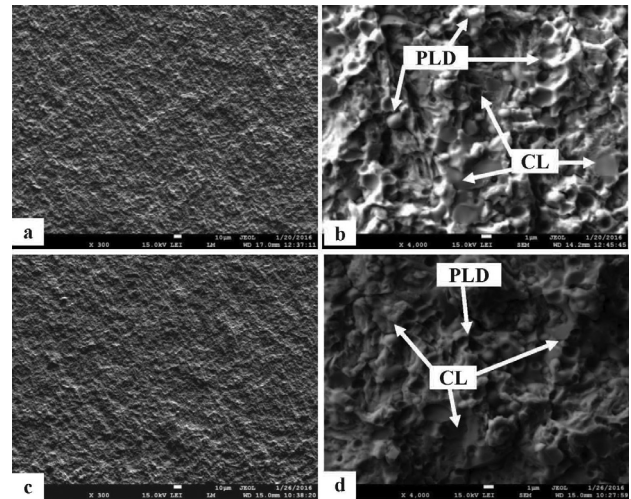


Figure 4: Representative SEM micrographs of the fracture surfaces of the specimens processed with: a) b) SZT at -196 °C for 17 h and tempered at 170 °C and c) d) SZT at -196 °C for 17 h and not tempered

tempered (the lowest K_{IC}) and the steel that was sub-zero treated at -196 °C for 17 h and tempered at 170 °C (higher K_{IC}). The fracture surface of the sample that was not tempered and the one that was tempered at 170 °C are shown in **Figure 4**.

At first glance, there is not any difference between the mentioned fracture surfaces; they appear flat and shiny, **Figures 4a** and **4c**. However, a more detailed observation, at higher magnifications, makes it clear that the fracture surface of the sample after low-temperature tempering contains an enhanced number of sites with micro-plastic deformation (PLD), located mainly at the carbide/matrix interfaces, **Figure 4b**. On the other hand, there are very few such sites on the fracture surface of the untempered specimen, **Figure 4d**, and the fracture surface contains a number of cleavage facets/regions (CL).

The topographies of the fracture surfaces of the CHT Vanadis 6 steel tempered at 170 °C and sub-zero treated, and the untempered one, expressed with surface roughness R_a are shown in **Table 2** and **Figure 5**. The average roughness of the fracture surface of the conventionally quenched steel tempered at 170 °C was $4.2703 \pm 0.248\text{ }\mu\text{m}$.

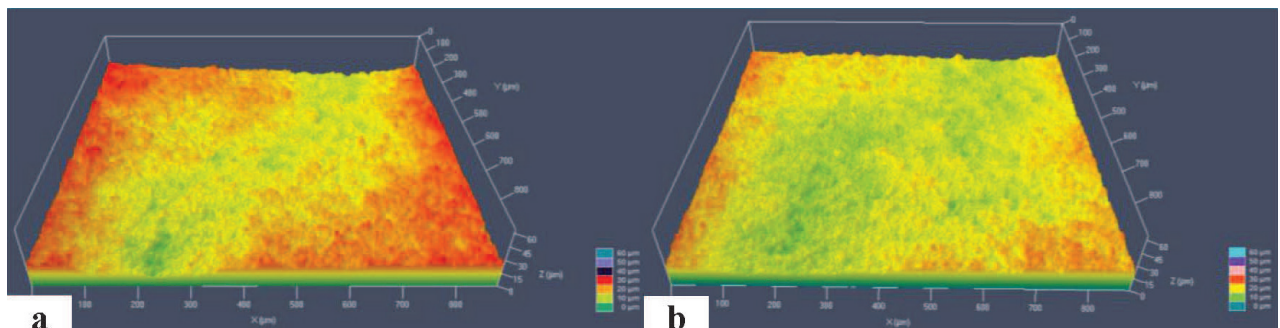


Figure 5: Confocal-microscope micrographs showing the fracture surfaces of K_{IC} specimens: a) CHT and tempered at 170 °C , b) SZT and not tempered

The roughness of the sub-zero treated and untempered Vanadis 6 steel was lower, i.e., $2.918 \pm 0.323 \mu\text{m}$. At this point, it should be noted that in this stage of experimental efforts, only the materials with the highest K_{IC} and the one with the lowest K_{IC} were checked with confocal microscopy. However, the measurements gave unambiguous differences in the roughness, where the higher roughness corresponded to the higher K_{IC} , i.e., to the conventionally quenched and low-temperature tempered sample.

Table 2: Roughness of the fracture surface of Vanadis 6 ledeburitic steel

No. of measurement	Tempered at 170 °C	SZT and untempered
1.	4.136	2.591
2.	4.750	3.507
3.	3.925	2.656
R_a (μm)	4.2703 ± 0.248	2.918 ± 0.323

4 CONCLUSIONS

The main conclusions based on the presented results are as follows:

- Sub-zero treatment reduces the amount of retained austenite and increases the population density of small globular carbides in the microstructure.
- The amount of small globular carbides decreases with the application of tempering treatment. The higher the tempering temperature, the more significant is the reduction of the carbide count.
- The hardness of the sub-zero treated material is higher than that of the conventionally quenched one. Also, this tendency is preserved when steel is low-temperature tempered.
- On the other hand, the hardness of the conventionally quenched steel becomes higher than that of the SZT one when tempered at the temperature of secondary hardening.
- The fracture toughness is generally higher for the conventionally quenched steel Vanadis 6 except in the case when the material is tempered to the secondary hardness. One can conclude that K_{IC} follows the reciprocal value of the hardness, i.e., the harder the material the lower is the fracture toughness.
- The differences in K_{IC} are reflected in the topographies of the fracture surfaces, represented by the

surface roughness – the higher the fracture toughness, the higher is the roughness of the fracture surface.

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