EFFECT OF COLD TREATMENT OR DEEP CRYOGENIC TREATMENT ON SELECTED PROPERTIES OF CASE-CARBURIZED 18CrNiMo7-6 STEEL[1]

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Abstract

A comparative study on the influence of cold treatment (CT) and deep cryogenic treatment (DCT) on the properties of case-carburized steel was made. Specimens made of 18CrNiMo7-6 steel underwent carburizing with two different surface carbon concentrations. After thermo-chemical treatment samples were subjected to cold treatment, performed at -80°C, or deep cryogenic treatment, at -180°C, and tempered. Basic material properties, such as surface hardness, cross-section hardness profile, value and state of internal stress and retained austenite content, were measured and evaluated. Charpy u-notch impact test was employed to determine steel toughness. The effect of CT and DCT on the wear resistance of specimens was assessed by means of three rollers–cone method. Microstructures of specimens were analyzed by means of light microscopy. Cold treatment and deep cryogenic treatment allowed to continue martensitic transformation, however a value of decrease of residual austenite content was similar regardless of the employed temperature of treatment. Austenite transformation was accompanied by an increase of hardness, measured at surface or cross-sections of specimens. Reduction of wear of specimens was also observed, but more beneficial effect was achieved for the CT. Sub-zero treatment processes, irrespective of the applied temperature, caused the decrease in impact strength of steel. Measurements of residual stresses showed the occurrence of compressive stresses in all specimens. After DCT and tempering diminishing of value of stress was observed, probably due to effect of precipitation of finely dispersed carbides.

Key words: Cold treatment; Deep cryogenic treatment; Carburizing.

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1 INTRODUCTION

The joint usage of deep cryogenic treatment is novelty in so far conducted technological processes. Despite the fact that carburizing (in its current form) is known from tens of years, there are still no researches made on the subject of combining this technology with deep cryogenic treatment.

There are a couple of issues, connected with the effect, which sub-zero treatment has on steel. One of them is the continuation of martensitic transformation at sub-zero temperature for steels, in which the martensite finish temperature is lower than the ambient temperature. The effect of almost complete martensitic transformation at the temperature of sub-zero treatment has been confirmed by x-ray diffraction measurements.[1]

Another interesting issue is precipitation of finely dispersed carbides – the hardening phases, in steel microstructure.[1,2] The precipitation of these carbides (described as η carbides)[2] is accompanied by lower rate of internal stresses, which has its own effect on other properties of steel – it lowers the tendency to form micro-cracks and, in turn, causes higher wear resistance.[1]

The sub-zero treatment of carburized steel elements is present in literature as treatment used to lower the content of retained austenite in steel microstructure, causing the increase in martensite content, or, as a treatment for improving the work characteristics of carburized elements, not connected with retained austenite. It may be used, for example, as countermeasure in a situation, when the element was carburized with too much carbon in layer, which would create too much retained austenite after hardening.[3]

Thanks to the increased carbon content in the layer through carburizing, and through causing the transformation of retained austenite into martensite, through sub-zero treatment it is possible to increase hardness, wear resistance, acquiring better dimensional stability and lower susceptibility for crack forming during the grinding process.[4] In the case of elements which require higher abrasive wear resistance, it is advised to acquire higher value of carbon content in the layer, quenching from lower range of hardening temperature, which will allow to acquire of hard (but brittle) carbides close to steel's surface. In this case the steel is usually subjected to another heating and quenching processes, which purpose is to spheroidize carbides and create wear resistant layer, which has the microstructure of martensite matrix with iron carbide precipitations.[5]

The transformation effect from austenite to martensite and the precipitation of finely dispersed and uniformly distributed carbides, invisible in this form of steel which was treated conventionally, is presented in paper.[6] In this paper you can find information about significant influence of sub-zero treatment on tribological parameters of the carburized layer. Paper[7] shows the increase of wear resistance of 815M17 (17NiCrMo6-4) steel over 350% in proportion with steel treated traditionally (carburizing, hardening and tempering). The increase of wear resistance may be accompanied by small decrease of tensile strength,[8] however, taking into consideration the advantages which come from increased tribological properties, this decrease, in the case of most typical applications of carburized steel is acceptable. Also, the authors stated that, sub-zero treatment processes (both at -80°C temperature, as at -196°C) considerably improve dimensional stability of carburized steel.[9]

