Multistage cryogenic treatment of X153CrMoV12 cold work steel

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Abstract. The article presents the influence of multistage cryogenic treatment (MCT) on the microstructure and properties of X153CrMoV12 cold work steel. Basic mechanical properties of steel, such as hardness and wear resistance (3 rolls-cone system) were tested. The microstructure of steel was observed by means of scanning microscopy. Test results were compared with the properties of steel subjected to conventional heat treatment and deep cryogenic treatment (DCT). The cryogenic processes of X153CrMoV12 steel influenced on the percentage of strengthening phases in the material matrix microstructure. In the case of MCT and DCT, similar changes in the microstructure were observed, consisting in increasing of the amount, more uniform distribution and refinement of carbide phases. Changes in the microstructure of steel affected the mechanical properties of the material.

1 Introduction

Multistage cryogenic treatment (MCT) processes, consisting of cyclic cooling down of the load to cryogenic temperature (e.g. -180°C) and heating it up to a certain temperature (e.g. temperature of -100°C or 20°C), are one of the most promising directions of cryogenic treatment technology development.

There is no data in the literature on the description of the phenomena occurring during this modified variety of cryogenic treatment. In a few articles, mainly of a commercial nature, MCT cryogenic treatment is presented as a favorable sub-zero treatment giving an effect similar to the result obtained during DCT treatment, i.e. mainly increasing the durability of tool steels [1, 2], as well as obtaining favorable changes in the sound of musical instruments [2]. In these marginal literature reports, the authors fail to take economic aspects, as according to the authors of this work, the lower cost of such treatment is very important in comparison with DCT, while maintaining similar effects on elevated mechanical properties of tool steels. There is also no exact data on how to perform this treatment, i.e. the number of repetitions in the cooling / heating cycle and used temperatures. An example treatment scheme is available in [2], but the author does not disclose the details of the heat treatment process (Figure 1).
Notes on MCT treatment can also be found on the websites of commercial contractors for this heat treatment [3, 4]. Due to the commercial nature of the information, this treatment is described quite enthusiastically and in comparison with conventional heat treatment or DCT is referred to as "the best" treatment [4]. The authors predict that the multistage cryogenic treatment may intensify precipitation processes occurring in tool steels. It can be assumed that repeating the process of holding steel at cryogenic temperature and heating it, e.g. to ambient temperature, i.e. severalfold repeating the carbide nucleation stage by forming clusters of carbon atoms occurring in the sequence of strengthening phases, will positively affect the mechanical properties of steel. The assessment of the influence of heat treatment on durability of tools requires a review of the relationship between the microstructure and mechanical properties of materials, such as hardness or wear resistance. The parameters of individual heat treatment operations should be analysed, as well as, the obtained microstructure, with particular emphasis on the content of residual austenite, the morphology of the martensitic matrix and the amount and distribution of carbides of alloying elements.

2 Materials and methods

The X153CrMoV12 cold work tool specimens were subjected to tests. The actual chemical composition of steel is shown in Table 1.

| C   | Si  | Mn  | P      | S   | Cr  | Mo  | Ni  | Al  | Co  | Cu  | V    | W    |
|-----|-----|-----|--------|-----|-----|-----|-----|-----|-----|-----|------|------|
| 1.56| 0.31| 0.51| <0.0019| <0.0020| 11.47| 0.65| 0.36| 0.03| 0.11| 0.10| >0.9624| 0.25 |

The steel samples were austenitized in a vacuum and quenched in nitrogen at a pressure of 4 bar. The tempering processes were carried out under a nitrogen atmosphere. The sub-zero processes was carried out in an industrial cryogenic chamber. In tests a DCT process in temperature at -180 °C and a 24 hour holding time. In the case of MCT processes, the treatment consisted in cooling the charge to -180 °C and subsequent heating and cooling of the steel several times in fixed temperature range (-180°C, -100°C or 20°C). The cryogenic processes were carried out immediately after the hardening operation.

All samples were tested in three modes of heat treatment, presented in Table 2.
Table 2. Parameters of heat treatment.

| Mode          | Austenitizing | Quenching  | Cryogenic treatment       | Tempering |
|---------------|---------------|------------|---------------------------|-----------|
| Q+T           |               |            |                           |           |
| Q+DCT+T       | 1060°C        | Nitrogen, 4 bar | DCT (-180°C, 24h)      | 520°C     |
| Q+MCT-100+T   |               |            | MCT-100 (-180°C/-100°C, 6 cycles) |           |
| Q+MCT20+T     |               |            | MCT 20 (-180°C/20°C, 6 cycles) |           |

Observations of metallographic specimens etched with nital were performed using the JEOL JSM-IT100 scanning electron microscope at magnifications in the range of x2000 ÷ 10000. Residual austenite content tests were carried out using a PSF-3M Rigaku diffractometer. The sin$^2$ψ method and the Cr lamp were used. The HRC surface hardness tests were performed using a Struers DuraScan70 hardness tester, performing 5 readings on each of the samples. Test of resistance to wear was carried out in a 3 rollers-cone system with a continuous lubrication of samples, at constant unit pressure of 100, 200 and 400 MPa. Conical counter-specimen was made of 100Cr6 steel quenched and hardened to the hardness of 64 HRC and ground to a roughness Ra = 0.32 micrometers.

