

# EFFECT OF THE COOLING RATE AFTER SEMI-SOLID PROCESSING ON THE RESULTING MICROSTRUCTURE IN HIGH-CHROMIUM TOOL STEEL

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#### Abstract

Sharp-edged carbides in high-alloy tool steels reduce their toughness appreciably. Since they form during solidification, they are virtually impossible to remove by conventional routes. One of the techniques available to meet this purpose involves thermomechanical treatment with transition through the semi-solid state. It relies on using temperatures above the solidus, thereby re-dissolving the carbides in the austenitic matrix. The resulting structure consists of polyhedral austenite grains embedded in lamellar austenite-carbide network. Using appropriate forming parameters, this network can be removed by thermomechanical treatment.

An experiment was carried out on X210Cr12 steel, combining semi-solid processing and subsequent thermomechanical treatment in a press. Hot plastic deformation led to re-dissolution of carbides from the eutectic network to the matrix. The rate of cooling after deformation played a major role in the evolution of the resulting microstructure. It was found that by varying the cooling rate one can obtain a broad range of hardness values. Water quenching led to fine-grained structures consisting of the M-A constituent and fine chromium carbides whose hardness exceeded 860 HV10. By contrast, very slow cooling caused pearlite to form, producing structures with a hardness of 230 HV10. The process represents an alternative to known and well-established treatment routes which are normally used for tool steels with chromium carbides.

Keywords: Semi-solid processing, carbide network, cooling rate, X210Cr12

## 1. INTRODUCTION

Mechanical properties and behaviour of tool steels are given, to a great extent, by the types, shapes, sizes, distribution and amount of carbides they contain [1]. Carbides which remain stable even during austenitizing greatly improve wear resistance but also reduce toughness, particularly when they are too large and their distribution is non-uniform [1, 2]. Carbide formers, such as tungsten, molybdenum, chromium and vanadium have a major effect on the size of primary carbides.

One method for removing primary carbides involves short-time conversion to semi-solid state. As the material is heated near the liquidus temperature, carbides dissolve into austenitic matrix which thus becomes enriched with alloying elements. While the material is in the semi-solid state, globular austenite particles may form in the liquid phase. This fact and the thixotropic behaviour can be useful for shaping difficult-to-form materials [3-5]. Hence, products with intricate shapes can be manufactured in a single forming step. An important aspect is the choice of the processing temperature which controls the liquid fraction and the rheological behaviour of the material as well as the resulting microstructure [6]. Steels upon semi-solid processing often contain polyhedral austenite grains embedded in a eutectic network [7-9].

Even though austenite is ductile, the carbide network cannot sustain plastic deformation of sufficient magnitude at room temperature. However, it has been experimentally found that these carbides can be redistributed uniformly in an austenitic matrix, provided that deformation of appropriate magnitude and intensity is applied. Such carbides can contribute to strength and can pin austenite grain boundaries against growth during hot





deformation. Deformation and elevated temperature may also cause these carbides to partially dissolve in austenite. After reprecipitation, they can contribute to the strength of the matrix. To achieve optimal properties, the matrix can be altered by additional heat treatment, such as quenching and tempering. By varying the cooling rate, one may obtain mixed microstructures with various hardness levels [6, 10].

## 2. EXPERIMENTAL PROGRAMME

One of the key parameters of a route that combines short-time conversion to semi-solid state and subsequent thermomechanical treatment is the choice of the post-deformation cooling rate. Varying this parameter can lead to mixed microstructures containing both martensite and pearlite, and thus to various mechanical properties. A typical example of tool steels with sharp-edged  $M_7C_3$  chromium carbides is X210Cr12 steel.

#### 2.1. Experimental material

X210Cr12 is a hyper-eutectoid chromium steel with high hardenability (**Table 1**). It possesses good wear resistance, cutting performance and high compressive strength. When produced by the conventional metallurgical route, it is characterized by poor toughness due to carbide banding [11]. It is used for cold-shearing tools, forming tools and extrusion tooling. In addition, it is a suitable material for small durable dies for shaping plastic and powder materials, glass, porcelain and ceramics.

