MICROSTRUCTURAL EVALUATION OF CR-V LEDEBURITIC TOOL STEEL AFTER SUB-ZERO TREATMENT AT -140 °C

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Abstract

The microstructure, the phase constitution and hardness of Cr-V ledeburitic tool steel Vanadis 6 subjected to sub-zero treatment with several soaking time in nitrogen vapors have been investigated. The metallurgical aspects include reducing the amount of retained austenite and increasing carbide count, so wear resistant and dimensional stability are better as compared to conventionally heat treated material. The matrix is martensitic with certain amount of retained austenite, irrespectively to the time of sub-zero treatment. The amount of retained austenite has been significantly decreased from 20.2 vol. % to minimum 3.2 vol. % at 48 h soaking time. The microstructures have been characterized using the light microscopy, scanning electron microscopy and X-ray diffraction. The microstructure of sub-zero treated steel contains eutectic, secondary and increased count of small globular carbides. The count of small globular carbides for conventionally heat treated samples was around 48 x 10^3 / mm^2 and for sub-zero treated samples was increased more than four times with maximum 209 x 10^3 / mm^2 at 24 h soaking time. These particles have size of up 500 nm but 100 nm in most cases. The hardness has been increased as compared to no sub-zero treated samples from 875 ± 16 HV 10 up to 954.6 ± 14 HV 10 at holding time 48 h.

Keywords: Sub-zero treatment, carbides, Cr-V ledeburitic steel, retained austenite, microstructure

1. INTRODUCTION

Several challenges must be overcome in the pursuit of stronger, lighter and less expensive manufactured products. A common approach used in the past has been to heat-treat materials, which provides greater control over the range of properties that a given material may have. One of major problems of materials through by conventional heat treatment is the amount of retained austenite [1]. Sub-zero treatment (SZT) can produce harder, more wear-resistant materials with many other beneficial properties. SZT is the process of cooling a material to temperatures far below room temperature. This method of processing was first recognized when parts that were transported via train had been packed with dry ice, resulting noticeable increases in wear resistance [2]. The SZT is an add-on process to the conventional heat treatment (CHT) of steels, which can play an important role in finishing of tools and components. In particular, the SZT is frequently used for the processing of high carbon, high alloyed steels, where their austenite is not fully transformed to martensite. The group of Cr-V ledeburitic steels is a typical example [3].

The application of the SZT can result in considerable changes in microstructure and properties of processed steel. The main reason of that is, besides the variety of materials used for the investigations, a great variability of parameters of experimental techniques used. Some of the claimed benefits of SZT include increase wear resistance, increased dimensional stability and increase hardness of the materials.

We more often encounter investigation of SZT in liquid nitrogen at -196 °C. However, some researchs indicate that SZT at higher temperatures could have a better effect on changes in the microstructure. Reitz et al. [2], for instance showed an increase of hardness when the SZT was done at -140 °C compared to -196 °C. In the article the results of the investigation of differences in the microstructure, the amount of retained austenite and the hardness between the CHT Vanadis 6 steel and the same material at -140 °C are discussed in article.

1.1. Experimental

The experimental material was the 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 by PM. Samples for the microstructural investigations were cylinders with 17 mm in diameter and height of 6 mm. Conventional heat treatment (CHT) consisted of the following steps: heating up to the desired T_A (1050 °C) in a vacuum furnace, holding at the temperature for 30 min and nitrogen gas quenching (5 bar). Sub-zero treatment has been applied after quenching in liquid nitrogen vapor (-140 °C) for 4, 10, 17, 24, 36 and 48 hours. No tempering of the steel was carried out in order to highlight the microstructural changes due to SZT itself.

Metallographical samples were prepared by standard preparation line and etched with the Villela-Bain reagent for the light microscopy or with a picric acid for the SEM-observation.

The microstructure was recorded using the light microscope NEOPHOT 32 and the scanning electron microscope JEOL JSM 7600 F device equipped with an EDS-detector (Oxford Instruments), at an acceleration voltage of 15 kV. For details of the categorization of the carbides check the Refs [4, 5]. The amount of retained austenite (γ_R) was measured by X-ray diffraction, according to the ASTM E975-13 standard [6]. X-ray patterns were recorded using a Phillips PW 1710 device with filtered $Co_{\alpha1,2}$ characteristic radiation, in the range 20 - 144° of the two-theta angle. Macro-hardness measurements were completed by the Vickers (HV10) method. Each metallographic specimen was measured for 5 times, and the mean value and standard deviation from the measurements of each specimen was calculated.

