

INTERACTION OF SUB-ZERO TREATED Cr-V LEDEBURITIC STEEL WITH ALUMINA, 100Cr6 STEEL AND BRONZE IN DRY SLIDING

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Resume

The interaction of the Vanadis 6 steel, processed without/with an application of sub-zero treatment, with alumina (hard counterface), 100Cr6 ball bearing steel (counterface of an intermediate hardness) and CuSn6 (soft counterface) has been examined. Obtained results infer that the wear performance against alumina is the best for no sub-zero treated material quenched from higher austenitizing temperature (highest hardness). In dry sliding against 100 Cr6 ball bearing steel, the best wear resistance has been achieved for the material after SZT at -196 °C/10 h. The interaction of Vanadis 6 steel with CuSn6 results in a considerable counterpart material transfer to the samples of Vanadis 6-steel whereas the extent of the transfer is rather independent on both the austenitizing temperature and the SZT parameters, within the range of parameters used for the investigations.

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1. Introduction

The fact that sub-zero treatment (SZT) of chromium (Cr) and chromium-vanadium (Cr-V) ledeburitic steels can bring some benefits to their wear resistance has been known for at least last four decades [1]. On the other hand, the attempts to clarify the metallurgical background being responsible for these benefits are much “younger”, e.g. they started in the middle of 1990s [2]. The latest investigations arrived to claims that the reasons for the improvements in the wear resistance are based in the sub-structure of the martensite formed at cryo-temperatures [3, 4] and in the alterations in microstructural changes taking place on subsequent tempering of the sub-zero formed martensite [4 - 6].

It should be noted that obtained results on the improvements in the wear resistance of Cr- and Cr-V ledeburitic tool steels are very different.

Already Stratton [7] has reported a summary of practical results referring that the wear behaviour was improved for the tools made of AISI D2 steel by 817 % while only an improvement by 131% was achieved for those made of the CPM-10V steel. Also, the experimental (laboratory) results obtained on the same material, but by various researchers, are mutually incomparable in many cases since the investigators have used different conditions for the testing. Das et al [8], for instance, have used a rotating disc made from WC-coated steel (hardness of 1750 HV) as a counterpart, different loading (from the range 78.48 – 137.34 N), sliding speed 2 m/s, rel. humidity of 60% and an ambient temperature for the measurements while Pellizzari and Molinari [9] have used counterparts made of X210Cr12 steel (61.3 HRC), load of 150 N, sliding speed of 0.21 m/s for the measurements of the wear resistance of AISI D2-steel.

2. Experimental procedure

The experimental material was PM ledeburitic 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. The samples were plates (10 x 18 x 70 mm), polished up to the mirror finish ($R_a = 0.04 \mu\text{m}$) after the heat treatment. Heat treatment of the material comprised the austenitizing (temperatures 1000, 1025, 1050 or 1075 °C) in a vacuum furnace, hold at the final austenitizing temperature (T_A) for 30 min. and nitrogen gas quenching (5 bar pressure). Immediately after quenching, the specimens were subjected to SZT realized at the following regimes: -90 °C/4 h, -196 °C/4 h and -196 °C/10 h. One set of the samples was processed without SZT for the comparison of obtained results. The tempering regime was kept constant, e.g. it has been performed in two cycles, each at a temperature of 530 °C for 2h, whereas the material was cooled down to an ambient temperatures after each tempering.

The hardness has been measured by standard Rockwell C method. For each combination of heat treatment parameters, seven measurements were done and the mean values were calculated. Dry sliding wear tests have been performed with a CSM pin-on-disc tribometer. Balls with a diameter of 6 mm made from sintered alumina (Al_2O_3), 100 Cr6 steel (735 HV 10) and CuSn6-bronze (149 HV 10), respectively, have been used as counterfaces. The normal load applied was 5 N. The tests have been carried out at a linear velocity of 0.064 m/s, to the total sliding distance of 100 m, at an ambient temperature and in laboratory air, at a relative humidity of 40 – 50 %. To obtain sufficient statistical approach, the experiments have been carried out on 3 samples for each combination of heat treatment parameters.

For the characterization of the wear behaviour against alumina and 100Cr6 steel, respectively, the width of wear scars has been measured on light microscope ZEISS NEOPHOT 32 at a magnification of 50x. Ten

measurements have been made on each scare and the mean value, which was used for further assessment, was then calculated. The volume loss and the wear rate of samples were calculated from the width of the wear tracks using methods described in the ASTM - standard [10]. For the investigation of interaction with CuSn6-bronze, no wear rate has been determined because the bronze is too soft to be capable to make any substantial wear on tool steel.

