HEAT TREATMENT OF Cr- AND Cr-V LEDEBURITIC TOOL STEELS

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Resume

Cr- and Cr-V ledeburitic cold work tool steels belong to the most important tool materials for large series manufacturing. To enable high production stability, the tools must be heat treated before use. This overview paper brings a comprehensive study on the heat treatment of these materials, starting from the soft annealing and finishing with the tempering. Also, it describes the impact of any step of the heat treatment on the most important structural and mechanical characteristics, like the hardness, the toughness and the wear resistance. The widely used AISI D2 steel (conventionally manufactured) and Vanadis 6 (PM) are used as examples in most cases.

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1. Introduction

High carbon and chromium ledeburitic steels were developed during the World War I, as a possible substitution for high speed steels for cutting operations. However, it was early recognized that they have insufficient hot hardness and are too brittle for these purposes. On the other hand, Cr- ledeburitic steels gained a wide popularity in cold work applications, due to high wear resistance and compressive strength. Further development of this group of material had two main directions. The first is the effort to get a maximum toughness at an acceptable hardness and wear resistance. Typical representative of this group of materials is now widely used AISI D2 steel. The second trend can be characterized by the development of materials with increased wear resistance. It was found that the alloying with vanadium, typically up to 4 wt.%, combined with enhanced amount of carbon form extremely hard MC-carbides. When classically manufactured, unfortunately, the high vanadium containing steels have also important drawbacks. First of all, large segregations take place during slow solidification in industrial ingots, which leads to anisotropy of mechanical properties. The MC-carbides have a strong tendency to grow during solidification. As such, they make the forging and machining of the steels difficult or impossible. In industrial applications, when dynamically loaded, coarse MC-carbides are responsible for limited toughness. Therefore, the upper limit of vanadium content for the Cr-V ledeburitic steels manufactured by classical ingot metallurgy was found to be of about 4 wt.% V.

For the manufacturing of Cr-V ledeburitic steels with higher vanadium content, the only possible way how to produce them is the powder metallurgy (P/M) of rapidly solidified particles. The technology, based on the rapid solidification

Typical Cr- and Cr-V ledeburitic steels.

Material	Chemical composition (wt.%)			
	C	Cr	V	Others
D3 (19 436, 1.2080, X210Cr12)	1.95	12	-	-
D2 (19 573, 1.2379, X155CrVMo12.1)	1.5	12	1.1	1.2 Mo
K190 (1.2380, X220CrVMo 13 4)	2.3	12.5	4	1.1 Mo
Vanadis 6	2.1	6.8	5.4	1.5 Mo
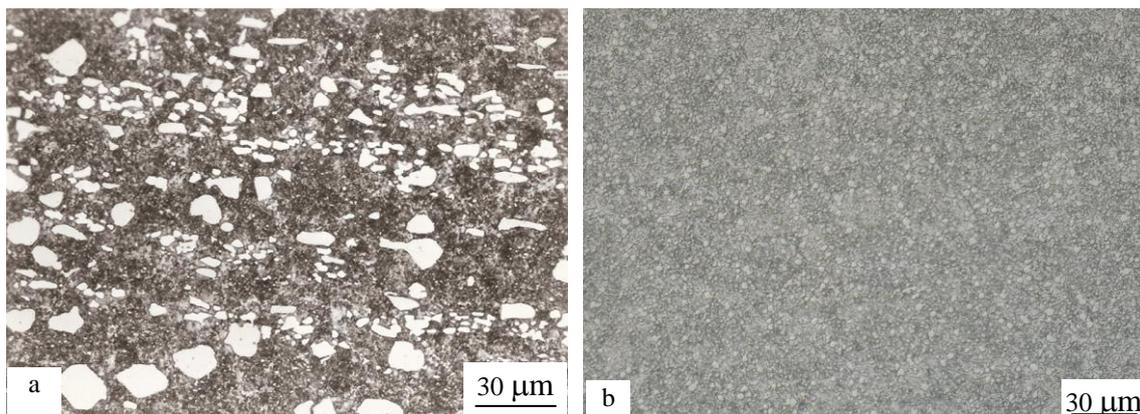
Fig. 1. Light micrographs showing the microstructure of ingot metallurgy made Cr-V ledeburitic steel (2% C, 7.8% Cr, 6% V) and PM made steel Vanadis 6 of similar chemical composition.
(full colour version available online)

of supercooled melt in small droplets, restricts the segregations in both the time and the space. As a result, materials with an excellent combination of isotropic and fine structure and high level of mechanical properties can be fabricated (P/M steels). The vanadium content, and proportionally also the amount of carbon, can be increased up to more than 10% without the risks of difficulties in forging and/or machining. The major representants of Cr- and Cr-V ledeburitic steels are presented in Table 1. Typical examples of the microstructures of conventionally manufactured and P/M steel of similar chemical composition is in Fig. 1.

