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Deep cryogenic treatment of H11 hot working tool steel

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ABSTRACT

Purpose: The investigation procedure and results of exploring the impact of deep cryogenic treatment on the wear resistance and microstructure of the X37CrMoV5-1 hot work steel.

Design/methodology/approach: The wear resistance and microstructure of the X37CrMoV5-1 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 optical and transmission electron microscopes.

Findings: The result show a significant improvement of wear resistance through DCT, especially at high sliding velocities that are typical of many industrial applications of hot working steels (e.g. closed die forging). Apart from this, some effects of DCT on the microstructure were found, which contributed to better understanding of this process.

Practical implications: The article describes the influence of DCT on the microstructure and properties of X37CrMoV5-1 (H11) hot working tool steel. Wear resistance of specimens treated using DCT was analysed on pin-on-disc wear tester and compared to that of specimens treated using standard quenching and tempering. Furthermore, the specimens' microstructures were analysed by TEM.

Originality/value: Unlike conventional cold treatment, which is commonly used for elimination of retained austenite, deep cryogenic treatment (DCT) primarily improves the wear resistance of tools. This effect is supposed to result from preferential precipitation of fine η-carbides whose formation mechanism is the subject of several recent investigations performed mainly on high speed steels.

Keywords: Tool steel; Microstructure; Wear resistance; Dilatometry; Precipitation

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PROPERTIES

1. Introduction

Deep cryogenic treatment of steels has been the subject of numerous research and experimental studies over the last 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, deep cryogenic treatment is a single processing operation which immediately follows conventional quenching (and precedes tempering). One should, however, distinguish between deep cryogenic treatment and the conventional cold treatment. The latter has been commonly used in industry since the 1950s. The purpose of cold treatment (same as in multiple tempering) is to eliminate retained austenite from the microstructure of the hardened steel. In high-alloyed tool steels, retained austenite remains stable down to the approximate temperature range of -80°C to -120°C. In cold treatment, a temperature below this level is reached only once in the process. The treatment improves the material's mechanical properties (namely strength) and enhances the dimensional stability in tools processed in this manner.

By contrast, in 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 subsequent long holding (on 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 upon conventional cold treatment (i.e. with materials with zero retained austenite content).

The improvement in tool life by deep cryogenic treatment has been recently reported in various types of forming and cutting tools. In forging dies from the X37CrMoV5-1 steel, the improvement in service life by 40% was proven repeatedly [2] when compared to conventional quenching and tempering without 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 deep cryogenic treatment to longer tool life [3]. Another widely published series of experiments was conducted on turning tools from the HS10-4-3-10 high-speed steel [4]. In this case, too, the improvement in cutting edge life thanks to deep cryogenic treatment was demonstrated.

The microstructure aspects of these effects were explored in 1990s by Collins and other researchers [1, 5, 6, 7, 8]. In case of the X155CrVMo12-1 steel, a high 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 deep cryogenic treatment are summarised and described by Collins as the cold treatment modification of martensite. The author believes that the modification consists of formation of a large number of lattice defects which serve as nuclei for 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 quantitative image analysis of optical micrographs. From today's perspective, this is an insufficiently detailed analysis of microstructure.

Another in-depth investigation of the behaviour of the X155CrVMo12-1 steel upon deep cryogenic treatment was reported by Das et al. [1, 9, 10, 11]. Tests of wear resistance revealed its improvement by 12-39% upon cold treatment and by 34-88% upon deep cryogenic treatment when compared to conventional quenching and tempering. Das reported that 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 one.

The most recent findings on deep cryogenic treatment have been reported in 2011 by Oppenkowski [1] who carried out an in-depth analysis of several types of high-speed steels which were processed using various technologies, including cold and deep cryogenic treatments. He proved that steels upon different hardening procedures 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 precipitation of the large amount of fine carbides during subsequent tempering.

The present paper describes the investigation procedure and results of exploring the impact of deep cryogenic treatment on the wear resistance and microstructure of the X37CrMoV5-1 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 optical and transmission electron microscopes.

2. Heat treatment

Specimens of the X37CrMoV5-1 tool steel had a diameter of 55mm and a height of 10mm. These specimens were treated using schedules listed in Tab. 1. Two specimens were prepared with each schedule. One was used for wear resistance testing and the other for microstructure observation. The hardness of all heat treated specimens was 52 HRC.

3. Wear resistance

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 by 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 thelength of the ball's path is recorded by means of strain gauges. The testing equipment is shown schematically below (Fig. 1).