In paper,[3] in which the influence of different sub-zero treatments on the microstructure and residual stresses of carburized steel was researched, it was stated, that the optimal process is sub-zero treatment at -120°C. The authors suspect that the highest degree of compressive stresses is adequate for elements, for which fatigue wear is typical damage source. Paper[10] states, that the sub-zero treatment conducted at temperature 88K after carburizing, hardening and tempering of 18NiCrMo5 steel, caused the increase of fatigue wear for about 25%. Authors of abovementioned paper gave attention to decreased scatter of results acquired for steel after sub-zero treatment and the small increase of hardness after such treatment. The increase in hardness was explained as the possibility of occurrence of segregation of carbon particles and alloy elements, which takes place during the last stage of sub-zero treatment, heating up to ambient temperature. Similarly explained was the increase of wear resistance of carburized, hardened and tempered steel used for gear wheels in work.[11] The topic of fatigue strength was also taken up by the authors of paper.[12] In that paper, it was shown that cold treatment at temperature -80°C caused the increase of fatigue strength of carburized En353 steel specimens for 71% in proportion to strength of specimens after conventional treatment (without sub-zero treatment). Whereas fatigue strength of specimens sub-zero treated at temperature -196°C was lower than for the specimens without sub-zero treatment for about 26%. After the fractographic research, the authors proved, that from the side of fatigue strength of steel, the combination of microstructure consisting of higher content of austenite and fine carbides, acquired after cold treatment of carburized steel is more favorable than microstructure with lower quantity of austenite and even finer carbides, obtained after deep cryogenic treatment.

Also, it is worth to mention the results of tests of deep cryogenic treatment of carburized steel at temperature -269°C in liquid helium.[6] Authors of this research work have stated however, that the use of so low temperature is not favorable from the side of abrasive wear resistance for steel after carburizing.

The statistics for gear wheel damage prove, that 13,2% of gear wheel's malfunctions is tied with the occurrence of surface layer wear.[7] This process was the cause of lower work time. Lowering of gear wheel surface layer's wear can significantly improve endurance and reliability of gear transmissions. The acquiring of optimal parameters of sub-zero treated carburized layers demands the performance of complex studies in the field of materials properties (for example in the field of microstructure, which has to take into consideration the presence of retained austenite and carbide precipitations, intersection stress pattern, fatigue resistance and impact strength). These qualities have substantial influence on the operational qualities of steel. The exact identification and diagnosis of the effects which accompany sub-zero treatment of carburized layers and the knowledge of steel properties after such thermal and thermo-chemical treatment can allow to formulate the technological guidelines for usage of this treatment in production of gear wheels.

The purpose of this paper is to diagnose this issue in reference to the 18CrNiMo7-6 steel, which is one of the most often used steels for the heavy loaded carburized steel machine parts. The correct understanding of the treatment effects (which were mentioned above), requires the conduction of the detailed research, which will make it possible to identify the phenomena and their mechanisms, which occur during the sub-zero treatment of the carburized layers. Such knowledge may be of great importance and be a valuable part of the thermal and thermo-chemical science. For the practical knowledge, however, it is important to determine the possibilities for use of the carburized and sub-zero treated steel as material for gear wheels.

2 PURPOSE AND SCOPE OF THE PAPER

The purpose of this paper was to check the influence of sub-zero treatment, both as its short term variant (cold treatment) and deep, long term variant (deep cryogenic treatment), on the chosen characteristics of carburized layer through theoretical studies and next, by the experimental research. During the research, authors tried to determine the process influence on the following factors: microstructure, hardness, internal stresses, content of retained austenite, impact resistance (with the use of Charpy pendulum machine) and wear resistance of the carburized 18CrNiMo7-6 steel.

The technological part of paper consists largely of the series of gas carburizing processes (performed at 920°C, oil quenching from 880°C) in the carburizing atmosphere, which is produced from liquid organic compounds and setting the carburizing parameters from the demanded surface layer content of carbon - point of view. The carburizing process was performed to achieve:

- Near-surface carbon concentration about 0,8 % (for elements in danger of fatigue bending and dynamic loads),
- Near-surface carbon concentration about 1,1 % (for elements which require high contact fatigue strength and wear resistance).

