

# DEEP CRYOGENIC TREATMENT OF H11 HOT-WORKING TOOL STEEL

## GLOBOKA KRIOGENSKA OBDELAVA ORODNEGA JEKLA H11 ZA DELO V VROČEM

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Unlike a conventional cold treatment, which is commonly used for the elimination of retained austenite, a deep cryogenic treatment (DCT) primarily improves the wear resistance of tools. This effect is supposed to result from the preferential precipitation of fine  $\eta$ -carbides, whose formation mechanism has been the subject of several recent investigations, performed mainly on high-speed steels. This article describes the influence of a DCT on the microstructure and properties of X37CrMoV5-1 (H11) hot-working tool steel. The wear resistance of specimens treated using DCT was analysed using a pin-on-disc wear tester and compared to that of specimens treated using standard quenching and tempering. In addition, the specimens' microstructures were analysed by TEM. The results show a significant improvement in the wear resistance as a consequence of the DCT, especially at the high sliding velocities that are typical of many industrial applications for hot-working steels (e.g., closed die forging). Apart from this, some effects of DCT on the microstructure were found, which contributed to a better understanding of this process.

Keywords: tool steel, wear resistance, microstructure

Različno od konvencionalnega podhlajevanja, ki se navadno uporablja za odpravo zaostalega avstenita, se kriogena obdelava (DCT) uporablja predvsem za povečanje obrabne odpornosti jekla. Domneva se, da to izvira iz preferenčnega izločanja drobnih  $\eta$ -karbidov. Mehanizem njihovega nastanka je predmet številnih raziskav, narejenih predvsem pri hitroreznih jeklih. Članek opisuje vpliv DCT na mikrostrukturo in lastnosti X37CrMoV5-1 (H11) orodnega jekla za delo v vročem. Obrabna odpornost vzorcev, obdelanih z DCT, je bila določena na napravi za določanje obrabe "pin-on-disc" in primerjana z obrabno odpornostjo vzorcev, obdelanih z navadnim kaljenjem in popuščanjem. Mikrostruktura vzorcev je bila analizirana s TEM. Rezultati kažejo občutno izboljšanje odpornosti proti obrabi pri DCT-obdelavi, posebno pri velikih hitrostih drsenja, ki so značilne pri mnogih vrstah industrijske uporabe jekel za delo v vročem (npr. kovanje v utopih). Poleg tega so bili ugotovljeni vplivi DCT na mikrostrukturo, kar prispeva k boljšemu razumevanju tega procesa.

Ključne besede: orodno jeklo, odpornost proti obrabi, mikrostruktura

## 1 INTRODUCTION

The deep cryogenic treatment of steels has been the subject of numerous research and experimental studies over the past twenty years. Its practical significance has been growing with new findings, which suggest that the process may substantially extend the life of various types of tools. From the technical viewpoint, a deep cryogenic treatment is a single processing operation that immediately follows the conventional quenching (and precedes tempering). One should, however, distinguish between a deep cryogenic treatment and a conventional cold treatment. The latter has been commonly used in industry since the 1950s. The purpose of a cold treatment (same as in multiple tempering) is to eliminate the retained austenite from the microstructure of the hardened steel. In high-alloyed tool steels the retained austenite remains stable down to the approximate temperature range  $-80$  °C to  $-120$  °C. In a cold treatment, a temperature below this level is reached only once in the process. The treatment improves the material's mechanical properties (i.e., the strength) and enhances the dimensional stability for tools processed in this manner.

In contrast, in a deep cryogenic treatment the elimination of retained austenite is no more than a side effect. A number of reports suggest that in steels the supercooling below  $-150$  °C and a subsequent long holding time (of the order of hours or tens of hours) at the low temperature leads to a substantial improvement in their resistance wear in service. This applies even in comparison with materials subjected to a conventional cold treatment (i.e., materials with a zero retained austenite content).