2. Heat treatment

2.1 As-delivered state

Cr- and Cr-V ledeburitic steels are normally distributed to the end-users in soft-annealed state. The main reason is to deliver the materials with acceptable low hardness suitable for machining operations. The microstructure after the soft annealing consists of alloyed pearlite, secondary (SCs) and eutectic (ECs) carbides, Fig. 2. As shown in the micrograph, Fig. 2a, for the P/M made Vanadis 6 steel, the carbides are fine and uniformly distributed throughout the matrix while those for the AISI D2-steel form clusters and bands oriented longitudinally to previous deformation, Fig. 2b. Previous investigations have shown that the equilibrium of Vanadis 6 is formed by the chromium-based M_7C_3-carbides and vanadium-based MC-carbides [1]. On the other hand, the M_7C_3 is only stable carbide for the AISI D2 steel [2].
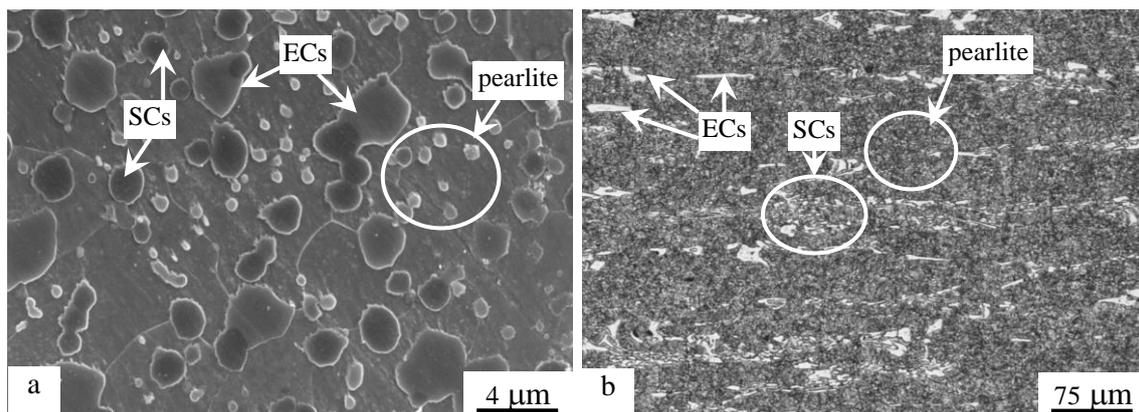
Fig. 2. Microstructure of soft annealed Vanadis 6 steel (SEM, left) and AISI D2 steel (light micrograph, right).

The soft annealing should be carried out carefully. The steels are heated up to a temperatures close above the A_1, with a hold at a given temperature for 2-4 h for Cr-ledeburitic steels and up to 24 h for Cr-V steels, respectively. Subsequently, the materials are cooled slowly, at a cooling rate of 10 °C/h or less, to a temperature close to the A_1, but below. A hold at this temperature then follows, with a subsequent slow furnace cooling.

2.2 Austenitizing

The tools made of Cr- and Cr-V ledeburitic steels are used in heat treated state only. Proper heat treatment is strictly recommended for the tools, to ensure the appropriate hardness, strength and wear resistance. On the other hand, this procedure should be carried out carefully. Otherwise, the risk of lowering of toughness can become. The most convenient heat treatment of this type of steels is so-called vacuum heat treatment.

The first step in the heat treatment is austenitizing. Because of poor thermal conductivity of the materials, the heating up to final temperature should be slow, with several ramps enabling to minimize thermal gradients between surface and core and subsequent too large distortion of components.

The transformation of the 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 these materials 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. It is worth noticing that the role of MC- and M_7C_3-phases is clearly different since they differ considerable in thermal stability. For example, soft-annealed Cr-V ledeburitic steel Vanadis 6 contains 16 vol.% of M_7C_3-carbides and 13 vol.% of MC-carbides [3, 4]. The M_7C_3-carbides underwent almost complete dissolution in the austenite up to 1100 °C while the MC-phase amount was reduced only slightly also after austenitizing at 1200 °C [4]. The dissolution of carbides leads to saturation of the matrix with alloying elements. Since the chromium rich carbides undergo the dissolution in a considerably larger extent than the MC-phase, the absolute value of the chromium amount in the matrix increases more rapidly, Tab. 2.

The situation in the case of Cr-ledeburitic steels is quite different. They do not contain the MC-carbides and the M_7C_3-phase is only stable carbide in most of these materials. This carbide undergoes dissolution in the austenite, in a similar way to that of Cr-V steels. However, a part of this phase is formed during the solidification in the eutectic mixture and

Amount of alloying elements (wt.%) dissolved in the matrix for the Vanadis 6 steel

