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**DEEP CRYOGENIC TREATMENT OF STEELS**

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*A short review of experimental data about the effect of deep cryogenic treatment, DCT, on the wear of tool steels is presented along with available hypotheses. Using Mössbauer spectroscopy, X-ray diffraction, mechanical spectroscopy and transmission electron microscopy, it is shown that the isothermal martensitic transformation proceeds in the course of DCT. At that, because of softness of the low temperature martensite, plastic deformation occurs during martensitic transformation and the gliding dislocations remove a part of carbon atoms from the solid solution. As the enthalpy of binding between carbon atoms and dislocations exceeds that of  $\epsilon$ -carbide and cementite formation, the precipitation of carbides is delayed during tempering. For this reason, the alpha solid solution contains clusters of carbon and alloying elements, which reserves potential for precipitation during the practical usage of tools increasing thereby the tools life. A new concept of DCT is developed and the correction of its technology is proposed accounting for the temperature range of the isothermal martensitic transformation.*

**Key words:** Tool steel; Heat treatment; Deep cryogenic treatment; Martensitic transformation; Plastic deformation; Tempering; Wear; Mechanical properties.

## 1. INTRODUCTION

Deep cryogenic treatment of tool steels was proposed in the first half of the nineteenth century and more attentively studied since the fifties (see, e.g., [1, 2]). In the nineties, first papers were published concerning a mechanism responsible for its favorable effect on the properties of tool steels, e.g. [3-6]. One distinguishes between the shallow, SCT, and deep, DCT, cryogenic treatments. The first one is used at temperatures between the room temperature, RT, and  $-100\text{ }^{\circ}\text{C}$  and mainly aims to decrease the fraction of the retained austenite. As a result, the hardness increases, whereas toughness decreases. The second one amounts to cooling in the liquid nitrogen and long time holding at this temperature, which increases wear resistance and toughness. However, in this case, the hardness can be even decreased.

A number of obtained results on DCT were analyzed in the review articles [7-10]. At the same time, a definite gap exists between the scientific research and practical applications of this treatment. A reason for that is some scattering of experimental results and the absence of knowledge about phenomena taking place in the as-quenched steels cooled down to cryogenic temperatures. The aim of this paper is to discuss available results and, based on the studies of a mechanism for DCT, propose some optimal technology for this treatment.

## 2. REVIEW OF EXPERIMENTAL RESULTS AND AVAILABLE HYPOTHESES

According to numerous studies, the increase in the wear resistance is the main effect of DCT. A pioneer work was published by Barron [11] who compared the effects of DCT at  $-196\text{ }^{\circ}\text{C}$  and SCT at  $-84\text{ }^{\circ}\text{C}$  on the abrasive wear of martensitic, stainless austenitic and plain carbon steels, as well as the cast iron. The wear resistance of martensitic tool steels was markedly improved due to DCT, whereas the not

significant effect, smaller than 10%, was obtained in austenitic steels. No effect was found for the plain carbon steels and the cast iron. Two essential preconditions for a positive effect of DCT were clearly demonstrated in this study, namely: (i) the thermodynamic instability of steels and (ii) the occurrence of carbon in the solid solution. In fact, these features were ignored by the following researchers. A main peculiarity of subsequent studies was a huge scattering of experimental data. E.g., for the same steel D2, Collins and O'Rourke [4] obtained the improvement in the wear resistance of about 10% in comparison with about 800 %, according to Barron [11].

The D2 type steels were the object for a number of studies on the DCT effect. The decrease of the sliding wear rate up to 700 % due to DCT at  $-180\text{ }^{\circ}\text{C}$  was reported by Meng et al [3]. A remarkable result of these measurements was that, at small sliding speeds of about 0.5 to 1.5 m/s, no difference occurred between the wear resistance in steels subjected to DCT or standard quenching, whereas, with increasing sliding speed, the wear rate has not changed in the first case and significantly increased in the second one. Pellizzari and Molinari [12] studied two cold worked tool steels of D2 type, X155CrMoV12 1 and X110CrMoV8 2, after different heat treatments: (i) quenching at RT and DCT at  $-196\text{ }^{\circ}\text{C}$  followed by double tempering at about  $500\text{ }^{\circ}\text{C}$ , (ii) applying DCT between two tempering stages and (iii) using one tempering stage after DCT. The decrease in the wear rate occurred if DCT was applied before double tempering, and even higher improvement of wear resistance has been achieved in case of single tempering. Oppenkowski [13] has also confirmed that DCT provides a possibility to use only single tempering. Das et al. [14] studied steel AISI D2 after austenitization at  $1020\text{ }^{\circ}\text{C}$ , DCT at  $-196\text{ }^{\circ}\text{C}$  for 12 to 84 h and tempering at  $210\text{ }^{\circ}\text{C}$ . The highest decrease in the sliding wear rate was observed for the holding time of 36 h.