During the thermo-chemical processes conducted in the GOAT-950 atmospheric soaking pit (Fig. 1a), with the use of mixtures consisting of different proportions of methanol and toluene, measurements of carbon potential were made, with the thin foil method. It was finally stated, that the assumed carbon potential of the atmosphere, and therefore the acquired concentration of carbon in the specimens surface layer will correspond with the following compositions of the liquid organic compounds mixtures:

- C_P = 0,8 %C – methanol 98,5% + toluene 1,5%;
- C_P = 1,1 %C – methanol 97% + toluene 3,0%.

Figure 1. Atmospheric pit furnace GOAT-950 (a) and CRYO-TEMPER processor (b).

The carburized and oil quenched specimens were subjected to sub-zero treatment in the CRYO-TEMPER processor (Fig. 1b) at temperatures -80°C and -180°C, with the treatment time 2 h and 32 h respectively. Specimens treated in this manner were next tempered at temperature 180°C (Fig. 2). Cold treatment was conducted directly after quenching, and the cryogenic treatment was conducted after

3 hours since the quenching operation. Cooling down and heating up of specimens were conducted in such way, to avoid the possibility of crack formations in treated specimens, caused by thermal shock microstructural transformations. Heating up was conducted in the chamber of processor, in nitrogen atmosphere, to avoid the condensation of steam on the specimens.

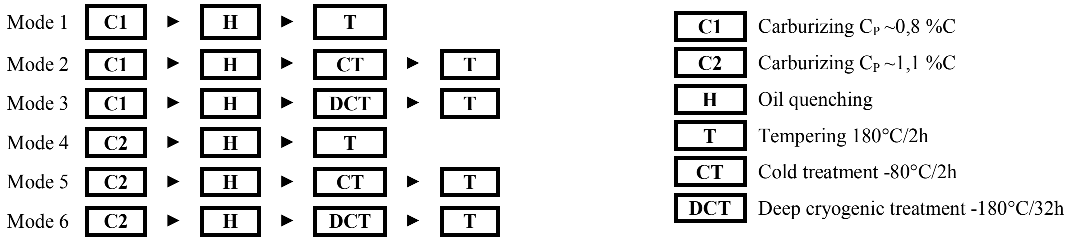

Figure 2. Modes of combinations of heat and thermo-chemical treatment.

After heat and thermo-chemical treatments, the specimens surfaces, which were subjected to tests, were grinded. During the grinding process, there was removed about 0,1 mm of the material's surface layer.

3 RESEARCH METHODS

Laboratory studies consisted of metallographic observations, hardness HV1 and HV10 measurements, determining of hardness profile HV0,1 and HV1 on the surface layer's cross-section, impact strength measurements, content of retained austenite, internal stresses measurements and determining the value of wear.

Surface hardness tests were performed on the ,,CV Instruments" hardness tester, making 5 imprints per sample. The results were statistically elaborated, with 95-% confidence intervals.

The hardness profiles were also determined with the use of ,,CV Instruments" hardness tester on metallographic specimens. The measurements were made with the load of 1 kG, on depth ranging from 0,05 to 1,5 mm.

The impact resistance measurements were carried out with the Charpy method, on specimens with length 55 mm, square cross section with side length 10 mm, with U-shaped notch, 2 mm deep and with the deep notch fillet radius of 1 mm. Test was performed according to research procedure PB/2-10/LB-4 and Polish Standard PN-EN 10045-1:1994 on the Impact hammer from ,,Alpha" company, with the pendulum's initial energy of 150 J.

Observations of the metallographic specimens (etched with 2-% nital) were performed on the metallographic microscope Nikon Eclipse LV150, with x1000 magnification. The wear resistance test was performed with the use of ,,3 rollers-cone" method on the I-47-K-54 machine, according to Polish Standard PN-83/H-04302. Research was made with load of 100 and 400 MPa. As the lubricant, LUX-10 oil was used, with feeding velocity of 30 drops per minute. On the basis of acquired results, the Lorenz diagrams were drawn.

The internal stresses tests were made with the use of x-ray apparatus for stress measurements from the RIGAKU company. The method $\sin^2\psi$ was used, with the Cr lamp. On the RIGAKU apparatus there were also performed measurements of the content of retained austenite.