Austenitizing temperature (°C)	1000	1050	1100	1150	1200
Cr	5.66 ± 0.56	5.96 ± 0.22	6.85 ± 0.33	6.96 ± 0.15	7.12 ± 0.09
V	1.72 ± 0.32	1.76 ± 0.48	2.24 ± 0.53	2.18 ± 0.43	2.66 ± 0.37
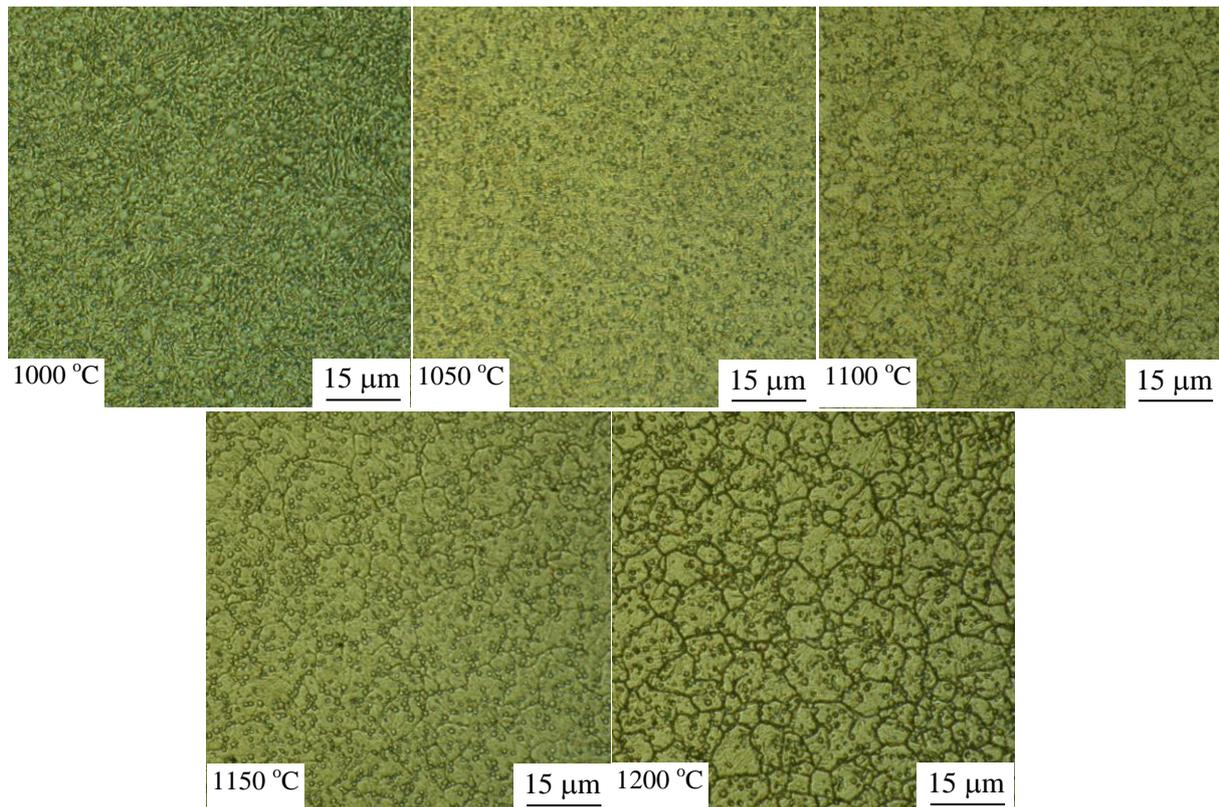
Fig. 3. Light micrographs showing the microstructure of the Vanadis 6 ledeburitic steel after quenching from different austenitizing temperatures.
(full colour version available online)

remains stable during austenitizing. As a result, as-quenched Cr-ledeburitic steels contain the M_7C_3-phase instead of MC-carbide, typical for Cr-V ledeburitic steels.

The upper limit of austenitizing temperature is given mainly by the beginning of the austenitic grains coarsening. The second reason why it is essential to keep the recommended austenitizing temperatures is the overall economy of the heat treating – the higher austenitizing temperature the increased costs are for realizing of the procedure. Figure 3 gives 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 slightly to 10 after quenching from the temperature of 1200 °C.

The Cr-ledeburitic steels are more sensitive to the grain coarsening. Mušutová [5] has found that the austenitic grain size of the AISI D2 steel increases from the size 9 (according to ASTM-method) to 7 when austenitized at temperatures of 1020 °C and 1120 °C, respectively. Typical microstructure of as-quenched AISI D2 steel is in Fig. 4.
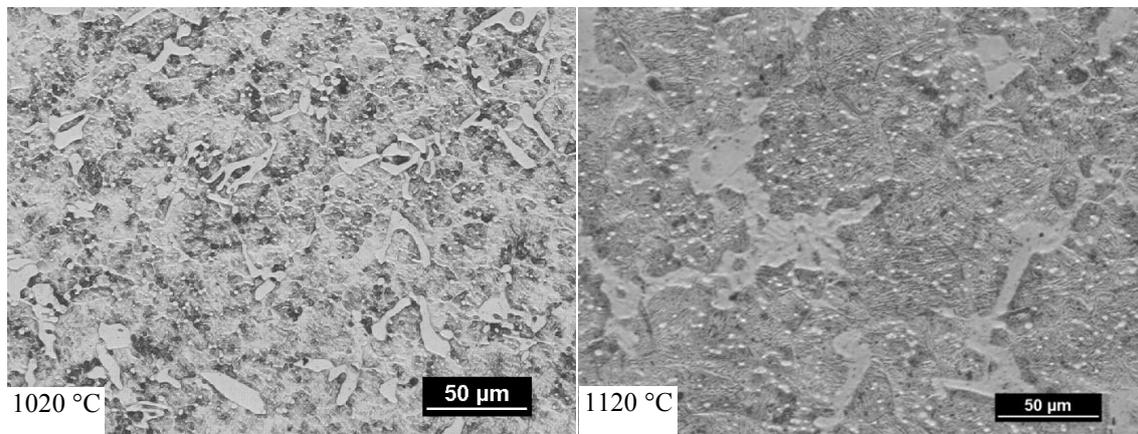
Fig. 4. Light micrographs showing the microstructure of the AISI D2 steel after quenching from different austenitizing temperatures.

2.3 Quenching

Standard medium used for quenching of ledeburitic steels is nitrogen gas under a high pressure. Due to good or excellent through hardenability of these steels there is no risk of hardness loss and, in addition, the method enables to keep smooth and bright surfaces and to minimize the distortion of tools.