3 Results and discussion

3.1 Microstructure

Microstructure images of X153CrMoV12 steel specimens subjected to heat treatment are shown in Figure 2. Structural imaging of steel subjected to nital etching allowed to determine the size of the original austenite grain and to observe the precipitation of primary carbides and larger precipitations of secondary carbides in the material matrix. The morphology of the matrix visible in the photographs can be described as lath martensite.

The average size of primary grains of austenite for all variants was approx. 10 μm, which corresponds to No. 10 in the ASTM scale. The largest amount of fine-disperse carbides in the form of plaques was observed in the case of a variant including deep cryogenic treatment DCT and multistage cryogenic treatment MCT20 (Figure 2c, 2d). This result is consistent with the observations of the authors of the work [5], in which the content of the carbide phase increased from 18 to 26%. An important difference between cryogenic treated steel and without cryogenic treatment is even distribution of carbides in the case of steel subjected to sub-zero processing, which is consistent with the results described in a number of publications [5, 6, 7, 8, 9].

![](image1.png)  a) Q + T

![](image2.png)  b) Q + MCT-100 + T
3.2 Retained austenite content

The X153CrMoV12 steel was austenitized at 1060°C to ensure high residual austenite content after the hardening operation.

X153CrMoV12 steel samples subjected to hardening contained approximately 26.2% residual austenite, the tempering process reduced this value to 7% (Figure 3). The X153CrMoV12 steel in the standard hardened condition contains up to 35% residual austenite and M7C3 carbides (primary and secondary), Mo (C, N) and MC. In the tempered condition at maximum hardness, strengthening is the result of the presence of M2(C,N) and Mo2C nanocarbides. After conventional heat treatment, more than 10% of residual austenite remains. In the article [5], it was found that the 48-hour process of cryogenic heat treatment process of D2 steel (equivalent of X153CrMoV12 steel) can promote the almost complete conversion of residual austenite into martensite. In this work, D2 steel hardened at 950°C contained 8% of residual austenite, and the cryogenic treatment allowed to lower this content below the detection capacity (> 1% volume).

Figure 3. Retained austenite content of X153CrMoV12 steel after various modes of heat treatment, XRD.

3.3 Hardness

The HRC surface hardness tests (Figure 4) showed that the X153CrMoV12 tool steel in the hardened condition had a hardness of 64.2 HRC. The process of cryogenic treatment of tempered samples made it possible to obtain a much higher hardness (66.7 to 66.9 HRC), which is certainly associated with the
continuation of martensitic transformation at lower temperatures and the formation of a structure with a much lower content of the soft phase component which is residual austenite (Figure 3).

![Figure 4. Hardness of X153CrMoV12 steel after various modes of heat treatment, HRC.](image)

The tempering process of hardened samples (without cryogenic treatment) caused a hardness reduction to 62.5 HRC. For cryogenic treated samples subjected to tempering, the hardness was reduced by even lower values (in the range of 59.5 ÷ 60.5). The shift of the highest hardness peak to lower temperatures and a lower hardness after tempering at a given temperature was observed for the cold working tool steel Vanadis 6 subjected to cryogenic treatment [10]. This fact was related to the faster occurring precipitation phenomena after the cryogenic treatment. The shift to higher temperatures of thermal effects associated with the precipitation processes of cementite [11] or secondary carbides [12] was observed by the authors of the work during calorimetric studies.

### 3.4 Wear test

The results of the steel wear test, in the summarised form of maximum linear wear after 100 minutes of friction test, are presented in Table 3. In all heat treatment variants and at all unit pressures of 100, 200 and 400 MPa, the samples were characterized by the wear with a regular course, manifesting the occurrence of steady friction state in the entire course of wear, except running-in period and final seizure (load of 400 MPa).

| Mode        | Decrease in maximum wear value, % |
|-------------|-----------------------------------|
|             | 100 MPa  | 200 MPa  | 400 MPa  |
| Q+T         | -        | -        | Seizure after 70 min |
| Q+MCT-100+T | 11.4     | 14.6     | Seizure after 70 min |
| Q+MCT20+T   | 22.1     | 30.9     | Seizure after 90 min |
| Q+DCT+T     | 29.0     | 25.7     | Seizure after 100 min |
The most advantageous variant of cryogenic treatment seems to be the DCT process, where the decrease in consumption, depending on the load, ranged from 25.7 to 29.0%. Tests conducted under all unit loads showed that the cryogenic processes caused a decrease in the value of steel wear resistant compared to steel subjected to conventional heat treatment.

4 Summary

The work allowed to state that the processes of cryogenic treatment of the X153CrMoV12 cold work tool steel affect the contribution of strengthening phases in the material matrix microstructure. In the case of multi stage cryogenic treatment and deep cryogenic treatment, similar changes in the microstructure were observed, consisting in increasing the amount, more uniform distribution and refinement of carbide phases. The above changes result from the refinement of the martensite substructure and the precipitation processes occurring in a different manner in the cryogenic treated material. The microstructural changes also included the obvious influence of sub-zero treatment on the content of residual austenite.

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