The fatigue with the rotary bending tests Z_{go} were performed with the use of testing machine from the Schenck company, of the PUN035Z type, with constant distribution of the bending moment along the measured axis. Tests were performed with the load modification frequency f=100 Hz and cycle base N_G=1x10^7.

4 RESULTS AND DISCUSSION

4.1 Metallographic Observations

The metallographic observations made with the use of light microscope showed, that the microstructure of carburized layer consists of the tempered laths of martensite and different quantities of retained austenite, which depend on near-surface carbon concentration and the used sub-zero treatment process (Fig. 3). The carbide precipitations were not observed, which may be caused by insufficient magnifying range (up to x1000).

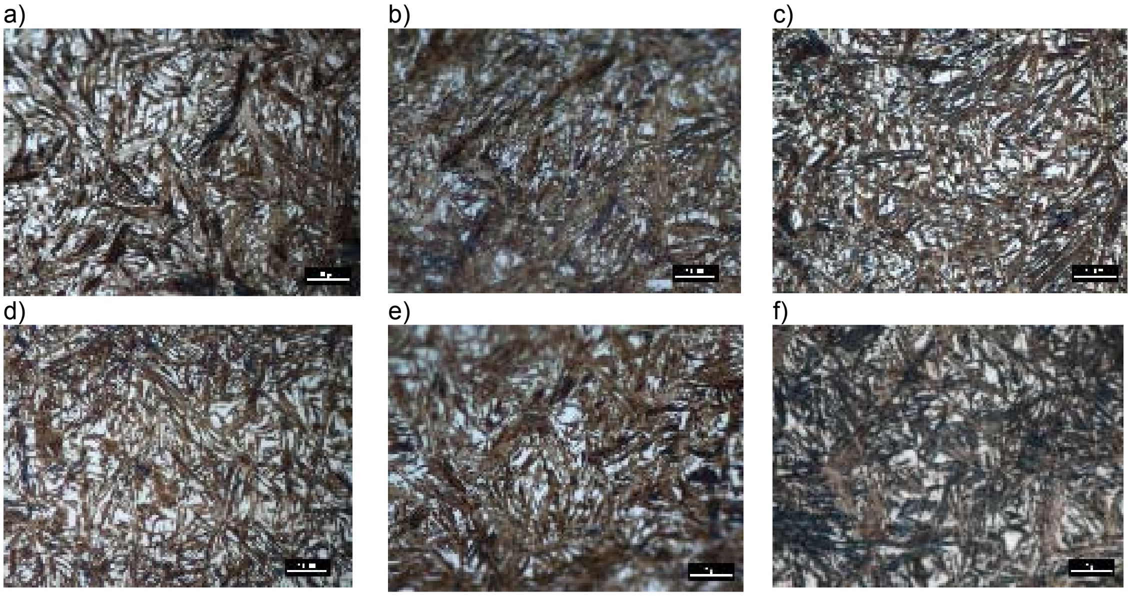

Figure 3. Microstructures of sub-surface zones of specimens heat-treated in modes 1-6 (a-f).

4.2 Surface and Cross-section Hardness

Surface hardness measurements (Fig. 4a) have shown, that after carburizing, the specimens have achieved hardness of about 655 HV1 (with C_P = 0,8 %C) and about 680 HV1 (with C_P = 1,1 %C).