Increased saturation of austenite with carbon and alloying elements, Table 2, induces the decrease of the temperatures M_s and M_f, respectively. In classical hardening procedures, the materials are quenched to an ambient temperature. As the M_s - temperature is located normally around 100 °C and the M_f well below the zero, retained austenite (γ_R) can be found in as-quenched material. The portion of γ_R increases with increasing austenitizing temperature because of the decrease of both the M_s- and M_f-temperatures. When austenitized at temperatures above 1100 °C, also the M_s of Cr- and Cr-V ledeburitic steels lies below 0 °C and lower bainite can be found in the material [4].

2.4 Sub-zero treatment (SZT)

This operation, followed immediately after quenching, is aimed as a possible way how to improve several properties (hardness, toughness, wear resistance, dimensional stability) of Cr- and Cr-V tool steels. However, the effect of SZT on the microstructure and the mechanical properties of the materials is not doubtless and the mechanisms being postulated for the changes in the microstructure are not entirely clear yet.

At the current state of knowledge, the following alterations in properties of Cr- and Cr-V ledeburitic steels were established:

As-quenched hardness increase [6 - 11], which is depicted in Fig. 5.

Improvement in wear performance – this can make hundreds of percents for AISI D2 steel [7 - 10, 12, 13], with a maximum at 36 h of soaking time in liquid nitrogen. For AISI D3 steel, the improvements make of several times [14].

Shift of the secondary hardening peak to lower temperatures [6] or it´s disappearance [15], this is illustrated in Fig. 6 for the Vanadis 6 steel.

Improvement in the dimensional stability of mechanical parts [16].

Reduction in toughness and fracture toughness for AISI D2 steel [17] but slight increase for Vanadis 6 [18, 19].
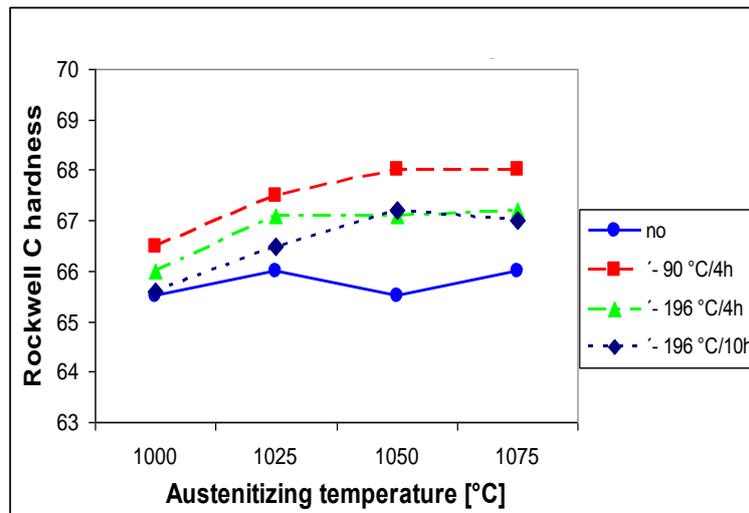
Fig. 5. As-quenched hardness of the Vanadis 6 steel as a function of austenitizing temperature and SZT.
(full colour version available online)

Tempering temperature

No- SZT

SZT
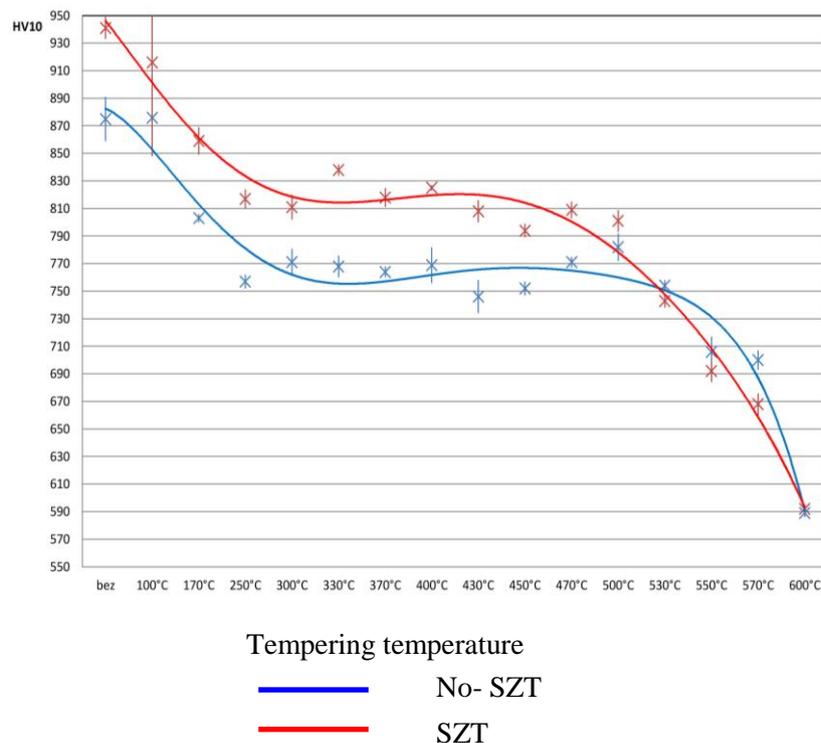
Fig. 6. Tempering chart of Vanadis 6 steel quenched from 1050 °C and no-SZT/SZT.
(full colour version available online)