In studies of high speed steels, the interesting finding was reported by Mohan Lal et al. [15] who have shown that the time of holding during DCT is more important than the decrease in its temperature, namely: the sliding wear resistance of steel M2 was higher in case of DCT at  $-110\text{ }^{\circ}\text{C}$  for 24 h in comparison with DCT at  $-180\text{ }^{\circ}\text{C}$  for 6 h. The same steel was tested by Mahmudi et al. [16] after austenitization at  $1200\text{ }^{\circ}\text{C}$ , DCT at  $-196\text{ }^{\circ}\text{C}$  for 90 min and final tempering at  $560\text{ }^{\circ}\text{C}$ . The DCT-caused improvement of the resistance to sliding wear was better by 20 % in comparison with the conventional quenching at RT and by 15 % as compared to CCT at  $-80\text{ }^{\circ}\text{C}$ . Even the higher effect on the abrasive wear of this steel, the improvement of about 43% in comparison with quenching at RT, was obtained by Googte et al. [17] due to DCT at  $-185\text{ }^{\circ}\text{C}$  for 24 h. The high-speed steel T142 (1.27C, 4Cr, 9.5W, 3.2V, 10Co) was studied by Tated et al. [18] after austenitization at  $1230\text{ }^{\circ}\text{C}$ , DCT and triple tempering at  $560\text{ }^{\circ}\text{C}$ . As compared to the non cryo treated tools, the highest decrease in the flank wear, by 42%, is obtained in case of DCT at  $-185\text{ }^{\circ}\text{C}$  for 8 h.

All these results have been mainly obtained within the first decade in the new millennium. Up to the current moment, subsequent studies add new experimental data but did not much contribute to understanding the essence of DCT.

One of the first hypotheses for DCT is based on the enhanced precipitation of  $\varepsilon'(\eta)$ -carbide, as it was shown by Meng et al. [3]. These authors were the first to study the microstructure using transmission electron microscopy and ascribed the obtained DCT effect to fine disperse particles of  $\eta$ -carbide precipitated during tempering instead of the  $\varepsilon$ -carbide. It is relevant to note in this relation that  $\eta$ -carbide was observed earlier by Hirotsu and Nagakura [19] in the high carbon martensite subjected to low temperature tempering without any preliminary DCT or SCT. Later

on, Taylor et al. [20] have shown that this “new carbide” is formed as a result of ordering in the carbon atoms distribution, which transforms the hcp  $\epsilon$  lattice into the orthorhombic  $\eta$  one. They denoted it as  $\epsilon'$ .

In a number of following studies, the precipitation of the  $\eta(\epsilon')$ -carbide was described as a reason for the improvement of wear resistance due to DCT. As stated in [5, 6, 16, 21, 22], these nano-carbide particles are precipitated during subsequent low temperature tempering or even at cryogenic temperatures in the course of DCT or during the following heating to RT [11, 23]. Such a statement was repeated in a number of subsequent publications (e.g., [15, 24, 25]), although at least two experimental facts are at variance with it. First, as mentioned above, the  $\eta(\epsilon')$ -carbide was found to precipitate in the high carbon martensite without any DCT. Second,  $\eta(\epsilon')$  is the intermediate carbide and it is dissolved at temperatures above 200 °C, whereas the positive effect of DCT occurs in steels tempered at significantly higher temperatures, from 400 °C to 500 °C.

The hypothesis of “low temperature conditioning” (e.g., [8, 15, 5, 9, 25]) includes the precipitation of fine carbide particles and describes preconditions for this event in terms of time-dependent decomposition of primary martensite, i.e. some crystallographic and microstructural changes like martensite contraction, decrease of entropy etc. According to this concept, the long time soaking at cryogenic temperatures results in migration of carbon atoms towards the dislocations followed by formation of nanoclusters which, in turn, serve as nucleation sites for nanocarbitides. This assumption is at variance with the absence of any detectable migration of carbon atoms in the Fe-C martensite at temperatures below –100 °C, as it follows, e.g., from Mössbauer spectra which are extremely sensitive to any small change in the atomic distribution [26].