The improvement in tool life due to a deep cryogenic treatment has recently been reported in various types of forming and cutting tools.<sup>1,2</sup> In forging dies from the X37CrMoV5-1 (H11) steel, an improvement in service life by 40 % was proven repeatedly,<sup>2</sup> when compared to conventional quenching and tempering without a deep cryogenic treatment. An extensive series of experiments on various types of punching tools, milling cutters and drill bits from several types of tool steels demonstrated a definite contribution of the deep cryogenic treatment to a longer tool life.<sup>3</sup> Another widely published series of experiments was conducted on turning tools made from HS10-4-3-10 high-speed steel.<sup>4</sup> In this case, too, the

improvement in the cutting edge's life thanks to a deep cryogenic treatment was demonstrated.

The microstructural aspects of these effects were explored in the 1990s by Collins and other researchers.<sup>5-8</sup> In the case of the X155CrVMo12-1 steel (the nearest equivalent grade: D2), a large content of fine secondary carbides was found in the deep cryogenically treated material, in addition to its improved wear resistance over that of the conventionally quenched and tempered material. The changes in microstructure taking place in the course of the deep cryogenic treatment are summarised and described by Collins as a cold treatment modification of martensite. The author believes that the modification consists of the formation of a large number of lattice defects that serve as nuclei for the precipitation of these fine carbides during tempering. It should be noted that all these conclusions have been drawn on the basis of nothing more than a quantitative image analysis of optical micrographs. From today's perspective, this is an insufficiently detailed analysis of the microstructure.

Another in-depth investigation of the behaviour of X155CrVMo12-1 steel (the nearest equivalent grade: D2) after a deep cryogenic treatment was reported by Das et al.<sup>9-11</sup> Tests of the wear resistance revealed its improvement by 12–39 % after cold treatment and by 34–88 % after a deep cryogenic treatment, when compared to conventional quenching and tempering. Das reported that a deep cryogenic treatment leads to a permanent change in the carbide precipitation kinetics. According to his report, the material upon deep cryogenic treatment contains 22 % more carbides per unit volume than the conventionally quenched and tempered material.

The most recent findings on deep cryogenic treatment were reported in 2011 by Oppenkowski,<sup>1</sup> who carried out an in-depth analysis of several types of high-speed steels that were processed using various technologies, including cold and deep cryogenic treatments. He proved that steels, after various hardening proce-

dures, contain different types of martensite. According to his findings, deep cryogenically treated materials are characterized by a higher content of martensite with less tetragonal distortion and finer twin structures. These microstructural changes are probably the cause of the precipitation of a large amount of fine carbides during the subsequent tempering.

The present paper describes the investigation procedure and the results of exploring the impact of a deep cryogenic treatment on the wear resistance and microstructure of the X37CrMoV5-1 (H11) hot-work steel. The wear resistance was measured at 400 °C using the pin-on-disc method with a rotary tribometer. The microstructure of the steel was examined using light and transmission electron microscopes.

## 2 EXPERIMENTAL

### 2.1 Heat Treating

The experimental specimens of the X37CrMoV5-1 (H11) tool steel had a diameter of 55 mm and a height of 10 mm. These specimens were treated using the schedules listed in **Table 1**. Two specimens were prepared with each schedule. One was used for wear-resistance testing and the other for microstructural observations. The hardness of all the heat-treated specimens was 52 HRC.