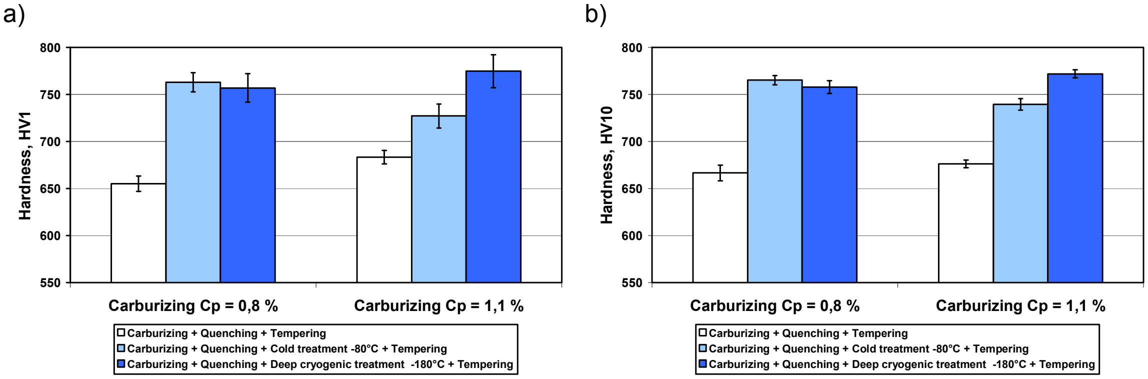

Figure 4. Mean values and 95 % confidence intervals of HV1 (a) and HV10 (b) surface hardness of specimens after various modes of heat and thermo-chemical treatment.

For treatment mode 1, which consisted of carburizing with lower carbon quantity in layer, after processes which were run for both cold treatment variants (modes 2 and 3), there was large increase in surface hardness HV1 (for about. 15%) to value of 755-760HV1. Whereas after carburizing with lower carbon quantity in layer (modes 4, 5 and 6), there were observed growths in hardness after cold treatment at temperature -80°C were slightly lower (increase in hardness up to about 725 HV1), and after deep cryogenic treatment at temperature -180°C slightly higher (increase in hardness up to about 775 HV1).

Surface hardness measurements conducted with load 10 kG have shown analogical tendencies in hardness changes, with slight differences in acquired hardness values (Fig. 4b).

It was stated, that the hardness increase may be tightly connected with: transformation of retained austenite during hardening process into martensite, the precipitation of finely dispersed carbides, which appear as early as during the heating the carburized steel to ambient temperature or during the tempering, which has its confirmation in literature.[6,10]

The results of hardness measurements on the specimen's cross-sections have allowed to evaluate the layer's thickness and determining of the influence, which sub-zero treatment variants have on the steel hardness. Evaluating of the layer's thickness according to hardness criterion 550 HV described in standard EN ISO 2639,[13] the carburized layers have thickness of about 1,2 mm (Fig. 5a,b).

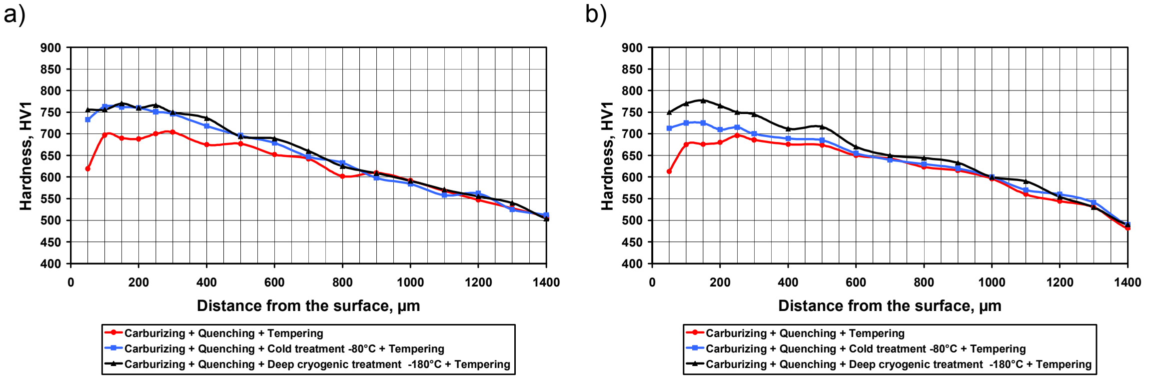

Figure 5. Hardness profiles of specimens after various modes of heat and thermo-chemical treatment: a – carburizing with C_P = 0,8 %C, b – carburizing with C_P = 1,1 %C.

Tendencies for variations of hardness on the intersections of layers after sub-zero treatment processes were consistent with those observed during the surface hardness measurements. In both cases of carburizing, cold treatment at temperature -80°C, and even more deep cryogenic treatment at temperature -180°C, influenced the increase of hardness in the whole layer's cross-section, however, the effect of increased hardness was more sharp for specimens carburized to higher surface concentration of carbon, then quenched and treated cryogenically. Moreover, hardness measurements performed under load of 1 kG indicated the occurrence of lower hardness at layer depth about 50 µm, which could be probably caused by the decarburization, and oxidation during hardening, or the occurrence of grinding burns during the mechanical treatment of the heat and thermo-chemically treated specimens.