Any hypothesis for DCT should take into account a number of so far inexplicable experimental data about the increase in the fraction of small carbide particles with average diameter below 1  $\mu\text{m}$  (e.g., [16, 17, 27, 28]) and even of 20 to 60 Å [5]. It can be only note in this relation that ultrafine precipitates of cementite of about 20 to 40 Å in size were detected in steels of D2 type, X220CrMoV 13 4 and X155CrMoV12, just after quenching at RT, as well as after subsequent DCT at –196 °C [26, 29]. These cementite particles are obviously precipitated due to the auto-tempering of martensite in the course of slow cooling down to RT. It is also important that Gogte et al. [17] presented some impressive evidence of how DCT itself, without subsequent tempering, can cause the fracture of coarse primary carbides and create the population of fine carbides resembling the “milky way” in the high-speed steel T42.

### 3. MECHANISM FOR DEEP CRYOGENIC TREATMENT

#### 3.1. Isothermal martensitic transformation

The common feature of above mentioned hypotheses is that they deny any role of martensitic transformation in the effect of DCT on properties of tool steels. This statement is based on the conviction that it is finished at temperatures above –100 °C. In other words, the martensitic transformation is identified only with its athermal mode.

However, the martensitic transformation continues with the isothermal kinetics in the temperature range below – 100 °C. Some signs of martensitic transformation at a constant temperature were sporadically observed even in the beginning of nineteenth century (see, e.g., [30-32]). The first fundamental research was carried out in 1948 by Kurdjumov and Maximova [33] in consistency with Kurdjumov's

concept of martensitic transformation in steels as a typical phase transition which includes the nucleation and growth stages.

The replacement of the burst athermal kinetics by the "soft" isothermal one is controlled by a balance between the driving force of transformation and the amplitude of thermal atomic vibrations. The increase in the driving force with decreasing temperature slows down in the cryogenic temperature range because of strengthening of the austenite, which retards the burst character of transformation. In turn, the nucleation stage appears until the energy of atomic vibrations remains sufficiently high (see about details [34]). A further decrease in temperature retards the nucleation and, finally, the transformation ceases resulting in the transformation kinetics with a maximum at some intermediate temperatures.

The following experimental data give the evidence of martensitic transformation within the temperature range below  $-100$  °C. Using mechanical spectroscopy, some transformation was revealed in steels of D2 type, X153CrMoV12 and X220CrMoV13-4, with a maximum at about  $-150$  °C (Fig. 1).

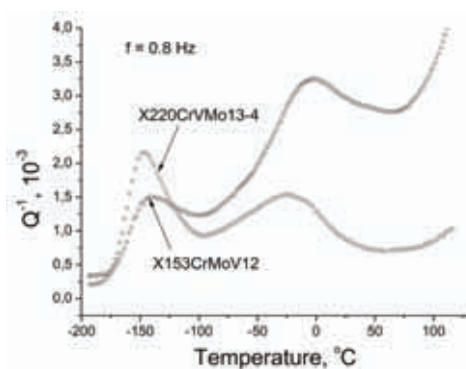


Fig. 1. Temperature dependence of internal friction for steels X153CrMoV12 and X220CrMoV13 4

The temperature of both internal friction, IF, peaks does not change with the frequency, i.e. they do not belong to the relaxation processes. As shown in [29], a broad damping in between  $-50$  °C and  $+50$  °C is controlled by the vibrations of dislocations of which mobility increases with increasing temperature and, thereafter, decreases at approaching RT, where the carbon clusters formed during the ageing of martensite prevent dislocation movement. The low temperature peak belongs to martensitic transformation. Its intensity is proportional to the fraction of the transformed martensite. It follows from Fig. 1 that transformation proceeds with maximal intensity at  $-150$  °C, which is consistent with the results in [33] obtained on the modelling steel X100Mn6. These data demonstrate that, besides the long time holding at cryogenic temperatures, the isothermal martensitic transformation also occurs in the course of permanent cooling-heating, which suggests that the virgin isothermal martensite is present in the tool steels held for DCT in liquid nitrogen and subsequently heated to RT. The X-ray diffraction measurements confirm this conclusion (Fig. 2).