The area portion of transferred counterface material (for the testing against 100Cr6 steel) has been determined using a light microscope NEOPHOT 32 at a standard magnification of 50 x. For the assessment, an image-analysis LUCIA - software has been used. Image analysis has been carried out on digitally acquired micrographs whereas twenty micrographs have been used for any heat treatment regime. The mean value was then calculated from acquired data. For the determination of transferred CuSn6 – bronze, the same method has been used but the images were acquired on the SEM.

Detail characterization of wear scars has been done using a scanning electron microscope JEOL 7600-F using three regimes – detection of secondary electrons (SE), backscattered electrons (BE) and acquisition of EDS-maps. The acceleration voltage for the images acquisition was 20 kV for both the SE- and BE-regimes but it was lowered to 5 kV for the EDS-analysis and mapping.

3. Results

Figure 1 shows the as-tempered hardness of the material as a function of both the T_A and the SZT. Tempering at a temperature of 530 °C, normally used in order to achieve a peak of secondary hardening, led to less or more significant hardness decrease compared to that of no SZT material. This can be attributed to the acceleration of phase transformations on tempering due to SZT, which was described and

discussed recently [3 - 6] and verified in our experiments, also.

Figure 2 shows the effects of both the austenitizing temperature and the sub-zero treatment on the wear resistance when sintered alumina has been used as a counterpart. At lower austenitizing temperature, the best wear performance had the material SZT at $-196\text{ }^{\circ}\text{C}/10\text{ h}$ and $-196\text{ }^{\circ}\text{C}/4\text{ h}$. On the other hand, the wear rate was the lowest for no-SZT steel when higher austenitizing temperature has been used for the material processing and it increased with decreasing sub-zero treatment temperature.

Figure 3 shows an example of worn surface formed by the sliding of Al_2O_3 counterpart against the Vanadis 6 steel processed at austenitizing temperature of $1075\text{ }^{\circ}\text{C}$, sub-zero treated at $-196\text{ }^{\circ}\text{C}/4\text{ h}$ and tempered. Irrespective of the type of heat treatment used for the processing, the worn surfaces exhibit enhanced roughness compared to no-worn material. However, the worn surfaces are shiny and metallic in nature in dominant area portion, Fig. 3a. One exception, however, can be easily found inside the wear scar, Fig. 3b. The material undergoes extensive plastic deformation on the contact surface and, as a result, an oxidation proceeded easily. This has been confirmed by EDX-microanalyses.

To explain the wear behaviour against the alumina it should be noted that the main factor influencing the wear resistance is the hardness of the material when higher austenitizing temperature was applied, e.g. the harder steel the better is the wear resistance. This is why no-SZT steel had the best wear performance and the wear rate increased with deeper cooling and/or with prolonged hold at the cryo-temperature. Also, these findings are consistent with the observations [11]. According to the claims presented in [11], the role of carbides is negligible in the wear behaviour when the hardness of counterpart is higher than that of the carbides. The hardness of alumina and carbides is comparable and, in addition, the volume fraction

of harder MC phase is relatively low after applied heat treatment [12]. At lower austenitizing temperatures, as indicated by obtained results, other factors can also play role in the wear behaviour. These factors were firstly mentioned by Collins [2] and then described by Das et al. [6] and they are represented mainly by the microstructural changes, which comprise greater amount of small carbides embedded in metallic matrix in SZT-material. Hence, the wear performance of SZT steel was found to be better than that of no-SZT material.

The wear rate of the Vanadis 6 steel after sliding against the 100Cr6 steel, as a function of the T_A and the parameters of SZT is depicted in Fig. 4. The highest wear was recorded on the material without SZT while the lowest one was found on the material sub-zero treated at $-196\text{ }^{\circ}\text{C}$ for 10 h. In addition, the wear rate becomes lower with an increase of T_A , as a logical consequence of higher hardness of the material processed at higher austenitizing temperature.