2. EXPERIMENTAL RESULTS

Figure 1 show light micrograph of the examined steel after conventional quenching and after SZT done for 17 h. The microstructure consists of matrix and undissolved carbides. The matrix contains the martensite and retained austenite. Count of small globular carbides increase in comparison with convectional heat treated samples, but optical microscopy fails to bring more details. This was also confirmed in previous research by Das et al. [7] and Surberg at al. [8].

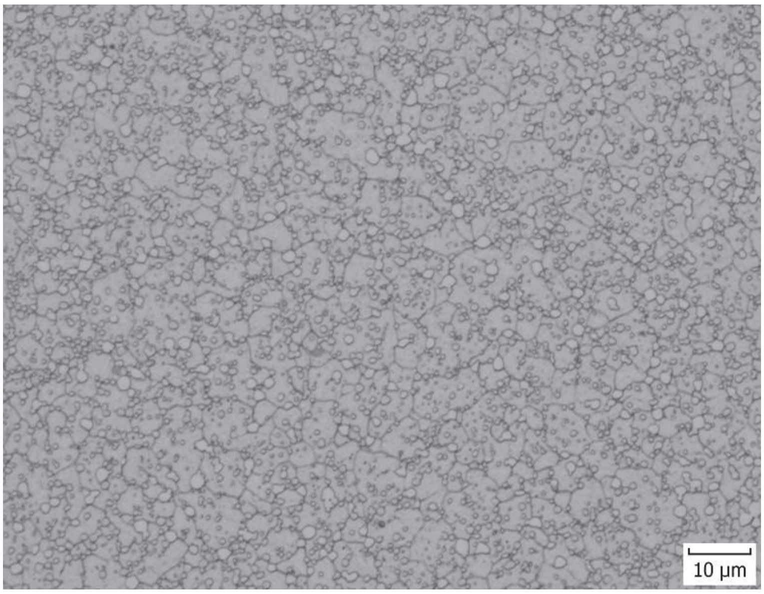

Figure 1 Light micrograph showing the microstructure of the Vanadis 6 after SZT at -140 °C for 17 h

SEM micrographs, **Figures 2 a), b**) show examples of typical microstructures of the Vanadis 6 tool steel after conventional heat treatment (CHT) and after SZT at -140 °C for 4 h. The microstructure is formed by matrix and three types of carbides: eutectic carbides (ECs), secondary carbides (SCs) and small globular carbides

(SGCs). The count of ECs and SCs is invariant over the heat treatment parameters range used in experiments because the ECs do not undergo the dissolution during austenitizing and the level of dissolution of the SCs is constant at fixed austenitizing temperature. The population density of SGCs increased with holding time at the temperature of sub-zero treatment. This is clearly evident by comparing of the microstructures in **Figure 2 a)** and b), respectively. **Figures 3 a), b)** show SEM micrographs of samples after SZT at -140 °C for 24 h and 48 h, in these micrographs it is clearly shown that the population density of SGCs is highlighted at long durations of SZT.