These improvements/deteriorations in wear performance and other properties have been attributed to considerable alterations in the substructure of the material. They can be summarized as follows: i) more complete $\gamma - \alpha'$ transformation (reduced portion of γ_R), ii) alterations in the lattice parameters of the martensite [20, 21], iii) more extensive plastic deformation of virgin martensite formed during the SZT, connected with very high dislocation density inside the martensitic domains [11, 20, 22], iv) precipitation of nanosized coherent η (or ε) - carbides on subsequent tempering/re-heating from sub-zero temperature [17, 22], v) higher population density (and refinement) of small carbides

in as-tempered (at low temperature) SZT material [23]. This is illustrated for re-heated SZT material up to a room temperature in Fig. 7.
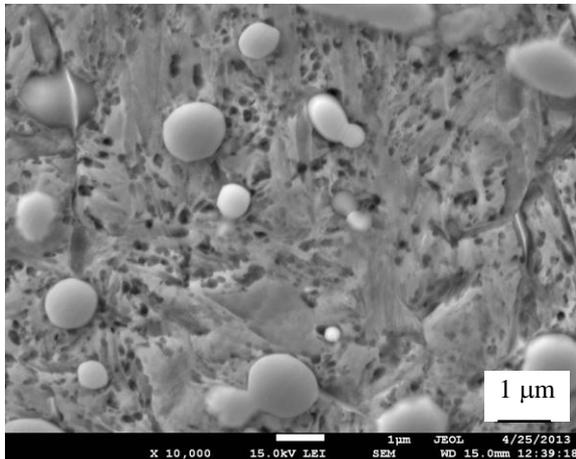
Fig. 7. SEM micrograph showing the microstructure of Vanadis 6 steel after quenching from 1000 °C and SZT at – 196 °C/4 h.

2.5 Tempering

As-quenched Cr- and Cr-V ledeburitic steels contain the α' (or a mixture of martensite and bainite), γ_R and undissolved carbides. When sub-zero processed, the structure of some constituents can differ from those conventionally heat-processed as described in chapter 2.4.

Tempering should follow the quenching and/or sub-zero treatment as soon as possible. Otherwise, the γ_R could have been stabilized. It is necessary to temper at least twice in order to fully convert the γ_R into the α' and to temper the newly formed (secondary) martensite. During the tempering, the alloying elements and carbon diffuse out from solid solutions forming precipitates responsible for secondary hardening effect. Cooling down from the tempering temperature induces the transformation of γ_R to new martensite.

The final as-tempered hardness of Cr- and Cr-V ledeburitic steels is a result of the competition between the softening of the martensite due to carbides precipitation,

the precipitation of carbides itself and the γ_R to α' transformation (both cause the strengthening). For discussed materials, there is a hardness decrease up to the tempering temperature of about 350 °C, since the softening of martensite is the dominant process and other above mentioned sub-processes play only minor role. At higher temperatures, the sub-processes of the transformation of retained austenite and carbides precipitation become great importance and the secondary hardness peak occur at the tempering temperature close 500 °C. Beyond the maximum of secondary hardness, the Cr-V ledeburitic steels undergo the softening because of coarsening of precipitates and also due to the fact that during the last tempering cycle, no more austenite can transform to the martensite.

The material tempered after sub-zero period behaves in a different way, Fig. 6. In our previous works [18, 24] it was established that the tempering at the temperatures of secondary hardness peak led to lower hardness of SZT material than that of no SZT. This difference in tempering response can be attributed to the alterations in sub-structure of the martensite, namely to the facts that

i) virgin martensite formed at very low temperatures contains considerably enhanced number of dislocation, due to extensive plastic deformation

ii) carbon atoms are segregated/clustered at the dislocations, due to their capture by moving dislocations. These carbon clusters can serve as nucleation sites of precipitated carbides during subsequent heating/tempering.

This leads to acceleration of phase transformations during subsequent tempering and the SZT steels become overtempered at temperatures normally used for the tempering of these materials to the secondary hardening peak. Similar results have been established by Stojko for ball bearing steels [25] and Das and Pellizzari for AISI D2 steel [15, 23, 26].

Moreover, the contribution of the γ_R to α' transformation, which proceeds during the cooling down from the tempering temperature, to the final hardness is less significant because of reduced γ_R content.

3. Mechanical properties

3.1 Hardness

The Cr- and Cr-V ledeburitic steels have a heterophaseous composition from the manufacturing procedure up to the final use of the tools. Therefore, the hardness of the materials is a result of the synergistic effect given by the superposition of the contribution of the matrix and that of the carbides. However, the role of the structural constituents upon the hardness is specifically given.

In the as-annealed condition, the matrix comprises the ferrite and its hardness is low. The hardness of the material is then mainly determined by the quality, size and distribution of carbides. Within the group of Cr- and Cr-V ledeburitic steels, the as-annealed hardness is mainly influenced by the amount of M_7C_3- and MC-carbides, respectively.

In as-quenched state, the matrix is responsible for a high hardness of the materials. Heat treatment leads to formation of matrix consisting mainly of martensite and a certain portion of retained austenite. When sub-zero processed, the hardness of steels is increased by several HRC units due to more complete martensitic transformation. Tempering at the temperatures up to 450 °C induces slight hardness decrease. Above that, the hardness increases due to the complex precipitation/transformation process. Beyond the maximum of the secondary hardness, the coarsening of precipitates proceeds and the hardness decreases. In the case of sub-zero treated steels, the tempering response of the materials is different as discussed in previous paragraphs.