The effect of SZT on the wear resistance should be evaluated based upon following consideration: Sub-zero treated Vanadis 6 steel has lower hardness than the conventionally treated one, after the tempering regime used for the experiments. A simple assumption in this case is that the wear resistance of SZT Vanadis 6 steel should be worsened, also. But, the current investigations infer an adverse tendency and this is in line with other observations, although obtained on different material (AISI D2-steel) and under different testing conditions [8]. The explanation is based on the fact that sub-zero treated AISI D2 - steel exhibits an increased population density of small globular carbide precipitates [5, 6, 9]. Also, our latest investigations fixed an enhanced volume fraction of small carbides in SZT Vanadis 6 steel, with a size less than 100 nm for the material austenitized at $1000\text{ }^{\circ}\text{C}$ and less than 300 nm for that quenched from $1075\text{ }^{\circ}\text{C}$, respectively – see

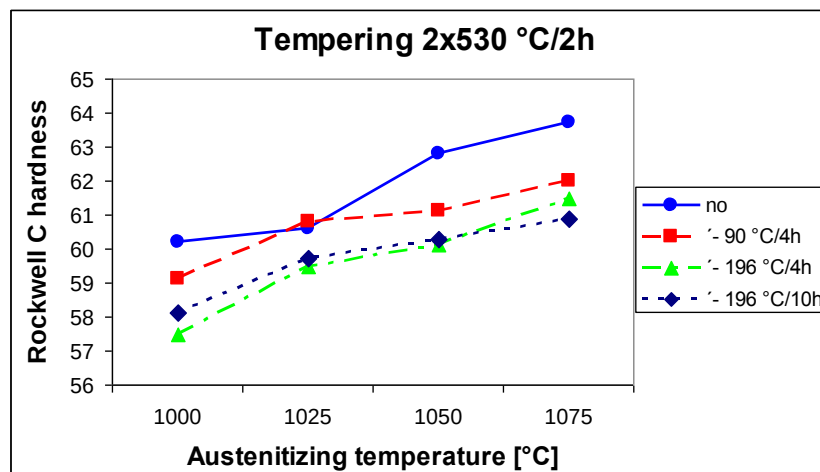


Fig. 1. As-tempered hardness of the steel as a function of austenitizing temperature and parameters of SZT. (full colour version available online)

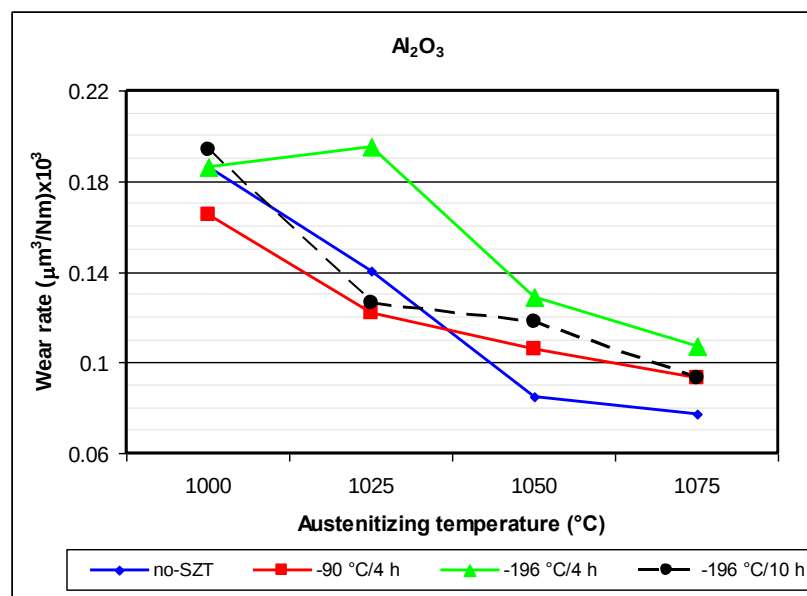


Fig. 2. Wear rate of the specimens made from Vanadis 6 steel and processed with different regimes, alumina was used as a counterpart. (full colour version available online)