Table 1.
Experimental heat treating schedules

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 hours, double tempering (610°C)
3 (H+12C+2T)	Heating to 1030°C, quenching in oil, deep freezing at -160°C for 12 hours, double tempering (610°C)
4 (H+20C+2T)	Heating to 1030°C, quenching in oil, deep freezing at -160°C for 12 hours, tempering (610°C)

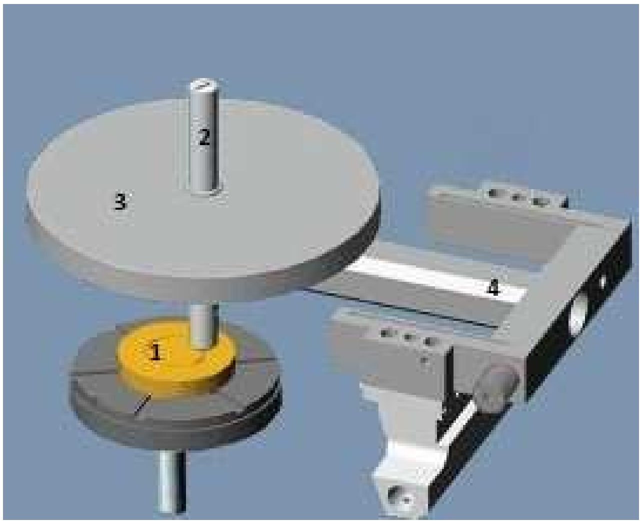

Fig. 1. Pin-on-disc equipment: 1. Test sample, 2. Indenter holder with ball indenter, 3. Weight, 4. Flexible arm

After the test, the profile of the resulting wear track is measured using a contact profilometer. The wear of the test specimen is calculated from the measured data as follows:

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

The specimens of the X37CrMoV5-1 were tested at the following parameters:

- Indenter holder with a ball indenter (Si_3N_4) with the diameter of D=6mm;
- Wear track diameter: r=3.00mm;
- Temperature: 400°C;
- Load: 10N;
- 5000 cycles (specimen revolutions).

The choice of the temperature of 400 °C is based on conditions of the most frequent industrial applications of the steel in question: forging dies, injection moulds and other tools whose surfaces may have high temperature in service. Example: during closed die forging, the temperatures of the die surface may reach approx. 250-500°C.

The wear rates of the specimens tested are given in Fig. 2. Results of the measurement suggest that deep cryogenic treatment dramatically improves the material's wear resistance. However, with the holding time at the deep cryogenic temperature increasing further, the resistance declines again. This occurrence was mentioned in several reports (e.g. [1]) but no satisfactory theoretical explanation has been found.

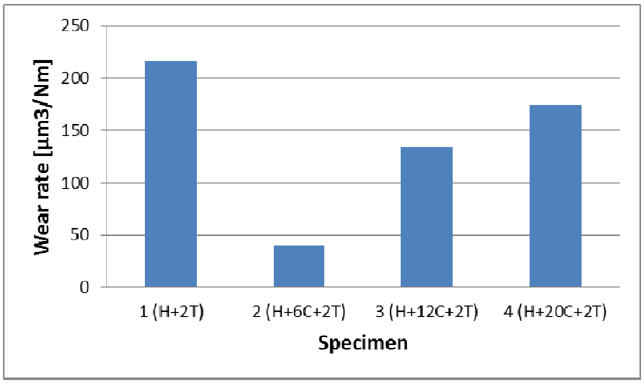

Fig. 2. Wear rates of pin-on-disc test specimens

4. Transmision electron microscopy

In order to document the impact of 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. [1]) 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 deep cryogenic treatment. For this reason, only low-temperature tempering at 180°C was applied. The heat treating schedules for specimens explored by means of transmission electron microscopy are detailed in Table 2.

Table 2.
Heat treatment of specimens for transmission electron microscopy observation

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 hours, tempering (180°C)

Thin foils for transmission electron microscopy observation were prepared from specimens 1T and 2T using jet electrolytic polishing in 6% solution of perchloric acid in methanol at a temperature of approx. -50°C. Thin areas transparent to electrons accelerated by a voltage of 120kV were found predominantly in 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 (see Fig. 3).

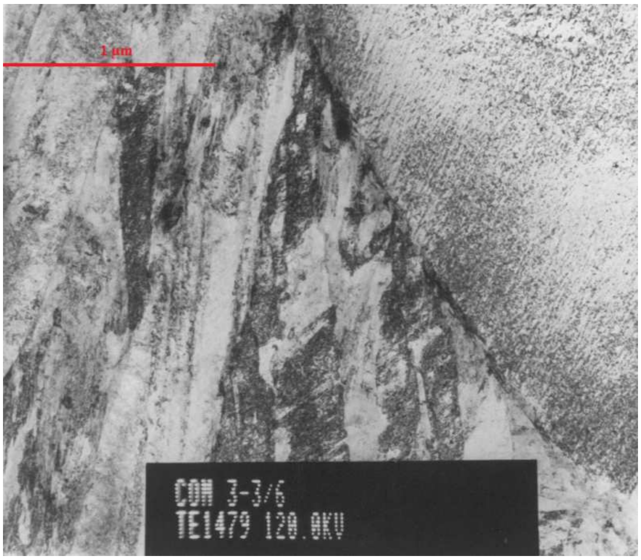

Fig. 3. TEM image of specimen 1T

In the 2T specimen, very fine precipitates at boundaries of ferritic laths and twins were observed. The crystallographic investigation has not been performed yet, as the electron diffraction patterns obtained are still not finished and ready for reliable phase identification. The second type of substructure revealed a specific striped contrast and diffraction patterns with streaks typical for structures after spinodal decomposition (see Fig. 4). In these areas, some globular and relatively coarse particles were observed-probably those, which were also observed using light microscope.