4.3 Retained Austenite Content

With consideration of the fact, that the content of retained austenite in microstructure after carburizing and hardening is influenced mostly by the temperature from which the specimens were quenched, it was chosen to acquire high content of retained austenite after hardening. In this way, after carburizing with carbon potential C_P = 0,8 %C and hardening and tempering at temperature 180°C there was acquired about 18 % of retained austenite, while during the carburizing with potential C_P = 1,1 %C and hardening and tempering, the content of austenite was about 26 % (Fig. 6a).

Measurements of the retained austenite content in the near-surface zone have shown, that independently from the used sub-zero treatment temperature, there was achieved similar level of martensitic transformation of the retained austenite, which was left in the microstructure of specimens after carburizing and hardening operations. Independently from the used sub-zero treatment temperature, in the case of steel carburized to the concentration of carbon in layer of 0,8 %, in the near-surface zone it was left about 10-11 % of austenite, while for steel carburized to the concentration 1,1 %C the content of austenite was higher and it was about 14-15,5 %.

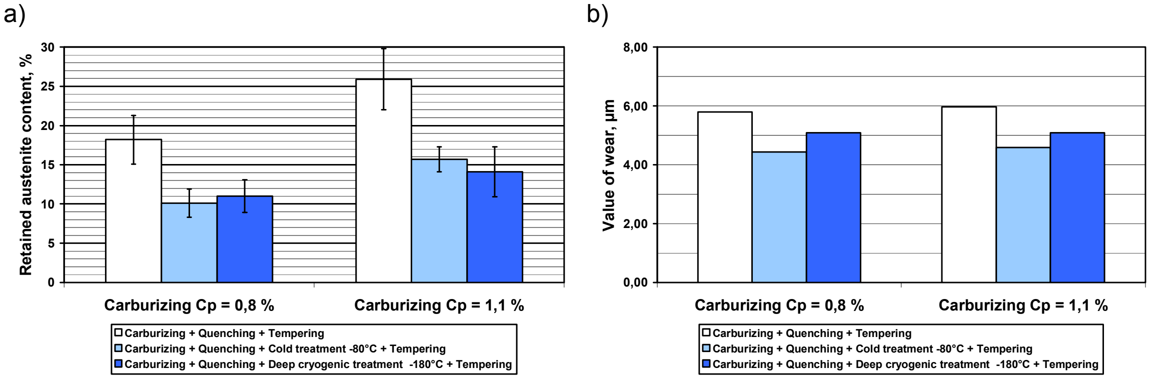

Figure 6. Mean values and standard deviations of residual austenite content in sub-surface zone (a) and mean values of wear rates (b) of specimens after various modes of heat and thermo-chemical treatment.

On the similar quantity level of retained austenite, the following factors may have had influence: delay between hardening and cold treatment and occurring at the same time stabilization of austenite. It was proven, that the 40 minutes delay between these operations may be the cause that only 60% of retained austenite will transform. Not without the influence on the content of retained austenite is nickel, which stabilizes its content.[4]

4.4 Abrasive Wear Test

Elementary load of 400 MPa has proven too high for specimens treated with all technological modes of carburizing and carburizing combined with sub-zero treatment, because the specimens have galled after about 60-80 minutes of testing. On the load of 100 MPa however, the galling did not occur; the comparison of average values of specimen’s wear, acquired after friction time of 100 minutes, is shown in Figure 6b.

Figure 6b shows, that the highest the highest value of wear was observed for samples subjected only to carburizing and hardening, independently from the concentration of carbon in layer. Slightly lower values of wear were observed in the case of specimens subjected to deep cryogenic treatment at temperature -180°C, while the specimens cold treated at temperature -80°C were characterized by the lowest value of wear.

While the lowering of the specimen's wear through increase of the content of martensite, at the cost of retained austenite, during the cold treatment at temperature of -80°C seems to be obvious, the lower influence of deep cryogenic treatment on the wear resistance seems to be unclear, especially considering the results of retained austenite content measurements, and its almost identical content for specimens sub-zero treated in both variants. The incoherence of results acquired in abrasive wear test and the quantity measurements of retained austenite may be explained by transformations occurring during the friction test itself. The stresses caused by load could be the source of martensitic transformation, increasing the specimens wear resistance during the test.