**Table 1:** Experimental heat-treatment schedules

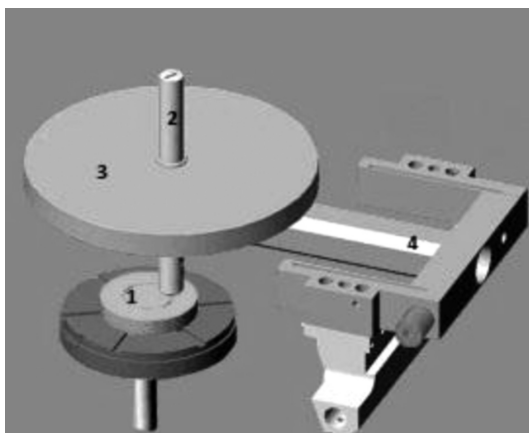
**Tabela 1:** Pregled eksperimentalnih toplotnih obdelav

Specimen Designation	Heat Treating Schedule
1 (H+2T)	Heating to 1030 °C, quenching in oil, double tempering (610 °C)
2 (H+6C+2T)	Heating to 1030 °C, quenching in oil, deep freezing at -160 °C for 6 h, double tempering (610 °C)
3 (H+12C+2T)	Heating to 1030 °C, quenching in oil, deep freezing at -160 °C for 12 h, double tempering (610 °C)
4 (H+20C+2T)	Heating to 1030 °C, quenching in oil, deep freezing at -160 °C for 20 h, double tempering (610 °C)

### 2.2 Wear Resistance

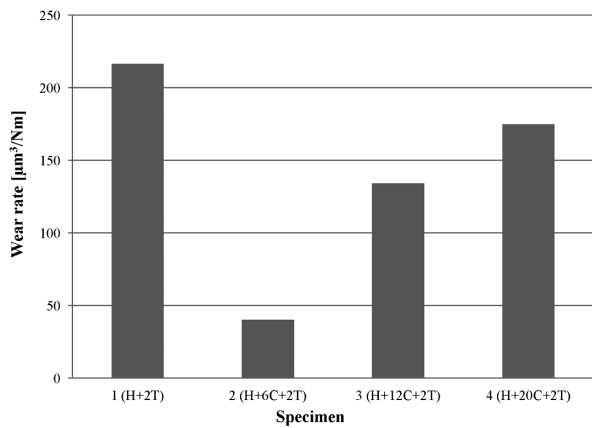
The wear-resistance tests were performed using the pin-on-disc method. The principle of this test is forcing a ceramic ball into the surface of a rotating flat specimen. The holder with the ball indenter is pressed using a defined force (exerted by a weight) against the specimen. The holder is attached to a flexible arm, through which the dependence of the friction coefficient on the length of the ball's path is recorded by means of strain gauges. The testing equipment is schematically shown in **Figure 1**.

After the test, the profile of the resulting wear track is measured using a contact profilometer. The wear of the



**Figure 1:** Pin-on-disc equipment: 1-test sample, 2-indenter holder with ball indenter, 3-weight, 4-flexible arm

**Slika 1:** Naprava "pin-on-disc": 1-vzorec, 2-nosilec kroglastega utiskovalnika, 3-utež, 4-gibljiva ročica



**Figure 2:** Wear rates of pin-on-disc test specimens  
**Slika 2:** Hitrost obrabe na preizkušancih "pin-on-disc"

test specimen is calculated from the measured data as follows:

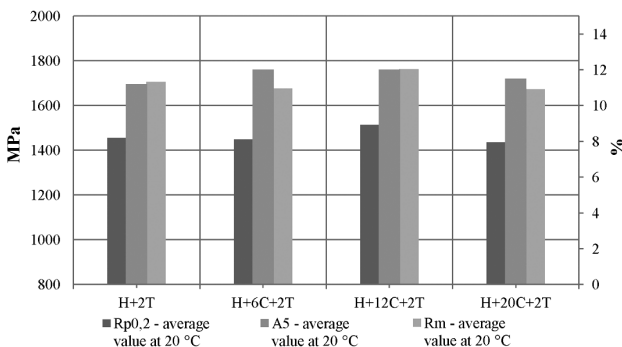
$$W/(\mu\text{m}^3/\text{N m}) = \frac{\text{Wear track volume } (\mu\text{m}^3)}{\text{Load (N)} \cdot \text{Path travelled by ball indenter (m)}}$$

The specimens of X37CrMoV5-1 (H11) were tested with the following parameters:

