Impact of semi-solid processing and cryogenic treatment on wear resistance of X210Cr12 tool steel

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Abstract. Semi-solid processing is type of process where metastable microstructure with carbide network may be obtained. Metastable austenite is embedded in carbide-austenite network even in ordinary tool steels. The paper describe semi-solid processing of the ledeburitic steel X210Cr12, with 2% carbon and 12% chromium. The semi-solid processing temperature was 1250 °C with dwell time 60 minutes. Two states for comparison were evaluated, with or without subsequent deformation. Another process used in this study is cryogenic treatment. Cryogenic treatment defined as an add-on process to conventional heat treatment, during which the material is brought to a temperature close to the liquid nitrogen temperature. The cryogenic treatment were used in this study for improving and comparing of impact on wear resistance combined with semi-solid processing. Wear resistance were evaluated based on pin-on-disc testing at ambient temperature.

1 Introduction

It is well known that even commonly used steels may gain unusual microstructures by some types of unconventional processing, thus expanding their field of application. One of the new investigated processes is semi-solid processing. By this process technology metastable microstructure with carbides can be obtained. The microstructure is significant due to their multi-phase structures that form in the part based on different concentration of chemical elements in liquid and solid phases [1-3]. Microstructures produced by semi-solid processing typically consist of polyhedral austenite grains embedded in a ledeburite network. In this state, the microstructure is very brittle. Consequent refinement, disperse or decomposition its fragment uniformly across the part is needed. The final microstructure will be fine-grained and homogeneous, containing a fine dispersion of carbides with appropriate mechanical properties [4, 5].

Tool steels produced by a conventional metallurgical route contain mostly sharp primary carbide of the M-C₃ type, these type of carbides is very difficult to eliminate by conventional processes. Subsequent forming follows semi-solid processing. It can remove sharp-edged chromium carbides by converting them into a very fine dispersion in a martensitic matrix [2-5]. The experimental steel for these experiments is ledeburitic cold work tool steel X210Cr12 [6]. The steel has relatively wide solidification range, appropriate for semi-solid processing [5].

Deep cryogenic treatment (DCT) is defined as an add-on process to conventional heat treatment, with process temperature up to -196 °C, with holding times in range of hours to days. It is generally
used for increasing the hardness, reduction amount of retained austenite, stabilizing martensite for dimensional and geometric stability of the precision parts, to improve wear resistance for most of steel grades and lead to longer lifetime of tools. DCT is in most cases applied between quenching and tempering process [7-9]. DCT leads to several microstructural changes, below -130 °C, retained austenite transforms to martensite same way as in cold treatment (-80 °C). Cooling down to deepest temperature (up to -196 °C) causes decomposition of primary martensite by time-dependent transformation. It resulted in the nucleation of plenty coherent nanometric carbides [10, 11]. These processes are starting during DCT, however the process is done during tempering period. The considerable improvement in wear resistance is supposedly caused by newly-formed carbides [8, 11, 12].

2 Experimental program

The material which was studied in these trials was X210Cr12 tool steel. Chemical composition of experimental steel is shown in Table 1. Experimental specimens with bar shape were 75 mm in diameter and 150 mm in length. The bar with diameter 65 mm from steel X210Cr12 were inserted into containers made of SJ355 low-carbon steel. The containers steel has higher melting point than our experimental steel. Containers were composed of a tube which was sealed with lids on both ends and welded closed. Initial microstructure were in annealed state, the microstructure was compound by large sharp chromium carbides with very fine cementite in ferritic matrix (Figure 1). The hardness was 220 HV10.

| Table 1. Chemical composition of experimental steel (wt. %) |
|------------|------|------|------|------|------|------|------|
| C          | Cr   | Mn   | Si   | Ni   | P    | S    |
| 2.0        | 12.0 | 0.3  | 0.35 | max. 0.5 | max. 0.03 | max. 0.035 |

In this paper, two main production ways were used. All samples were heated to the semi-solid temperature that was choose to 1250 °C. The temperature were determined based on previous research [2-5]. Soaking time was 60 minutes. Subsequent treatment were divided to 2 categories. 1st category was cooling to room temperature in water (samples are labelled as NoDef). 2nd category was included subsequent cooling for period of 35 seconds in water and then heating to forging temperature 1080 °C (recommended forging temperature according data sheet). After 30 minutes of soaking time there were 10× deformation to final thickness (40 mm) performed between flat dies in a hydraulic press. The final process was rapid cooling to room temperature in water (these category is labelled as Def). Scheme of forging is shown in Figure 2. Specimens subjected by semi-solid processing with subsequent cooling or forging were treated identically by different manners of post-processing procedures. In the experimental program were used these processes: tempering at 300 °C for 2 hours (labelled as Def_temp or NoDef_temp) and DCT at -160 °C for 24 hours in variant
with or without tempering at 300 °C for 2 hours (labelled as Def_cryo, NoDef_cryo, Def_c+t and NoDef_c+t).