4.5 Impact Strength

The impact strength test have shown, that the sub-zero treatment processes, independently from the used temperature and the carbon concentration in the layer of carburized specimens, cause small loss in the steel impact strength (Fig. 7a). The decrease of impact strength for sub-zero treated specimens may be explained by the higher amount of fragile phase – martensite, in specimens microstructure. This observation is consistent with the results of paper,[14] which was devoted mostly to the problem of steel's 4340 (equivalent of steel 36CrNiMo4 according to EN10083/1-91-A1-96) impact resistance. Noteworthy is the lesser scatter of results, acquired for sub-zero treated specimens, when compared with the results for specimens, which were not sub-zero treated.

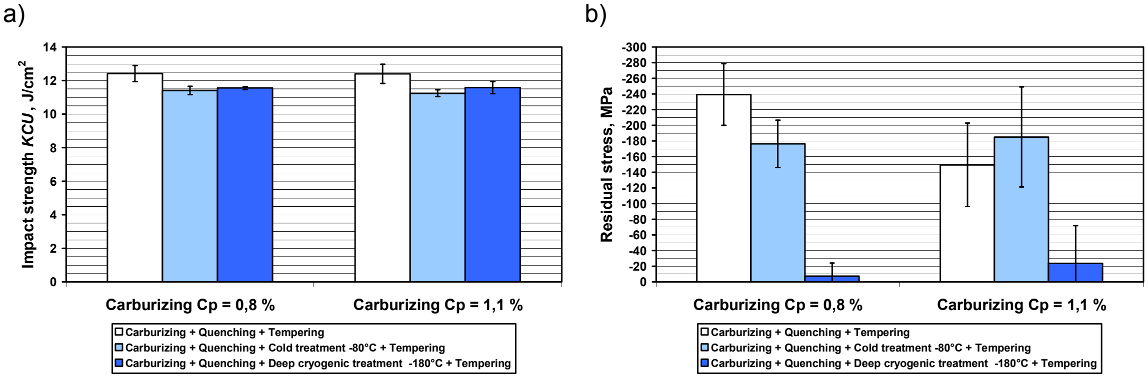

Figure 7. Mean values and 95 % confidence intervals of impact strength (a) and mean values and standard deviations of residual stresses (b) of specimens after various modes of heat and thermo-chemical treatment.

4.6 Residual Stresses

The internal stress tests have shown the presence of compressive stresses in all specimens treated with accordance to individual technological modes (Fig. 7b). The large scatter of results, manifesting itself through the wide range of standard deviations, was caused by the employed research method. Point measurements characterize themselves by the fact, that the acquired stress value is strongly connected with the microstructure, which means the content of martensite, retained austenite and carbides, which is different in every place.

The highest and comparable values of internal stresses were obtained in the case of variant consisting of carburizing, hardening, and tempering, or carburizing, hardening and cold treatment at -80°C temperature, and then tempering. The significant decrease of stresses (down to 10-20 MPa) was obtained in modes which were consisting of deep cryogenic treatment. Such considerable decrease of stress after treatment cycle consisting of: carburizing with different amounts of carbon in layer, hardening, deep cryogenic treatment and tempering, could be probably explained by the forming of fine carbides, which precipitate from the martensite.

4.7 Fatigue Strength

For the fatigue strength, test with the use of rotating bending method, there were chosen specimens from variants consisting of: carburizing with lower surface layer carbon concentration, and following sub-zero treatment (modes 2 and 3). The fatigue strength limit, determined under rotating bending conditions Z_{go} for mode *2* was 1020 MPa, and for mode 3, this value was similar, 1000 MPa (Fig. 8). These results may consist, that similar fatigue strength of steel, after sub-zero treatment processes, had resulted with the similar amount of retained austenite in the specimens microstructure.