It is seen that the isothermal martensitic transformation is suppressed at  $-170$  °C where the holding for 240 min does not change the intensity of martensitic reflections. The fraction of martensite was increased due to heating to  $-100$  °C with holding at this temperature for 30 min. In consistency with the evolution of  $(200)_\gamma$  reflection in Fig. 2, the holding at  $-150$  °C assisted the intensive martensitic transformation, which can be estimated on the decrease in the fraction of the retained austenite.

The quantitative information about a scale of the isothermal martensitic transformation in the course of holding at cryogenic temperatures was obtained using Mössbauer spectroscopy which allows to estimate the fraction of the martensitic and austenitic phases with the accuracy of about 1.0% (see in detail [26, 29]). Mössbauer

spectrum of steel X153CrMoV12 quenched at RT and cooled down to  $-196\text{ }^{\circ}\text{C}$  with subsequent holding for different time at this temperature is presented in Fig. 3. Again, in accordance with the IF and X-ray results, see Figures 1 and 2, no transformation occurs during the holding in liquid nitrogen.

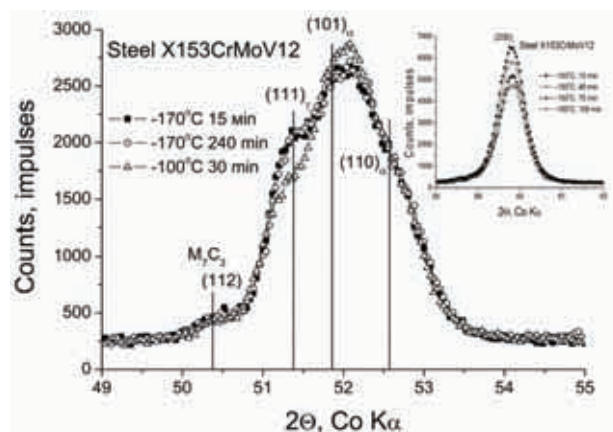


Fig. 2. Fragment of diffraction pattern of steel X153CrMoV12 quenched at RT, subsequently held at  $-170\text{ }^{\circ}\text{C}$  for 15 and 240 min and thereafter heated up to  $-100\text{ }^{\circ}\text{C}$  with holding for 30 min at this temperature. The evolution of the austenitic reflection (200)<sub>γ</sub> during isothermal martensitic transformation at  $-150\text{ }^{\circ}\text{C}$  is shown in the upper right corner. All the measurements were carried out at the temperatures of holding

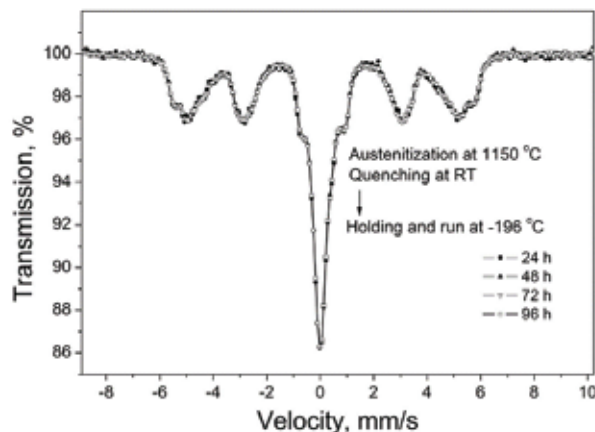


Fig. 3. Mössbauer spectrum of steel X153CrMoV12 after quenching from  $1150\text{ }^{\circ}\text{C}$  at RT and subsequent holding at  $-196\text{ }^{\circ}\text{C}$  for 24, 48, 72, 96 hours. All measurements are performed at  $-196\text{ }^{\circ}\text{C}$

In another Mössbauer experiment, the sample was heated to  $-150\text{ }^{\circ}\text{C}$  after preliminary cooling in liquid nitrogen, and, as expected, the isothermal martensitic transformation has been developed with holding time at this temperature. The data of Mössbauer measurements are presented in Table 1. All measurements were performed at  $-196\text{ }^{\circ}\text{C}$ , where martensitic transformation does not occur. About 8 % of the isothermal martensite was transformed during holding at  $-150\text{ }^{\circ}\text{C}$  in addition to that obtained below the martensitic point  $M_s$  ( $130\text{ }^{\circ}\text{C}$  in this steel) and during the cooling from RT down to  $-196\text{ }^{\circ}\text{C}$ .