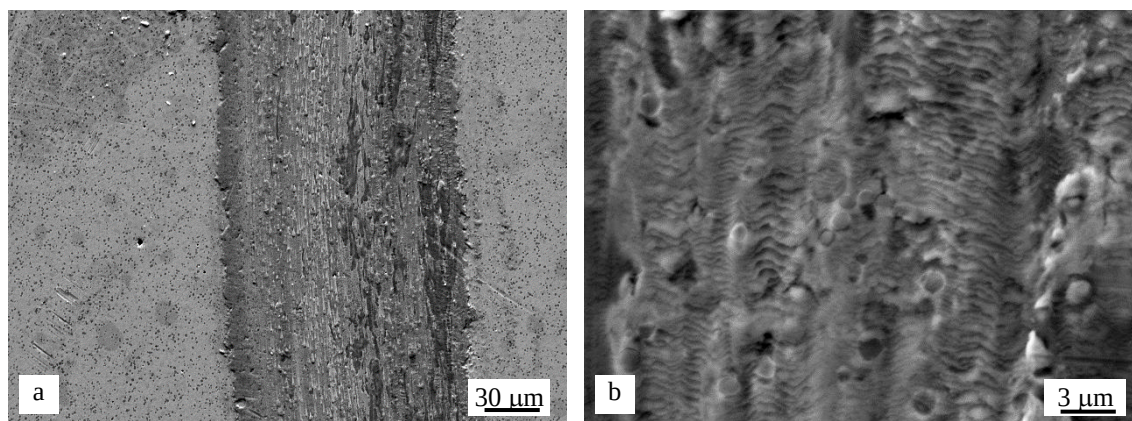


Fig. 3. SEM micrographs showing worn surface of Vanadis 6 steel processed from the austenitizing temperature of 1075 °C and SZT -196 °C/4 h. a – secondary electron overview image, b – detail (BE).

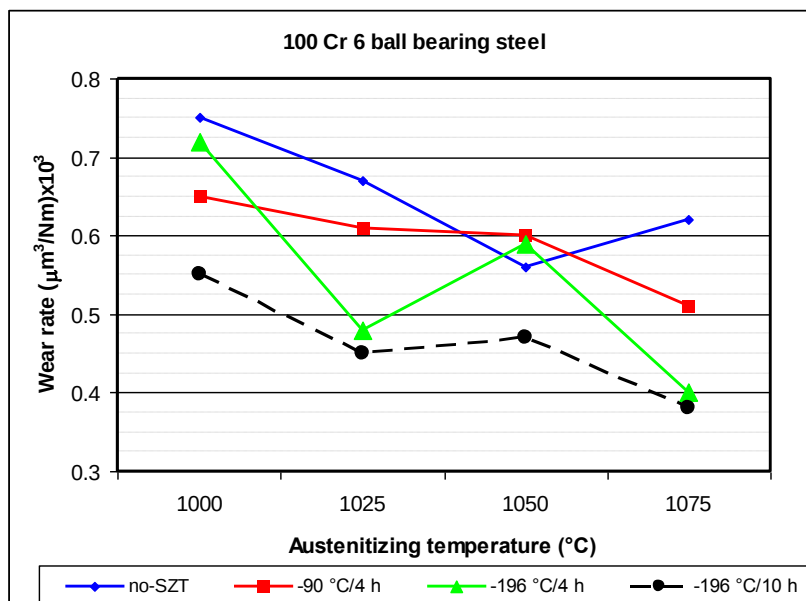


Fig. 4. Wear rate of the specimens made from Vanadis 6 steel and processed with different regimes, 100 Cr6 steel was used as a counterpart.
(full colour version available online)