![Figure 2](image)

**Figure 2.** Scheme of the forging process – visualization of 5× deformation (10× deformation is performed analogously)

### 3 Discussion and Results
Labelling of these specimens as well as its hardness is shown in Table 2. The results shown, that 10× deformation leads to significant growth of hardness values, subsequent DCT leads to increase as well. There is expected increased amount of small carbides leading to higher values of hardness. The effect of additional DCT is more visible for specimens after forging, where is due to deformation refined grain. Polyhedral austenite grains with uniformly disperse ledeburite network is creating during transition by semi-solid state, lower magnification is shown in detail (austenite grains embedded by ledeburite network). Microstructure after forging consist broken ledeburite network and a fine dispersion of chromium carbides and cementite particles combined with martensite-austenite matrix with carbides. Both microstructures examined using optical microscope (Def and NoDef state) we can see on Figure 3.

| Label   | Deformation | Description                               | Hardness HV30 |
|---------|-------------|-------------------------------------------|---------------|
| Def     | 10×         | No other post-processing                   | 780           |
| Def_temp| 10×         | Tempering 300 °C/2 hrs                     | 707           |
| Def_cryo| 10×         | DCT -160 °C/24 hrs                         | 906           |
| Def_c+t | 10×         | DCT -160 °C/24 hrs + tempering 300 °C/2 hrs| 781           |
| NoDef   | Without     | No other post-processing                   | 388           |
| NoDef_temp| Without    | Tempering 300 °C/2 hrs                     | 386           |
| NoDef_cryo| Without    | DCT -160 °C/24 hrs                         | 402           |
| NoDef_c+t| Without    | DCT -160 °C/24 hrs + tempering 300 °C/2 hrs| 402           |

![Figure 3](image)

**Figure 3.** Microstructure after semi-solid processing – NoDef (left) and after forging – Def (right)
3.1 Wear resistance
Deep cryogenic treatment is typically used for expected improvement in wear resistance. For testing of wear resistance were used method pin-on-disc. The principle of pin-on-disc test is forcing a ball into the surface of a rotating flat specimen. The ball indenter was pressed by a defined force (exerted by a weight) against the specimen. Testing was conducted at ambient temperature. Each testing for each manner of production were done 2× for each sequence. The test surfaces were grinded to hardness approx. with Ra (Arithmetical mean height) 0.5 µm. The testing device was high temperature tribometer by company CSM Instruments (Switzerland).

The test parameters for all specimens were as follows:
- 6 mm diameter ball indenter (Al₂O₃)
- 15 N load
- 240 rpm
- 1.5 mm radius of wear track
- ambient temperature
- 15000 cycles / 141.375 m (number of cycles / path travelled by the ball indenter)

The wear rate was calculated from the measured and selected test data using (1). The wear track volume was evaluated using a contact profilometer. Its value was found as the mean from 8 readings around the track. The results show difference between state after deformation (Def) and state without deformation (NoDef). The main aspect is microstructure of tested specimens, which correspond to the hardness of investigated sequences. Sequences with deformation has finer microstructure and approx. double hardness values. It is main reason why the wear rate is lower for these sequence (Figure 4). Percentage difference is approx. 32 % between specimens labelled as Def and NoDef. When we compare states without any other post-processing, the difference is approx. 45 %. The results of wear rate after additional processes (tempering, DCT or DCT with tempering) lead to different behaviour depending on the initial microstructure. In case of specimens after forging process, all additional processes lead to increase of wear rate. The specimen after DCT has lower wear rate comparing with sequences with tempering. Specimens without deformation with conducted semi-solid treatment has higher or equal values of wear rate comparing with other additional processes. Tempering leads to positive decrease of wear due to presence of newly created fine carbides around the polyhedral austenite grains. The amount of specimens / repetition is not enough for obtaining significant statistical file. The results showed the presumably tendencies of wear rates.

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

(1)

![Figure 4. Results of wear rate, sequences Def and NoDef with additional processes](image)

Wear tracks were analysis by optical microscope. Comparison of state Def vs. NoDef is shown in Chyba! Nenalezen zdroj odkazů.. The detail of worn surfaces shows the width of wear track. For deformed specimen (Def) the width is 507 µm in maximum, the state after semi-solid processing (NoDef) has width up to 610 µm. Whole wear tracks are shown on left side in Figure 5.
The average wear track depths for forged specimens (Def) was approx. 8 µm, for states after semi-solid processing (NoDef) it was approx. 11 µm.

![Figure 5. Comparison of wear tracks width - Def (upper) and NoDef (bottom)](image)

4 Conclusion
Experimental semi-solid processing with or without subsequent forging was carried out on X210Cr12 cold work tool steel. Forging was conducted with 10× deformation. The both types of specimens were treated identically by different manners of post-processing processes. In the paper is presented state after tempering, cryogenic treatment and after cryogenic treatment with subsequent tempering. Specimens including covering container with 75 mm in diameter and 150 mm in length were processed at semi-solid temperature 1250 °C, subsequent forging temperature was 1080 °C.

The hardness measured after performed procedures show significant difference between specimens with or without forging (Def or NoDef). The hardness is approx. double higher after forging. The hardness in state after no post-processing is 780 HV30 (Def), after semi-solid the hardness is 388 HV30 (NoDef). Cryogenic treatment applied as additional process leads to increase of hardness values. Forging state with DCT had hardness up to 906 HV30 (Def_cryo), semi-solid state after DCT had 402 HV30 (NoDef_cryo). Wear resistance of treated specimens were executed by pin-on-disk method at ambient temperature. Highest resistance was measured on specimen after forging without additional post-processing, comparing to process without forging and any additional process the increase in resistance (wear rate) is approx. 45 %. When we compare both performed procedures and their wear rates (including additional process) the percentage difference is approx. 32 %. Metallography of wear tracks shows wider track for specimen in state just after semi-solid processing (NoDef), the dimension difference is about 100 µm (after forging (Def) it is approx. 500 µm, after semi-solid processing (NoDef) it is approx. 600 µm). The average wear track depths for forged specimens (Def) was approx. 8 µm, for states after semi-solid processing (NoDef) it was approx. 11 µm.

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