3.2 Wear resistance

The resistance of Cr- and Cr-V ledeburitic steels against abrasive wear is determined by the amount of large primary, eutectic and undissolved part of secondary carbides. But, also the character of the abrasive particles plays an important role. If the abrasive particles are harder than the hardest structural component of steels, no improvement of the wear resistance can be expected [27]. In the case of softer abrasive particles like SiO_2, on the other hand, enhanced volume fraction of hard MC primary carbides leads to an improvement of the wear resistance by 75 – 100 %. Nevertheless, also in the case of the SiO_2-abrasive particles the situation is more complex – according to Jacobson [28], better wear resistance of the tool steels can be expected only if the SiO_2-particles are smaller than 100 μm.

If the Cr- and Cr-V ledeburitic steels undergo the adhesive wear, the presence of hard carbides itself is not relevant for better wear resistance [29]. It has been established that the interparticle spacing of carbide particles is the dominant factor influencing the adhesive wear resistance whereas this parameter has to be as minimal as possible [31]. Otherwise, the contact area between relatively softer matrix and counterpart material and, the probability of adhesion also, increases.

3.3 Three point bending strength

The three point bending strength is a standard measure of the toughness of Cr- and Cr-V ledeburitic steels. For its determination, standard samples with the dimensions 10 x 10 x 100 mm are used. Standard load rate is 1 mm/min and the samples are loaded in the centre.

The austenitizing and subsequent quenching increases the hardness of Cr- and Cr-V ledeburitic steels, which results in lowering of three point bending strength. The lowering of
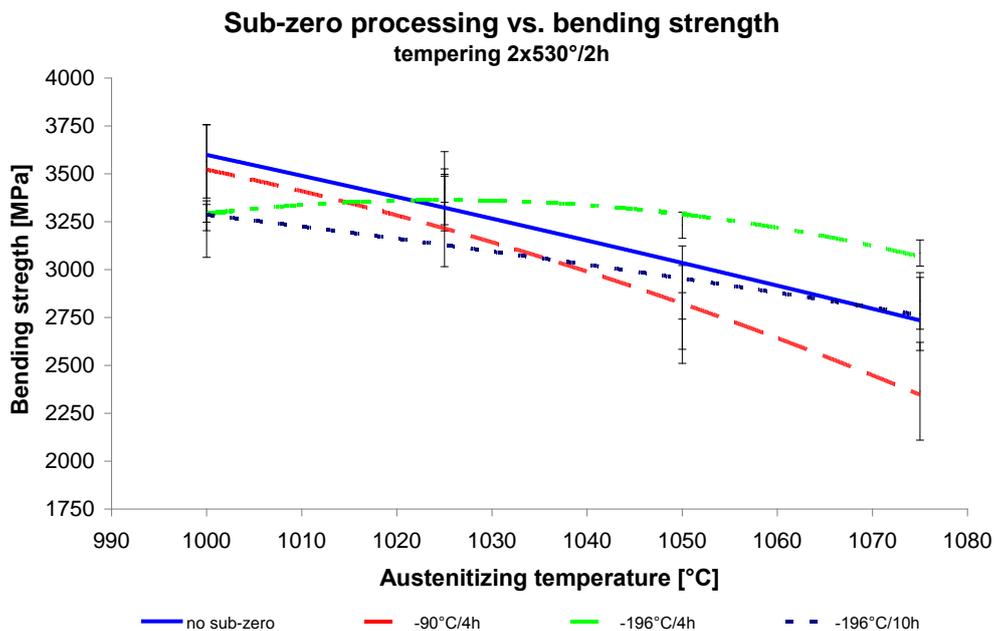
Fig. 8. Effect of austenitizing temperature and SZT on the three point bending strength.

(full colour version available online)
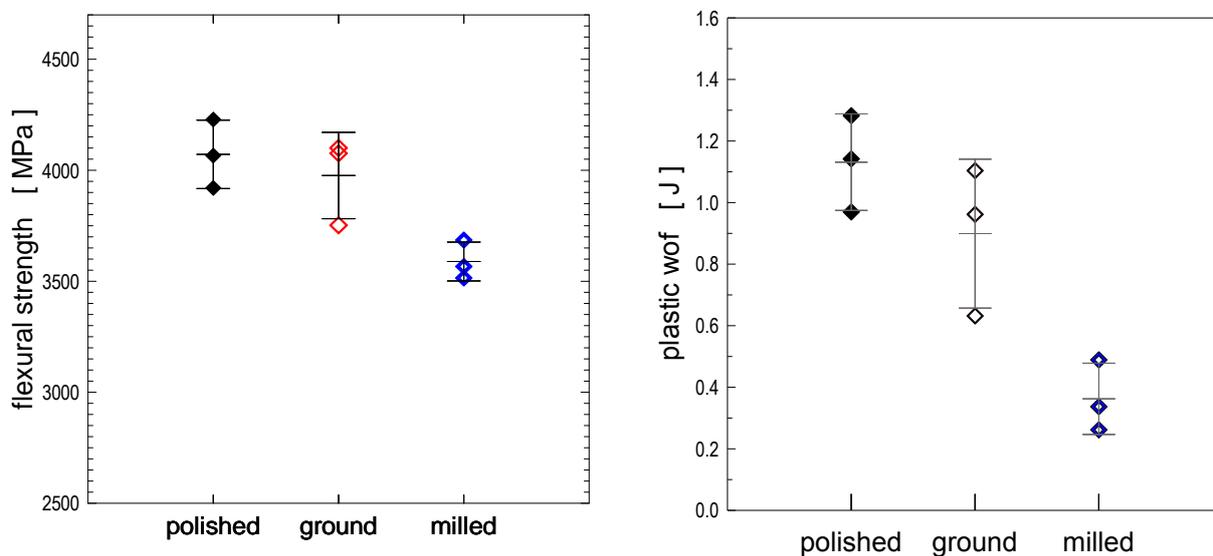
Fig. 9. Flexural strength and plastic component of work of fracture for heat processed samples from the Vanadis 6 ledeburitic steel with various surface quality: polished – up to mirror finish, ground ($R_a = 0.1$) and milled ($R_a = 6.3$).