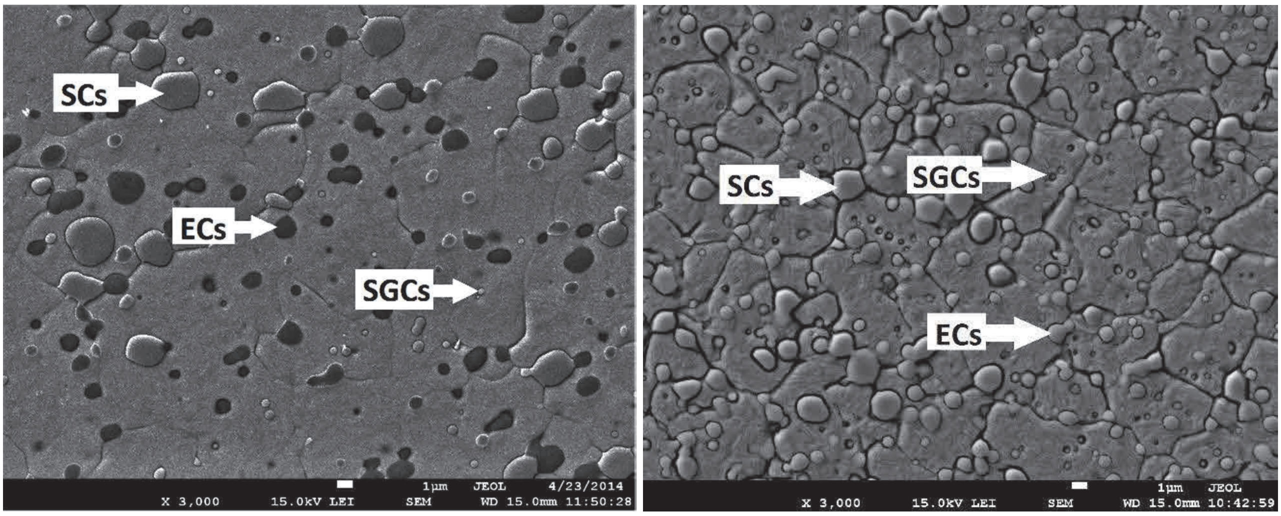

Figure 2 Microstructure of Vanadis 6 **a)** after CHT **b)** after SZT at -140 °C for 4 h

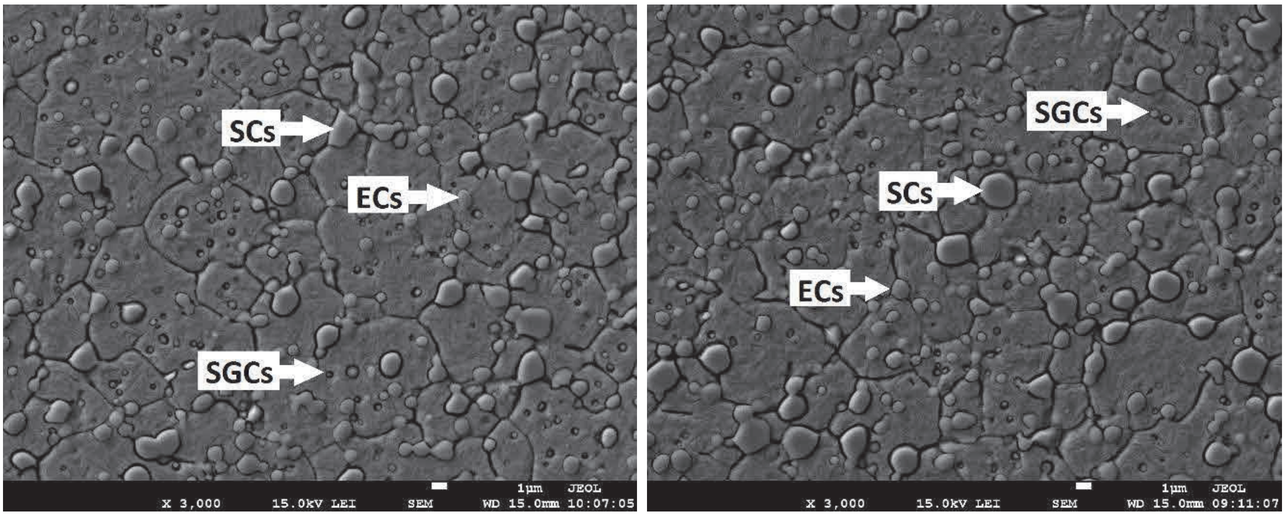

Figure 3 Microstructure of Vanadis 6 **a)** after SZT at -140 °C for 24 h, **b)** for 48 h

Numver of SGCs shown in **Figure 4**. It is obvious that count increased very rapidly up to 24h soaking time. The mean values of population density of SGCs for no-SZT samples and SZT samples for 4, 10, 17, 24, 36 and 48 h were 48 x 10^3 / mm^2, 176 x 10^3 / mm^2, 179 x 10^3 / mm^2, 198 x 10^3 / mm^2, 209 x 10^3 / mm^2, 193 x 10^3 / mm^2, and 183 x 10^3 / mm^2, respectively. Here it should be noted that values, with respect to the statistical uncertainty at a level of 95 %, considerably overlap for the samples treated for 4 and 10 h. It is shown that the maximum in the count of SGCs was achieved for 24 h soaking time.