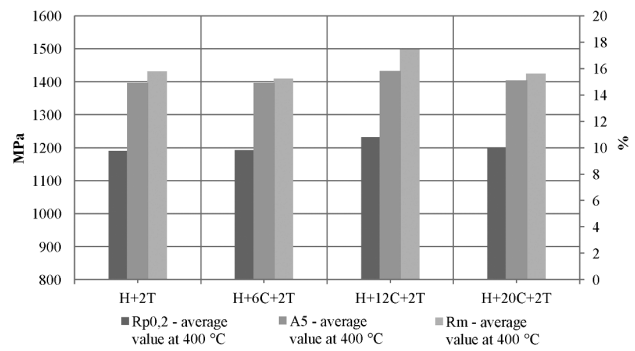
- Indenter holder with a ball indenter ( $\text{Si}_3\text{N}_4$ ) with the diameter of  $D = 6 \text{ mm}$
- Wear-track diameter:  $r = 3.00 \text{ mm}$
- Temperature:  $400 \text{ }^\circ\text{C}$
- Load:  $10 \text{ N}$
- 5000 cycles (specimen revolutions)

The choice of a temperature of  $400 \text{ }^\circ\text{C}$  is based on the conditions for the most frequent industrial applications of the steel in question: forging dies, injection moulds and other tools whose surfaces may experience a high temperature in service. For example, during closed-die forging, the temperatures of the die surface may reach approximately  $250\text{--}500 \text{ }^\circ\text{C}$ .

The wear rates of the specimens tested are given in **Figure 2**. The results of the measurement suggest that a deep cryogenic treatment dramatically improves the material's wear resistance. However, with the holding time at the deep cryogenic temperature becoming longer,



**Figure 3:** Results of tensile tests  
**Slika 3:** Rezultati nateznih preizkusov



**Figure 4:** Results of tensile tests  
**Slika 4:** Rezultati nateznih preizkusov

the resistance declines again. This occurrence was mentioned in several reports (e.g.,<sup>1</sup>) but no satisfactory theoretical explanation has been found.

### 2.3 Tensile tests

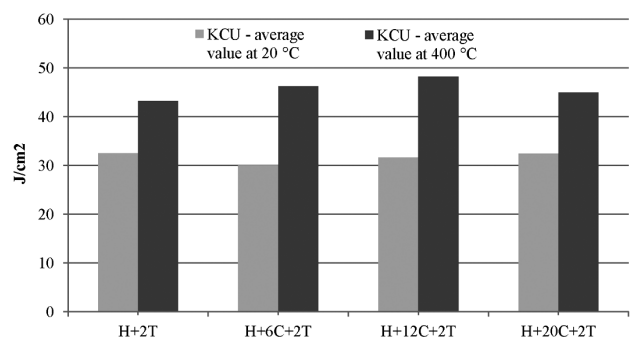
Standard tensile tests of specimens treated according to **Table 1** were performed at room temperature and at  $400 \text{ }^\circ\text{C}$ . For each testing temperature, three specimens were tested. As the following results (**Figures 3 and 4**) show, no significant influence of the deep cryogenic treatment on the tensile strength or the elongation was found.

### 2.4 Impact tests

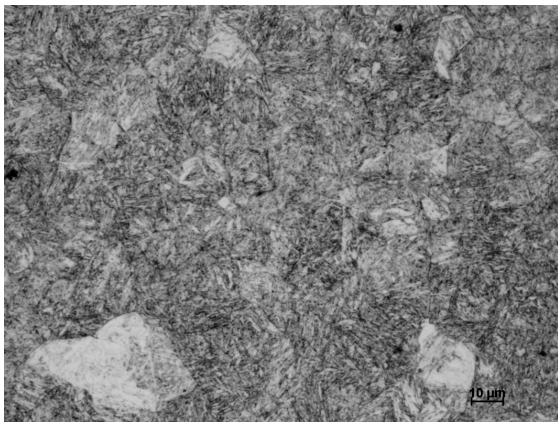
Standard specimens ( $10 \text{ mm} \times 10 \text{ mm} \times 55 \text{ mm}$ , with a U-formed notch, the radius of the notch is  $1 \text{ mm}$ , the depth of the notch is  $5 \text{ mm}$ ) treated according to the above-mentioned regimes undertook the Charpy impact tests at  $20 \text{ }^\circ\text{C}$  and  $400 \text{ }^\circ\text{C}$ . Similar to the results of the tensile tests, no significant influence of the deep cryogenic treatment on the notch toughness of the investigated material was observed (**Figure 5**).