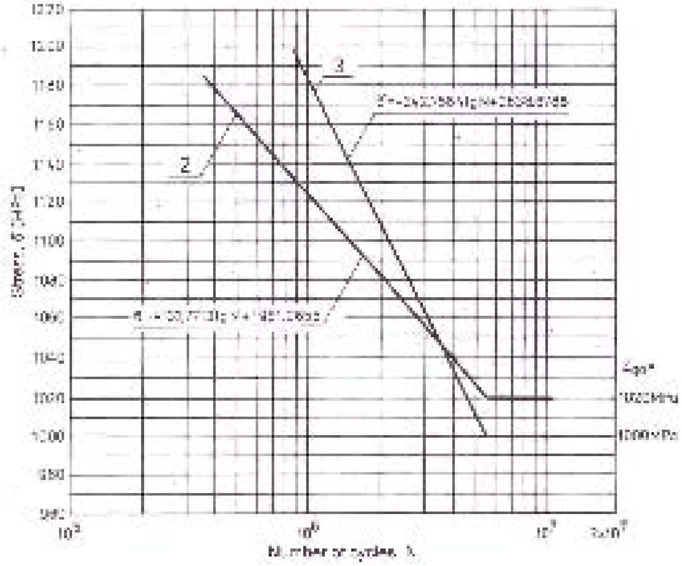

Figure 8. Rotating bending fatigue strength of specimens after modes 2 and 3 of heat and thermo-chemical treatment.

5 SUMMARY

Results achieved during the realization of research, in the scope of microstructure, hardness, content of retained austenite, stress level, impact resistance are mostly consistent with the research results presented in the scientific literature.

Two different surface carbon concentrations, achieved during the carburizing process, according to predictions, have resulted in obtaining different microstructure contents of retained austenite. The sub-zero treatment processes made it possible to decrease the amount of retained austenite, however, the degree of this decrease was similar, independently from the applied sub-zero treatment temperature. The lack of further, nearly complete martensitic transformation, despite the use of treatment temperatures lower then the martensite finish temperature for most of the carburizing steels, may be the result of stabilization of retained austenite. The sub-zero treatment processes were performed directly after quenching (in the case of conventional cold treatment) or after 3 hours (in the case of deep cryogenic treatment). This delay in time, as well as the low cooling rate of specimens down to holding temperature, could be cause of exceeding of the time necessary to avoid the stabilization of austenite. In addition, the large influence on the occurrence of this phenomenon could have the content of nickel in the tested steel, which acts as an austenite stabilizer.[4]

The martensitic transformation of the austenite retained after carburizing and hardening, which occurred thanks to the use of sub-zero treatment processes, was accompanied by the increase in surface hardness. While in the case of steel carburized to lower surface carbon concentration, there were no observed differences in hardness for steel sub-zero treated at different temperatures, and in the case of steel carburized with higher carbon concentration in layer, after cryogenic treatment at temperature -180°C there was significant increase in hardness in proportion to hardness of specimens cold treated at temperature -80°C. This effect, the increase in surface hardness, could be influenced by the finely dispersed carbides, which are formed as early as during the heating up of steel from sub-zero treatment temperature to ambient temperature[10] or during its tempering,[6] which precipitations could be favoured by the higher surface concentration of carbon, and lower cryo-holding temperature.

The decrease in wear values through increase of martensite content, at the cost of retained austenite, during the cold treatment at temperature -80°C seems to be non-debatable, while the lesser influence of the deep cryogenic treatment on wear rate requires further research, especially if we take into consideration the results of measurements of the content of retained austenite and its almost identical quantity after sub-zero treatment at various temperatures. Probably, the incoherence of the results obtained during the abrasive wear test and the measurements of the content of retained austenite may be explained with the transformations during the friction test itself. The stresses caused by load could be the source of martensitic transformation, increasing specimen’s wear resistance at the same time. Such explanation however, is not completely convincing, in respect of the observed effect of stabilization of the retained austenite.

Consistent with expectations, the impact strength tests have shown that the sub-zero treatment processes, independently from process temperature and the concentration of carbon in specimen’s carburized layer, have caused the decrease in impact strength value. Negligible decrease in impact strength of sub-zero treated specimens comes from higher content of fragile phase – the martensite, in the specimens microstructure.

Analyzing the results of residual stress measurements, it can be stated that probably the decrease of internal compressive stresses during the cryogenic treatment and next tempering is caused by the precipitation of fine carbides, which takes place after cryogenic treatment process. This observation is consistent with the results obtained in paper.[15]

The analysis of acquired results made it possible to relate them to results obtained in earlier research, and worldwide documentation.

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