The size of SGCs is about 500 nm but more typically around 100 nm. Enhanced count of SGCs was observed already after SZT and reheating to room temperature in the present study. Hence, one can suggest that the reason for the presence of SGCs can differ from what was proposed by Das et al. [9] e.g. they maybe originated as a result of enhanced stress situation of sub-zero processed material and its effort to relax the stresses. It was already discussed and explained recently [12].

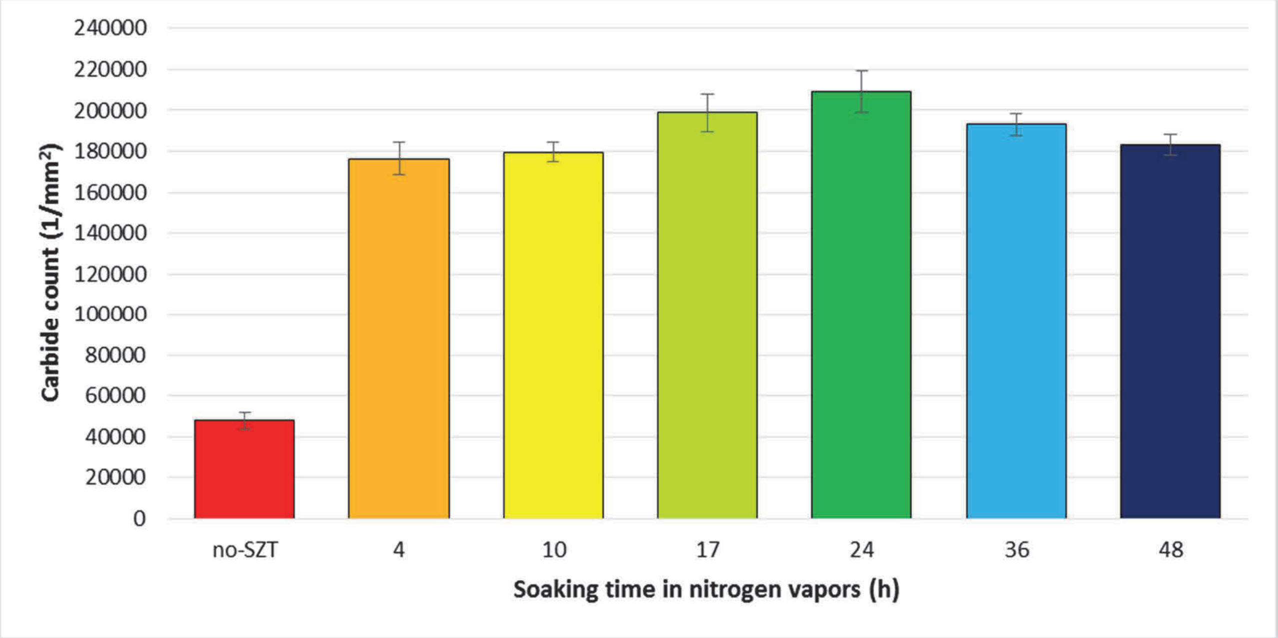

Figure 4 Count of SGCs for samples processed by CHT and SZT, with various soaking times in nitrogen vapors

The bulk hardness of SZT Vanadis 6 tool steel is shown in **Figure 5**. The hardness of CHT steel was 875 ± 16 HV 10. The hardness of the SZT steel soaked in nitrogen vapor for 4, 10, 17, 24, 36 and 48 h were 949 ± 13.5, 949.2 ± 15.6, 933.2 ± 16.8, 925.2 ± 13.3, 940.8 ± 6.4 and 954.6 ± 14 HV 10, respectively. These results infer that the as-quenched bulk hardness of Vanadis 6 steel is improved due sub-zero treatment, whereas the improvement is almost independent on soaking time in nitrogen vapors. Application of SZT at -140°C increases hardness, but after 4 h the effect of SZT on hardness is minimal.