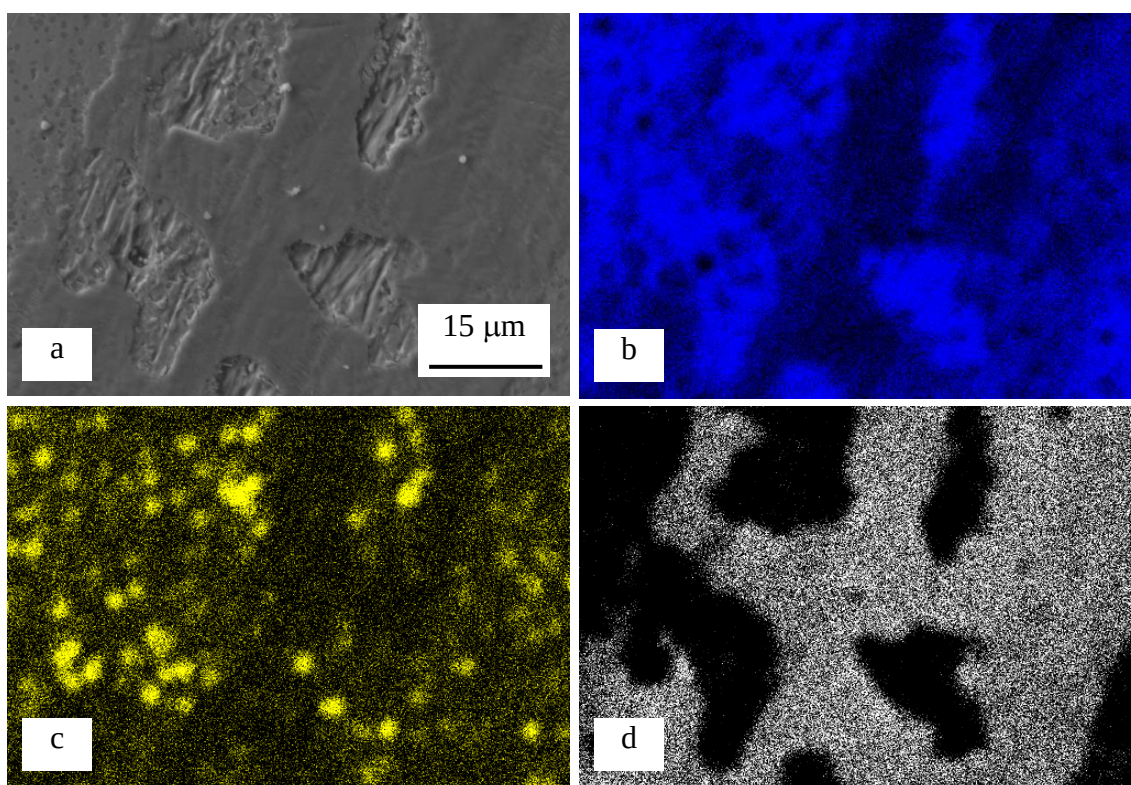


Fig. 5. Detail SEM micrograph of wear track formed on the Vanadis 6 steel austenitized at 1025 $^{\circ}\text{C}$ and SZT at -90 $^{\circ}\text{C}/4$ h and corresponding EDX-maps, a – image, b- iron, c – chromium, d – oxygen.
(full colour version available online)

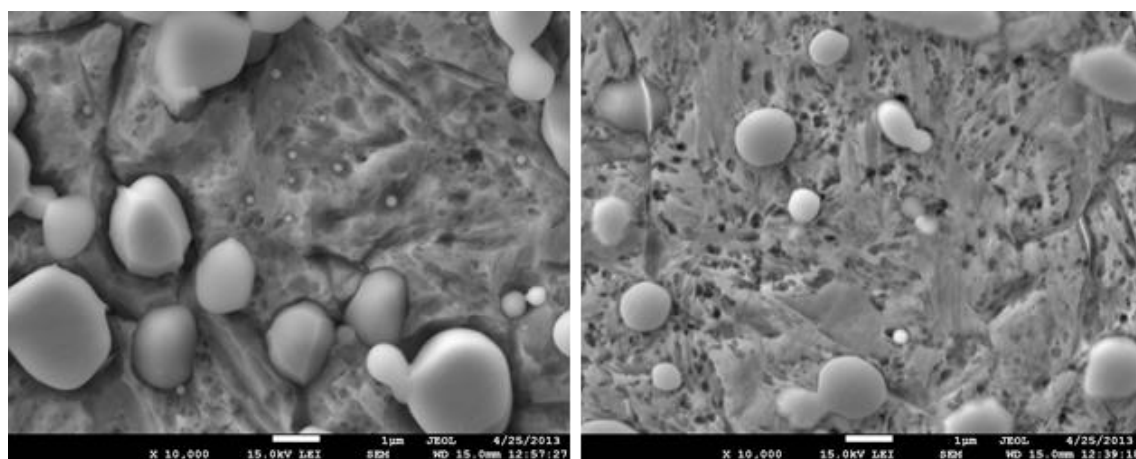


Fig. 6. SEM micrographs showing an example of microstructure of PM ledeburitic steel Vanadis 6 – left - as-quenched from 1000 °C, right – as-quenched from 1000 °C and SZT at -196 °C/4 h.

example in Fig. 6. This gives a natural explanation of better wear performance of sub-zero treated steel. In the evaluation of the wear resistance when tested against 100Cr6, the counterpart's material transfer (Figs. 5, 7) should be taken in the consideration, although our preliminary results indicate that the adhesion of 100Cr6 steel on the samples does not depend on the heat treatment route used prior to the material processing.