Thus, it is established that the isothermal martensitic transformation occurs during deep cryogenic treatment of the studied tool steels within the temperature range

in between  $-100\text{ }^{\circ}\text{C}$  and  $-170\text{ }^{\circ}\text{C}$  with its highest intensity at  $-150\text{ }^{\circ}\text{C}$ . This transformation also proceeds during cooling/heating in between  $-100\text{ }^{\circ}\text{C}$  and  $-196\text{ }^{\circ}\text{C}$  and does not occur during holding in liquid nitrogen.

Table 1.

**Areas (% , scatter  $\pm 1\%$ ) under components belonging to the martensite, retained austenite and carbides in Mössbauer spectra of steel X153CrMoV12 after quenching from  $1150\text{ }^{\circ}\text{C}$  at RT and subsequent holding at  $-196$  or  $-150\text{ }^{\circ}\text{C}$**

Temperature and time of holding	$-196\text{ }^{\circ}\text{C}$ , 96 h	$-150\text{ }^{\circ}\text{C}$ , 24 h
Martensite	58.6	66.6
Austenite	32.6	24.6
Carbides	8.9	8.3

## 2.2. Plastic deformation of martensite during DCT and its effect on properties

The virgin isothermal martensite is characterized by the following unique properties. Pietikainen [35] was the first to demonstrate that it is rather soft and acquires a low strength. Moreover [36], the microcracks are not formed in the course of the isothermal transformation and appear only during subsequent heating of the virgin martensite at temperatures of about  $-50\text{ }^{\circ}\text{C}$  where mobility of carbon atoms sufficiently increases and the ageing starts. Another feature of the low temperature martensite is its abnormally low tetragonality, which was first time observed by Lysak and Vovk [37]. Two main hypotheses were proposed for this phenomenon: (i) the intermediate fcc $\rightarrow$ hcp transformation occurs before the bcc martensite formation resulting in the partial location of carbon atoms in the tetrahedral interstitial sites which do not contribute to martensite tetragonality [38], (ii) the twinning on the  $\{111\}$  atomic planes transfers a part of carbon atoms from the *c* sublattice of the octahedral pores into the *a* and *b* ones, which decreases tetragonality [39]. The third feature of the isothermal martensitic transformation and, correspondingly, of the isothermal martensite is that carbon atoms do not migrate at transformation temperatures [26, 40].

Taking into account the properties of the low temperature isothermal martensite and based on the analysis of the above mentioned experimental data on DCT of tool steels, the following idea is proposed as a physical reason for the effect of DCT on the properties of tool steels: due to softness of the virgin martensite, the isothermal martensitic transformation is accompanied by plastic deformation.

The volume effect of transformation serves as a driving force of plastic deformation which, in turn, should be accompanied by the following effects: (i) the previously formed RT martensite is included in the deformation process, e.g., like that occurs with the completely brittle cementite during deformation of pearlitic steels; (ii) the gliding dislocations should capture carbon atoms and transport them creating carbon dislocation atmospheres; (iii) the partial removal of carbon atoms from the solid solution is a reason for the abnormally low tetragonality of martensite; (iv) the carbon atoms at dislocations do not take part in the ageing of martensite and prevent formation of the low temperature carbide phases.

The data of transmission electron microscopy give a convincing proof of plastic deformation during DCT (Fig. 4, see in detail [29, 40]).

Wilson [41] has shown that plastic deformation of the plain carbon steel martensite, as performed by the hardness test, suppresses precipitation of  $\epsilon$ -carbide during subsequent tempering. It is consistent with the result presented in Fig. 4.

It is natural to suppose that plastic deformation in the course of DCT should also delay precipitation of cementite and special carbides. TEM data in Fig. 5 confirm

this prediction. Carbides  $M_7C_3$  and  $M_2C$  are present in steel after quenching at RT and subsequent tempering at 500 °C (Fig. 5a). In contrast, the only coarse plates of  $\theta$ -cementite and no special carbides are seen in the structure formed at this temperature after DCT at  $-150$  °C and  $-196$  °C (Figures 5b, c). This is why the secondary hardness does not become clearly apparent in case of DCT (see, e.g., [40]).