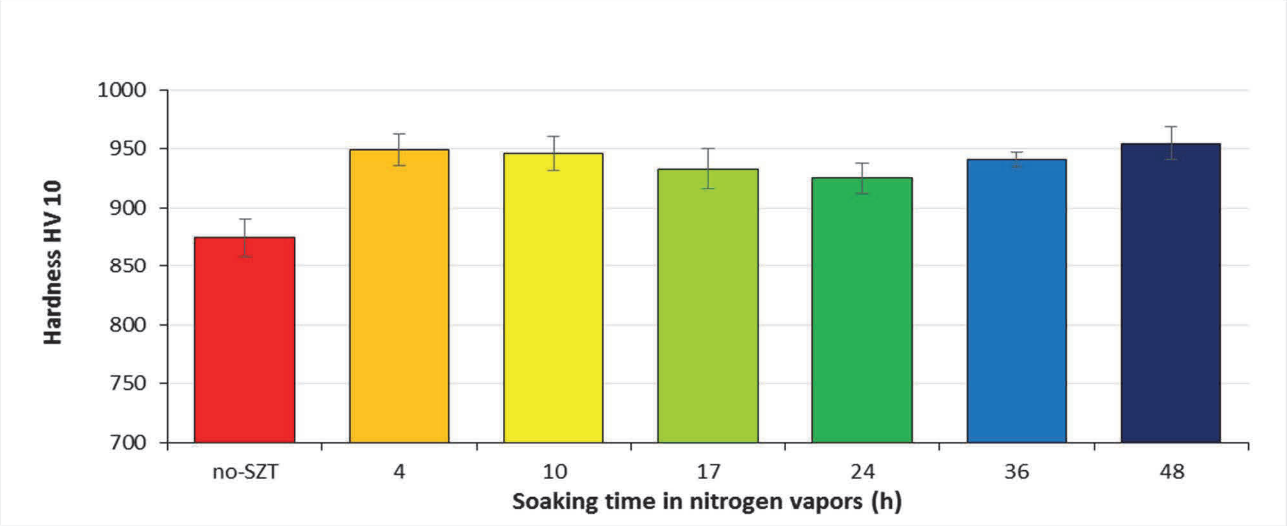

Figure 5 Bulk hardness HV 10 of the Vanadis 6 after SZT with different soaking time in nitrogen vapors

The amount of retained austenite is presented in **Table 1** (vol. %). The amount of retained austenite (γ_R) decrease relatively rapidly with the application SZT. In no-SZT samples γ_R was 20.2 vol. % and for SZT samples with 48 h holding time it was just 3.24 vol. %. The martensitic transformation is time-depend when material is exposed to very low temperature. The metallurgical aspects include reducing the amount of retained austenite so wear resistant and dimensional stability are better as compared to conventionally heat treated material.

Table 1 Amount of retained austenite (vol. %) in the samples SZT for various soaking time (h).

SZT (h)	No-SZT	4	10	17	24	36	48
γ_R -amount (vol. %)	20.2	3.94	4.64	4.25	4.16	3.6	3.24

These results are consistent with results in other publications [9, 10]. For instance, Das et al. [10] have reported that the AISI D2 steel is almost free of γ_R after SZT performed at -196 °C for 36 h. Tyshchenko et al. [11] have established that Cr-V ledeburitic steel X220 CrMoV 13 - 4 contained around 4 % of retained austenite after long-term SZT at -196 °C. It is relevant to note that the γ_R always exist in their microstructure after quenching and before tempering and the martensitic transformation is never completed in high-carbon ledeburitic steels.

3. CONCLUSIONS

The obtained experimental results lead to following major conclusions:

1) The SZT Vanadis 6 tool steel is composed of the matrix (the martensite and the retained austenite), ECs, SCs and SGCs (with a size up to 500 nm, mostly around 100 nm).
2) The count of small globular carbides is several times enhanced due to the SZT, with a maximum after SZT realized for soaking time of 24h.
3) The application of SZT reduces the amount of retained austenite in Vanadis 6 tool steel.
4) The as-quenched hardness of the Vanadis 6 tool steel manifests a marginal increase after SZT.

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