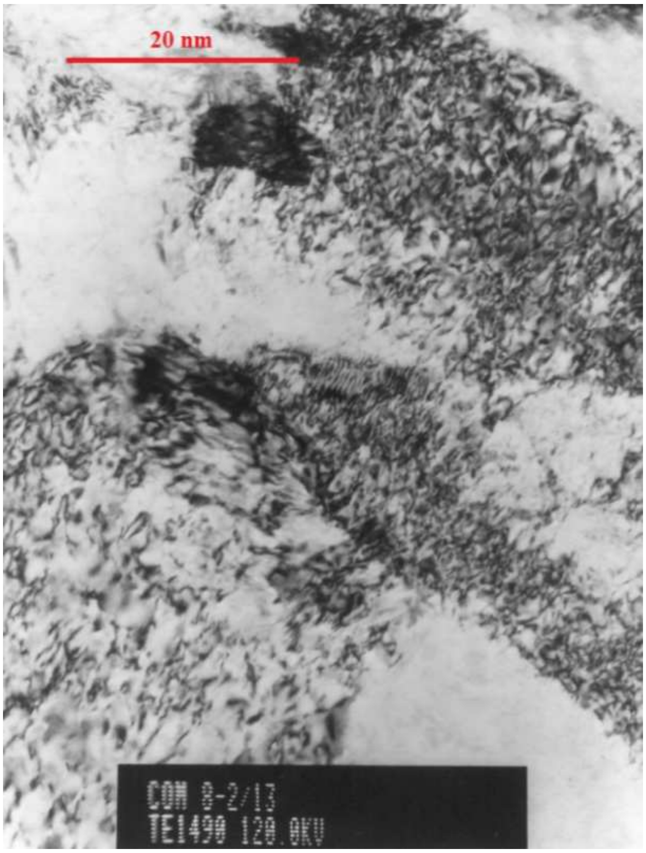

Fig. 4. TEM image of specimen 2T

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

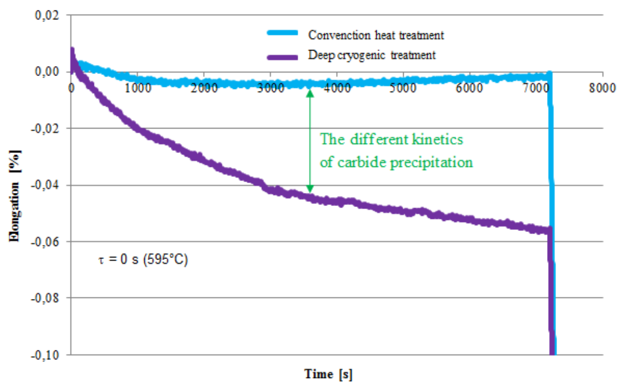

Fig. 5. Influence of cold treatment (-60°C) on carbides precipitation

5. Dilatometry

In order to understand the influence of DCT on carbides precipitation during annealing, dilatometric experiments were performed on quenching dilatometer Linseis RITA L78. For cold treatment performed at -60°C the entire process was recorded and compared to conventional hardening procedure. As Fig. 5 shows, significant influence of cold treatment on the dilatation response in the initial phase of tempering was found during this measurement. For deep cryogenic treatment at -160°C only the tempering process step was analysed because of technical limits of the dilatometer. The achieved results show a significant influence of the holding time at -160°C on the dilatation response during tempering (Fig. 6).

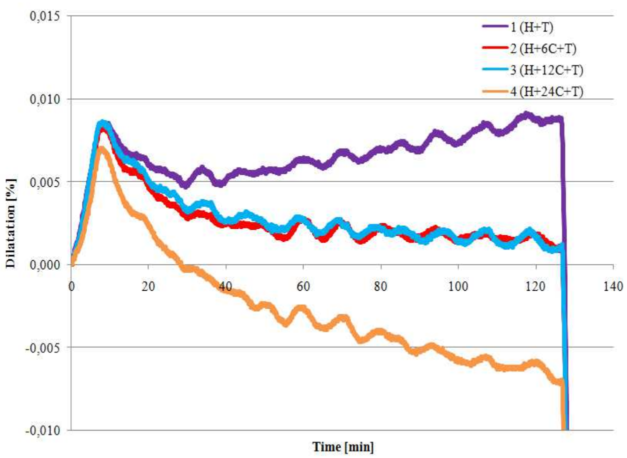

Fig. 6. Influence of deep cryogenic treatment on carbides precipitation

6. Conclusions

Tests carried out in this study have shown that deep cryogenic treatment of the X37CrMoV5-1 (H11) steel 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 deep cryogenic treatment is crucial. The optimum time appears to be 6 hours. Microstructure observation by means of transmission electron microscopy found two types of substructures which are likely to facilitate the precipitation of fine carbides during final tempering of the steel. Dilatometric analyses of the precipitation kinetics of specimens after conventional treatment, cold treatment and deep cryogenic treatment showed a significant effect of both cold and deep cryogenic treatment on the carbides precipitation during tempering.

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