### 2.5 Light Microscopy Observation

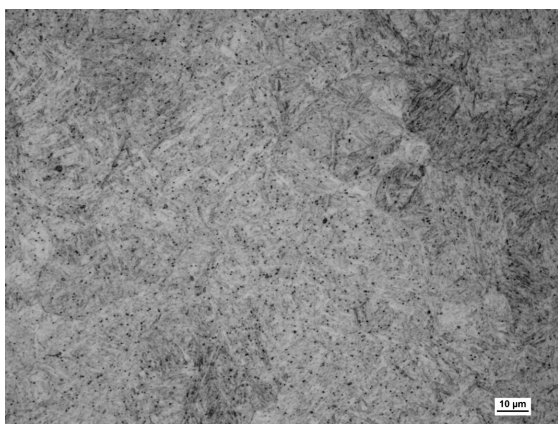
The specimens were prepared using a standard metallographic procedure: grinding and subsequent polishing.



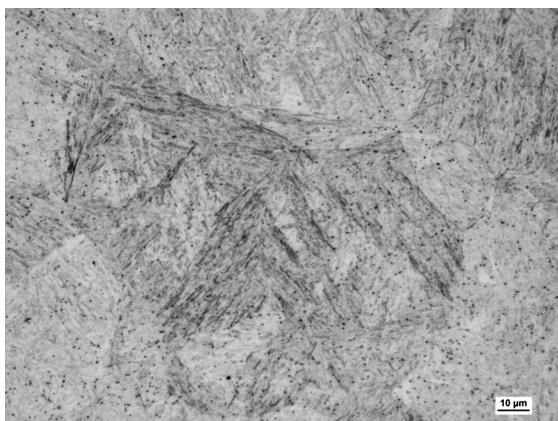
**Figure 5:** Results of impact tests  
**Slika 5:** Rezultati udarnih preizkusov



**Figure 6:** Microstructure of specimen 1 (H+2T); etched in nital 3 %  
**Slika 6:** Mikrostruktura vzorca 1 (H+2T); jedkano v nitalu 3 %

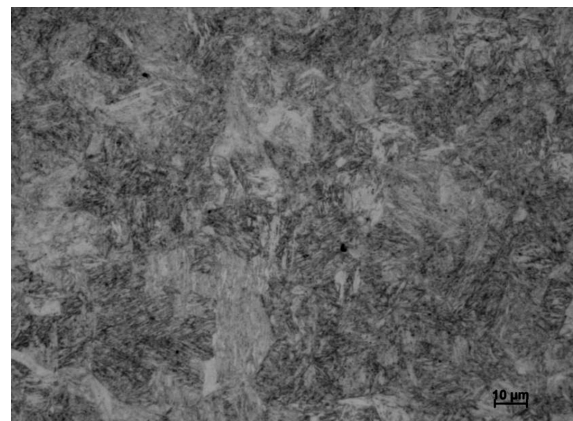


**Figure 7:** Microstructure of specimen 2 (H+6C+2T); etched in nital 3 %  
**Slika 7:** Mikrostruktura vzorca 2 (H+6C+2T); jedkano v nitalu 3 %