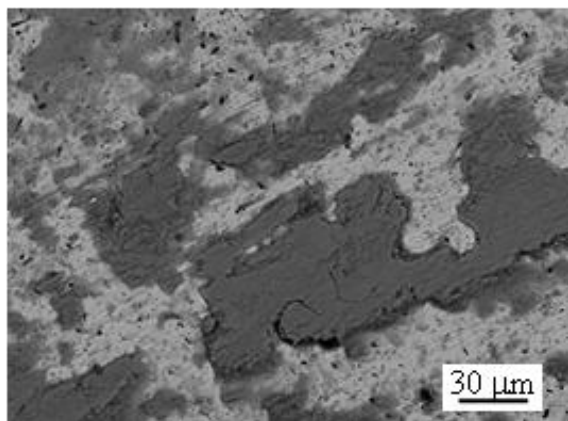


Fig. 7. Detail SEM micrograph showing the adhesive wear of Vanadis 6 steel austenitized at 1025 °C and SZT at -90 °C/4 h.

The evaluation of interaction between the Vanadis 6 steel and the counterface made of CuSn6 is influenced by low hardness of the counterface material. Hence, the method described in [10] can not be used for the determination of the phenomena that occur in dry sliding of CuSn6 against the Vanadis 6 steel. First of all it should be noted that dry

sliding of CuSn6-pin does not result in a weight (or volume) loss of the steel samples but in an extensive adhesion of counterface material on the steel samples only, Fig. 8. This is due to low shear strength of the bronze. The dependence of the extent of the counterface material adhesion on the heat treatment route can not be described unambiguously, Fig. 9. It is known that neither the matrix hardness nor the hardness of carbides are the decisive parameters influencing the adhesive wear, but, the distribution of the carbides (and their inter-particle spacing) plays the dominant role in the adhesive transfer of counterparts material [13].

Here, several effects have to be taken into the consideration:

The volume fraction of eutectic carbides is constant, irrespective to the heat treatment regime used.

The volume fraction of secondary carbides decreases with increasing austenitizing temperature, e.g. one can assume a negative impact of increasing temperature of austenitizing on adhesive material transfer. However, no increase of surface portion of adhered material with elevated austenitizing temperature is clearly evident.

The volume fraction of small globular carbides (precipitates) increases with an application of SZT, see also Fig. 6. However, no positive effect of SZT on adhesive material transfer is visible in the Fig. 9. Here, it is worth

noticing that the duration of SZT of 4 or 10 h is not enough to reach the maximum in improvements of the wear resistance and other properties. In relevant literature, longer soaking

times in liquid nitrogen are recommended for similar materials [8]. Hence, the experiments with prolonged sub-zero treatment are planned to be realized in the nearest future.