(full colour version available online)

the three point bending strength is more significant with increasing austenitizing temperature because of grains coarsening, Fig. 8 [31].

Low-temperature tempering results in slight increase of the three point bending strength because of the martensite softening. The increase of the tempering temperature to the range typical for the secondary hardness peak leads to lowering of three point bending strength. The first process responsible for that is the $\gamma_R - \alpha'$ transformation. At the same time, the

precipitation of carbides, from the martensite and the retained austenite, contributes to the embrittlement of the materials.

The sub-zero treatment does not have a doubtless impact on the toughness of Cr- and Cr-V ledeburitic steels. The toughness of as-quenched steels as well as that of low-temperature tempered is lower due to the SZT, due to higher hardness and internal stresses in the material. On the other hand, the toughness of no-SZT- and SZT- steels is equal or slightly better for SZT-materials when tempered at the tempering temperatures typical for the secondary hardening peak [18, 31] – this is related to the fact that SZT – steels are overtempered (and slightly softer) after an application of such a tempering regime, Fig. 8.

It should be useful to mention the effect of the surface quality on the toughness of Cr- and Cr-V ledeburitic steels. It is worth noticing that these materials belong to a group of very brittle steels and any surface defect can highlight the improvement/deterioration of toughness given by the structural state of the materials. The problems concerning the effect of surface roughness on three point bending strength of Vanadis 6 steel (as a typical example) has been investigated recently [32]. It has been established that the worsened surface quality induces relatively sharp decrease as in the three point bending strength so the plastic component of the total work of fracture, Fig. 9.

3.4 Fracture toughness

Fracture toughness is a measure, which characterizes the resistance of ledeburitic steels against the crack propagation. In these materials, the fractures are typical by "dimple morphology". It is a result of mechanism of the fracture propagation, where two main mechanisms take place, Fig. 10. The first is the carbide cracking. The carbides undergoing fragmentation, are mostly the M_7C_3-particles. The second propagation mechanism is decohesion at the carbide/matrix interfaces, which is connected with plastic deformation of the matrix. It is logical that much more deformation energy is required for the second mechanism and, also that the capability of the matrix to be deformed is the most important parameter describing the resistance of the materials against crack propagation.

Therefore, in real materials, the hardness of the matrix is the main parameter influencing the fracture toughness. Berns et al. [33, 34] have reported that for the steel X210Cr12, the fracture toughness decreases from $31 - 50\,\mathrm{MPa.m}^{1/2}$ in soft-annealed state to $14 - 22\,\mathrm{MPa.m}^{1/2}$ after austenitizing and quenching. Subsequent tempering led to slight increase in K_{IC}, but the level of the K_{IC} increase was proportioned to the as-tempered hardness.