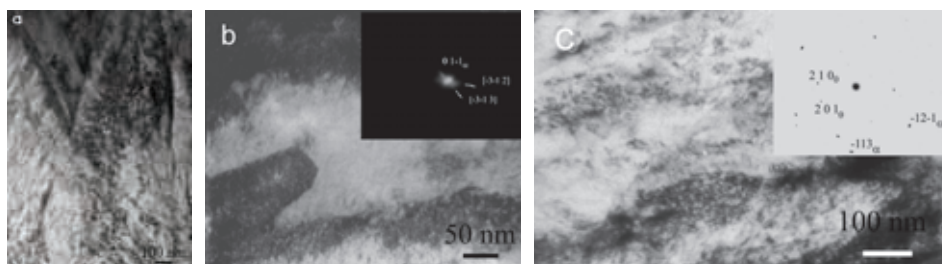


Fig. 4. Precipitation behavior in steel X153CrMoV12 due to tempering for 2 hours: (a)  $\epsilon$ -carbide after quenching at RT and tempering at 200 °C; (b) no  $\eta(\epsilon')$ -carbide after DCT at  $-150$  °C for 24 hours and tempering at 100 °C and (c) the same after DCT at  $-150$  °C and tempering at 200 °C.

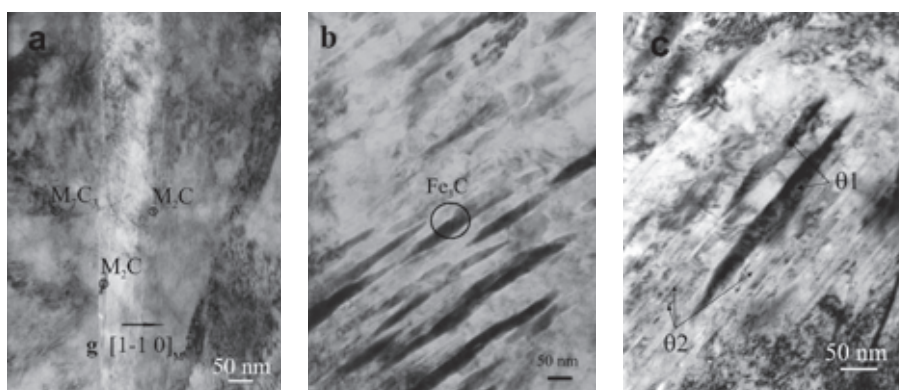


Fig. 5. Precipitation in steel X153CrMoV12 due to tempering at 500 °C for 2 hours after different preliminary treatments: (a) quenching at RT; (b) quenching at RT and DCT at  $-150$  °C for 24 h; (c) quenching at RT and DCT at  $-196$  °C for 24 h.

Using Mössbauer spectroscopy [40], it is also shown that, after DCT and tempering at 500 °C, the  $\alpha$ -solid solution contains clusters enriched in carbon and alloying elements (Cr, V) and, therefore, conserves a remarkable potential for precipitation, e.g., during subsequent tools service.

Considering this, one can interpret the above-mentioned remarkable influence of sliding speed on the wear rate (Fig. 6), as it was observed in [3].

The nature of this effect is clearly consists in the DCT-caused delay of special carbide precipitation. With increasing sliding speed, the temperature increases and the stress-induced precipitation of special carbides occurs

Therefore, it is shown that plastic deformation in the course of the DCT plays an important role in the carbide precipitation during tempering of tool steels. Based on this result, one can also interpret the above-mentioned fracture of primary  $M_7C_3$  carbides and the “milky way” carbide picture caused by DCT in the high-speed steel, as obtained by Googt et al. [17].

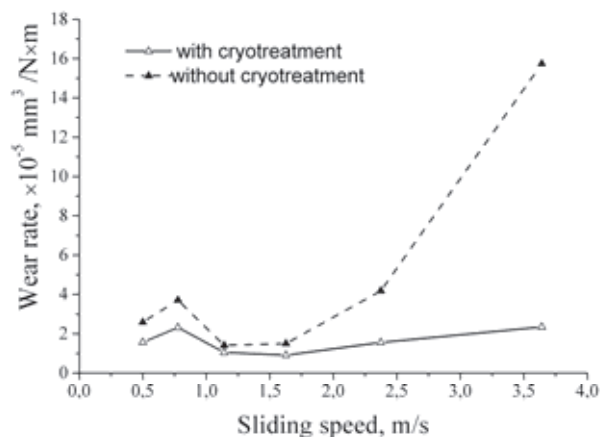


Fig. 6. Sliding wear rate of D2 type steel as a function of sliding speed, after [3]

The occurrence of the isothermal martensitic transformation during DCT, the plastic deformation caused by this transformation and its effect on the subsequent carbide precipitation shed light on the phenomenon of DCT and makes possible to derive some recommendations concerning its optimal technology.