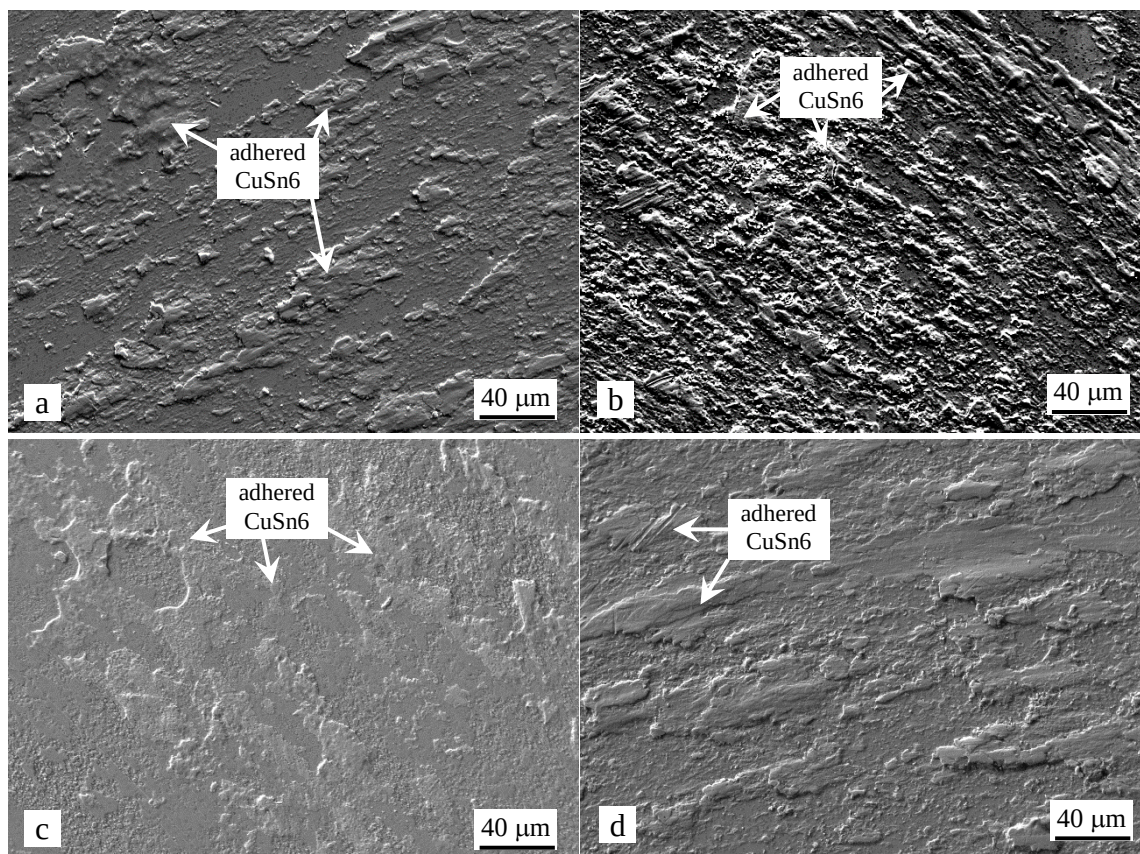


Fig. 8. Plan view SEM micrograph showing the wear scars of the Vanadis 6 steel: a – austenitized at 1000 °C and SZT -196 °C/4 h, b - austenitized at 1075 °C and SZT -196 °C/4 h, c - austenitized at 1000 °C and SZT -196 °C/10 h, d - austenitized at 1075 °C and SZT -196 °C/10 h.

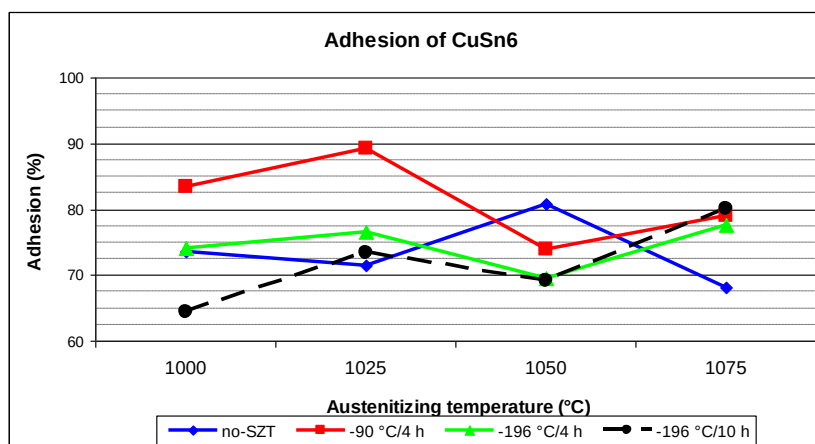


Fig. 9. The portion of the surface inside the wear tracks covered with CuSn6, as a function of the austenitizing temperature and sub-zero treatment. (full colour version available online)

4. Conclusions

The experiments with dry sliding of various materials against no-SZT- and SZT-Vanadis 6 steel give the following conclusions:

In dry sliding against alumina, the wear performance of Vanadis 6 steel is the best for no-SZT material quenched from higher austenitizing temperature while the wear behaviour of the material quenched from lower austenitizing temperature was found to be rather better for SZT steel.

In dry sliding against 100 Cr6 ball bearing steel, the best wear resistance has been achieved for the material after SZT at -196 °C/10 h.

The interaction of Vanadis 6 steel with CuSn6 can not be evaluated using standardized methods due to very low hardness and low shear strength of bronze. This results in a considerable counterpart material transfer and it's adhesion onto the Vanadis 6. The extent of the adhesion seems to be independent on both the austenitizing temperature and the SZT parameters, within the range of parameters used for the investigations.

When compared the wear response of the Vanadis 6 against all three types of counterparts one can suggest that the hardness of the steel is the most important parameters influencing the wear in the case of the sliding against alumina (hard material). In the sliding with 100Cr6 steel (comparable hardness to the Vanadis 6) it is the material status achieved due to SZT which influences the wear response of the material. For the dry sliding against bronze it is impossible to derive the wear performance of the steel based on obtained results.

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