For Cr-V ledeburitic steels made via PM, there are practically not relevant data on their fracture toughness. Only in our recent work [18] we attempted to evaluate the fracture toughness of the Vanadis 6 steel after application of various heat treatment regimes. Two austenitizing temperatures (1000 and 1050 °C) were applied for a standard tempering temperature of 550 °C. Some specimens were also sub-zero processed in liquid nitrogen at -196 °C for 24 h. The results are depicted in Fig. 11. The fact that higher austenitizing temperature led to lower fracture toughness has been expected, since it is logical that the matrix hardness was higher in this case. Nevertheless, the average fracture toughness after performed heat processing of Vanadis 6 steel was found to be slightly higher for SZT material, as a logical consequence of lower hardness of the steel after tempering at 530 °C.
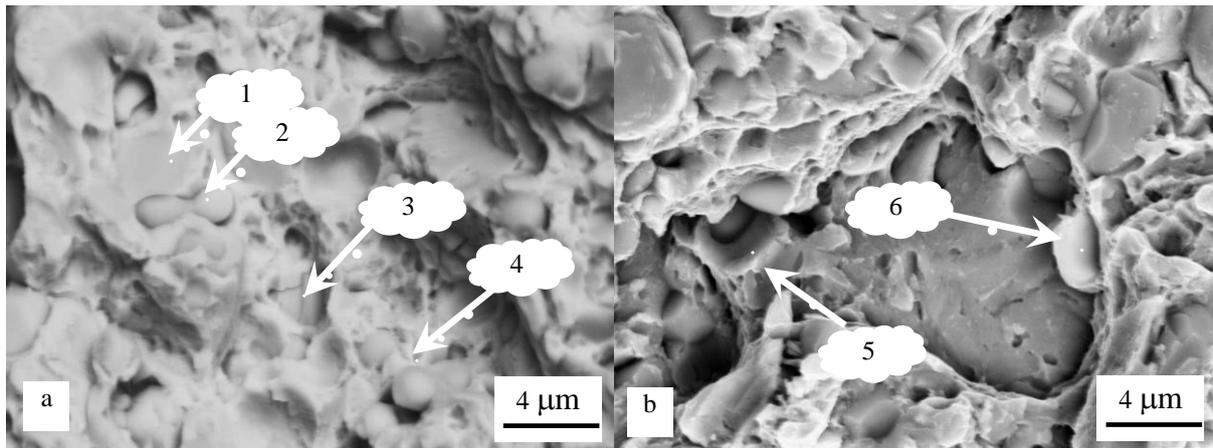
Fig. 10. Fracture surfaces of the samples made of Vanadis 6 steel, austenitized at 1075 °C, quenched and tempered 2x530 °C/2 h. a – no SZT, b – SZT -196 °C/4h, 1 – brittle cleavage, 2, 5 - decohesion at the carbide/matrix increase (more deformed matrix in the case of SZT), 3, 6 – fragmentation of M_7C_3, 4 – unaffected MC.
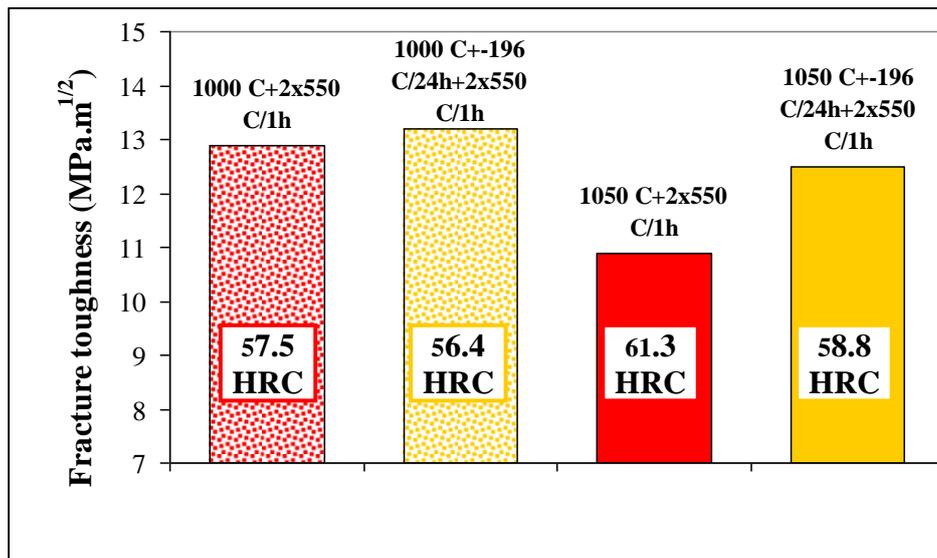
Fig. 11. Fracture toughness of SZT and no-SZT Vanadis 6 steel.
(full colour version available online)

4. Conclusions

After the soft annealing, the structure of Cr- and Cr-V ledeburitic steels is composed of the matrix and primary, eutectic and secondary carbides and spheroidized pearlite. The mechanical properties of the materials are determined mainly by the nature, volume fraction, size and distribution of carbides.

Chromium-based carbides undergo dissolution in the austenite during austenitizing while the vanadium-based particles remain stable up to high temperatures. The dissolution of carbides induces the enrichment of solid solution with carbon and alloying elements. If the optimal austenitizing temperature is exceeded, grain coarsening takes place and the retained austenite amount increases rapidly up to more than 50%.

Properly quenched materials have a hardness exceeding easily 60 HRC. Too high austenitizing temperature lowers the hardness, due to increased amount of retained austenite. When sub-zero treated, the materials have elevated as-quenched hardness, due to more complete γ to α' transformation.

Tempering of Cr-V ledeburitic steels is a complex process, which involves the softening of martensite, formation of new martensite from retained austenite during cooling down from the tempering temperature and precipitation of carbides during the holding at the tempering temperature. When properly done, the maximum secondary hardness peak occurs at the temperature of slightly above 500 °C.

The application of sub-zero treatment changes the tempering response of the materials – the hardness of SZT – steels is higher up to a certain tempering temperature and it is lower above that. Also, a loss or partial disappearance of secondary hardening peak occurs due to the SZT. These phenomena are attributed to changes in sub-structure of the martensite due to SZT, which are responsible for acceleration in the alterations in the materials proceeding on the tempering.

Abrasive wear resistance of Cr- and Cr-V ledeburitic steels is determined mainly by the volume fraction of undissloved carbides. However, if the abrasive particles are harder than the MC-phase, the effect of these carbides becomes negligible. In adhesive wear, the interparticle spacing of carbides is important.

Both the toughness and the fracture toughness of the Cr-V ledeburitic steels are influenced as follows: The higher austenitizing temperature the lower is both the toughness and the fracture toughness. The tempering has a positive effect on these characteristics, excepting the tempering temperatures range typical for secondary hardening peak, where slight decrease of toughness occurs. The application of SZT lowers the toughness of Cr- and Cr-V ledeburitic steels when they are tempered at low temperatures but, the effect can be adverse in the case of high temperature tempered materials.

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