#### SUMMARY

The isothermal martensitic transformation constitutes a decisive structural phenomenon in tool steels subjected to deep cryogenic treatment. Its temperature range can depend on the chemical compositions of steels. In the studied tool steels, it proceeds with the highest intensity near  $-150$  °C. In absence of ageing, the virgin low temperature martensite is rather soft, and plastic deformation of steel occurs due to the volume effect of transformation.

Because of dislocation slip during plastic deformation, the immobile at these temperatures carbon atoms are captured by dislocations and removed from the  $\alpha$  solid solution. The binding between carbon atoms and dislocations prevents precipitation of the low temperature  $\epsilon$ -carbide during subsequent tempering and shifts precipitation of cementite and special carbides to higher temperatures.

The cementite is the only carbide formed at temperatures up to  $500$  °C after DCT, which explains the insignificant effect of secondary hardness. Correspondingly, the  $\alpha$  solid solution contains clusters of carbon and carbide forming elements, which creates a remarkable potential for further precipitation. Admittedly, the stress-induced precipitation of special carbides can occur during subsequent usage of tools, which increases the tools life.

A change in technology of DCT can be recommended aiming the holding within the temperature range of the most intensive isothermal martensitic transformation, which can decrease the time of holding during DCT and be the most effective for the increase in the wear resistance of steels.

*В статье представлен краткий обзор экспериментальных данных о влиянии глубокой криогенной обработки (ГКО) на износ инструментальных сталей и существующих гипотез о ее физической природе. С помощью мессбауэровской спектроскопии, рентгеноструктурного анализа, механической спектроскопии и просвечивающей электронной микроскопии показано, что в процессе ГКО происходит изотермическое мартенситное превращение, во время которого имеет место пластическая деформация из-за низкой твердости новообразованного низкотемпературного мартенсита. Во время пластической деформации*



скользящие дислокации увлекают за собой атомы углерода из твердого раствора, а поскольку энтальпия связи атомов углерода с дислокациями превосходит энтальпию формирования  $\epsilon$ -карбида и цементита, происходит задержка выделения карбидов в процессе отпуска. Таким образом, альфа твердый раствор содержит кластеры углерода и легирующих элементов и сохраняет потенциал для выделений во время практического применения инструмента, продлевая срок его использования. Разработана новая концепция ГКО и предложена коррекция ее технологии с учетом температурного диапазона, при котором происходит изотермическое мартенситное превращение.

**Ключевые слова:** Инструментальная сталь; Термическая обработка; Глубокая криогенная обработка; Мартенситное превращение; Пластическая деформация; Отпуск; Износ; Механические свойства.

В статті наведено короткий огляд експериментальних даних про вплив глибокої криогенної обробки (ГКО) на зношування інструментальних сталей та існуючих гіпотез щодо її фізичної природи. За допомогою мессбауерівської спектроскопії, рентгеноструктурного аналізу, механічної спектроскопії та просвічуючої електронної мікроскопії показано, що під час ГКО відбувається ізотермічне мартенситне перетворення, під час якого має місце пластична деформація з причини малої твердості новоутвореного низько температурного мартенситу. Під час пластичної деформації ковзаючі дислокації захоплюють за собою атоми вуглецю із твердого розчину, а оскільки ентальпія зв'язку атомів вуглецю із дислокаціями перевищує ентальпію формування  $\epsilon$ -карбиду та цементиту, відбувається затримка виділення карбідів в процесі відпуску. Таким чином, альфа твердий розчин містить кластери вуглецю та легувальних елементів і зберігає потенціал для виділень під час практичного застосування інструмента, подовжуючи термін його використання. Розроблено нову концепцію ГКО та запропоновано корекцію цієї технології, зважаючи на температурний діапазон, при якому відбувається ізотермічне мартенситне перетворення.

**Ключові слова:** Інструментальна сталь; Термічна обробка; Глибока криогенна обробка; Мартенситне перетворення; Пластична деформація; Відпуск; Спрацювання; Механічні властивості.

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