**Figure 8:** Microstructure of specimen 3 (H+12C+2T); etched in nital 3 %  
**Slika 8:** Mikrostruktura vzorca 3 (H+12C+2T); jedkano v nitalu 3 %

Their microstructures were revealed by etching with nital 3 % and photographed using a NIKON EPIPHOT 200 light microscope. As the micrographs below show, the microstructures in all the specimens examined are very similar. They consist in all cases of martensite laths, within which some fine carbides can be found. Neither non-metallic inclusions nor other microstructure defi-



**Figure 9:** Microstructure of specimen 4 (H+20C+2T); etched in nital 3 %  
**Slika 9:** Mikrostruktura vzorca 4 (H+20C+2T); jedkano v nitalu 3 %

ciencies were revealed. Micrographs of the individual specimens are shown in **Figures 6 to 9**.

## 2.6 Transmission Electron Microscopy Observation

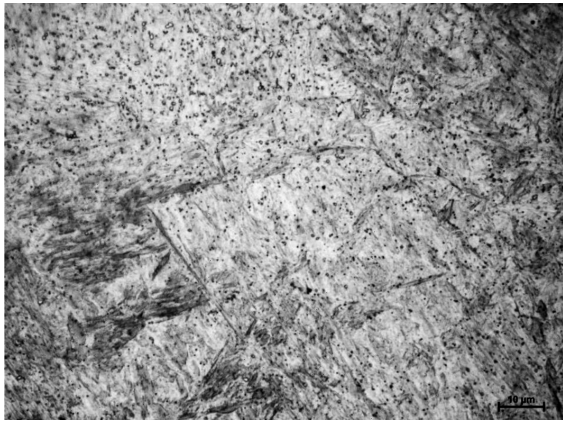
In order to document the impact of a deep cryogenic treatment on the martensite morphology more accurately, the tempering at 610 °C was omitted in the case of specimens for transmission electron microscopy observation. The reason was that some recent reports (e.g.,<sup>1</sup>) claim that at higher tempering temperatures, the precipitation of fine carbides in tool steels is accompanied by a gradual annihilation of some types of lattice imperfections formed during the deep cryogenic treatment. For this reason, only low-temperature tempering at 180 °C was applied. The heat-treatment schedules for the specimens investigated by means of transmission electron microscopy are detailed in **Table 2**.

**Table 2:** Heat treatment of specimens for transmission electron microscopy observation

**Tabela 2:** Toplotna obdelava vzorcev za presevno elektronsko mikroskopijo

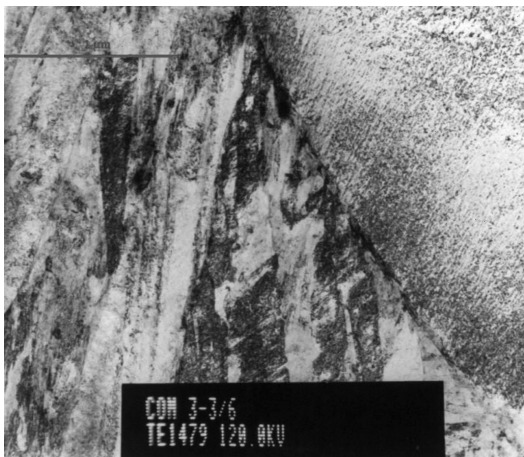
Specimen Code	Heat Treating Schedule
1T (H+2T)	Heating to 1030 °C, quenching in oil, tempering (180 °C)
2T (H+6C+2T)	Heating to 1030 °C, quenching in oil, deep freezing at -160 °C for 6 h, tempering (180 °C)

The microstructures of specimens 1T and 2T consist of tempered martensite. No significant differences between these specimens were observed in the light microscope (**Figure 10** – etched with Vilella-Bain reagent). The bands occur in both samples as a result of the segregation of alloying elements – this is a typical phenomenon in the forgings of hot-working tool steels. A Widmannstätten structure was evident in the more intensively etched regions (bands), whereas a rather featureless structure with a high density of precipitates was found in adjacent regions.

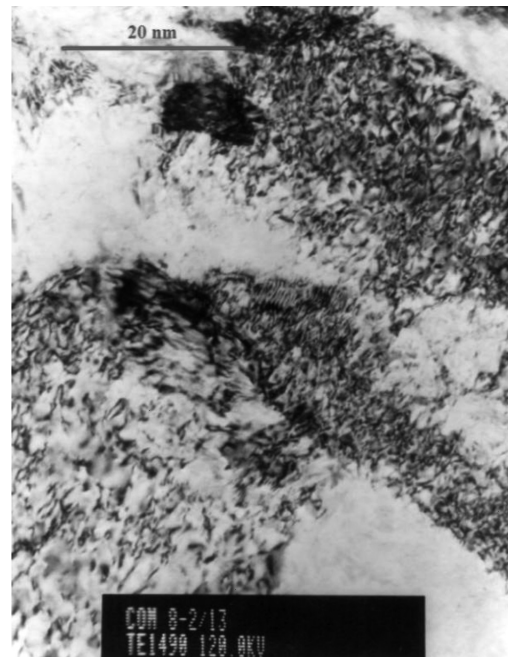


**Figure 10:** Microstructure of 2T specimen; etched in Vilella-Bain  
**Slika 10:** Mikrostruktura vzorca 2T; jedkano z jedkalom Vilella-Bain

Thin foils for the transmission electron microscopy observations were prepared from specimens 1T and 2T using jet electrolytic polishing in a 6 % solution of perchloric acid in methanol at a temperature of approximately  $-50\text{ }^{\circ}\text{C}$ . Thin areas, transparent to electrons, accelerated by a voltage of 120 kV were found predominantly in the above-mentioned bands of the featureless structure. Two different substructures were observed. Tempered martensite of plate-like, rather than lath-like, character with very thin twins (a width of several tens of nanometres) was observed in foils from both specimens (**Figure 11**). In the 2T specimen, very fine precipitates at boundaries of the ferritic laths and twins were observed. A crystallographic investigation has not been performed yet, as the electron-diffraction patterns obtained are still not finished and ready for a reliable phase identification. The second type of substructure revealed a specific striped contrast and diffraction patterns with streaks typical for structures after spinodal decomposition (**Figure 12**). In these areas, some globular and relatively coarse particles were observed – probably those that were also observed using the light microscope.



**Figure 11:** TEM image of specimen 1T  
**Slika 11:** TEM-posnetek vzorca 1T



**Figure 12:** TEM image of specimen 2T  
**Slika 12:** TEM-posnetek vzorca 2T

It seems that both phase transformations occur in the steel during a deep cryogenic treatment: the displacive martensitic transformation and the spinodal decomposition. It can be supposed that the spinodal decomposition occurred predominately in areas enriched with carbon and other alloying elements. Furthermore, the above-described substructures seem to promote the nucleation of very fine carbides during the annealing of the investigated steel.

### 3 CONCLUSIONS

Tests carried out in this study have shown that a deep cryogenic treatment of the X37CrMoV5-1 (H11) steel has no influence on its tensile strength and notch toughness, but it dramatically improves its resistance to wear at high temperatures (as determined by the pin-on-disc test). However, the length of the holding time at the temperature of the deep cryogenic treatment is crucial. The optimum time appears to be 6 hours. A microstructural observation in a light microscope did not reveal any substantial differences between the specimens hardened in the conventional manner and the specimens after a deep cryogenic treatment. On the other hand, the analysis of specimens upon deep cryogenic treatment by means of transmission electron microscopy found two types of substructure, which are likely to facilitate the precipitation of fine carbides during the final tempering of the steel. At this point, the impact of longer holding times at the deep cryogenic temperature on this substructure has not been analysed. The cause of the decline in the wear resistance with extended holding times cannot thus be determined.

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