С	Cr	Mn	Si	Ni	Р	S
1.8	11	0.2	0.2	0.5	0.03	0.035

Table 1 Chemical composition of X210Cr12 steel [wt. %]

This experimental material in its initial annealed condition contained large sharp-edged primary chromium carbides and very fine cementite in a ferritic matrix with a hardness of 220 HV10 (**Figure 1**). Approximate values of transformation temperatures were calculated using the JMatPro software [13]. According to the calculations melting starts at 1225 °C and ends at 1373 °C. At 1255 °C, the primary chromium carbides dissolve. After semi-solid processing, the steel consists of 96 % austenite in the form of globular grains and a ledeburite network (**Figure 2**), [7].



Figure 1 Initial microstructure of X210Cr12 Figure 2 Micrograph upon semisolid processing

Figure 3 Forging in a press, first blow

## 2.2. Processing in a press

In order to introduce the deformation necessary to break up lamellar ledeburite network and initiate austenite recrystallization, hot working in a press was used (**Figure 3**). Because the material becomes partially melted



in the process, the billet had been encased in a low-carbon steel container. This simplified the handling, reduced the formation of scale and made the temperature field more homogeneous. The container with a diameter of 30 mm, wall thickness of 6 mm, and length of 55 mm was made of SJ355 low-carbon steel whose melting temperature was above 1400 °C. Heating was carried out in an air furnace. Flat dies were used for the forming operation. A total of 7 different procedures were carried out (**Table 2**). The optimum heating temperature identified in earlier experiments was 1240 °C [7, 8].

Soaking at 1240 °C took 15 minutes. According to a preliminary calculation using JMatPro, the material at this temperature should contain 10 % liquid melt. In addition, this temperature is near the temperature of complete dissolution of primary chromium carbides. Nevertheless, their fraction is still approximately 7 % at this temperature. Soaking was followed by quenching in water for 3 seconds and reheating to the forming temperature of 1080 °C and holding for 1.5 minutes. Five alternating upsetting and drawing out reductions were carried out in the press. Each forming step involved a reduction by 20 mm. The aggregate forging ratio in each step was about 1.6.

Forging was followed by cooling in various media: water quenching (procedure 1), cooling in still air (procedure 2), quenching in oil at 80 °C (procedure 3) a cooling in a closed furnace (procedure 4). In procedure 5, water quenching was followed by tempering at 250 °C for 60 minutes (**Table 2**). Procedure 6 involved no deformation in order to allow the effect of deformation on microstructural evolution to be characterized. The effects of repeated dynamic recrystallization on microstructural evolution and grain refinement were studied using procedure 7. The workpiece was reheated to the forming temperature of 1080 °C between deformation steps.

Microstructure characterization was carried out using optical and scanning electron microscopes. The distribution of chromium was mapped by EDX, and microstructure constituents identified using EBSD. Mechanical properties were measured by HV10 hardness testing.

Procedure no.	Heating temperature[°C]/ Holding time [min]	Reheating temperature [°C]/ Holding time [min]	No. of forming steps [-]	Type of cooling	HV10 [-]	
1				Water	864	
2		1080 / 1.5	5	Still air	331	
3				Oil 80°C	778	
4				Closed furnace	229	
5	1240 / 15			Oil 80 °C - Tempering 250 °C / 1 hour – Still air	677	
6			0	Water	388	
7		1080 °C / 1.5 min. – 3 forming steps – 1080 °C / 1.5 min. – 3 forming steps 1080 °C /1.5 min. – 3 forming steps – water				

Table 2 Parameters of thermomechanical treatment procedures

## 3. RESULTS AND DISCUSSION

Microstructure was observed on longitudinal metallographic sections through the billet centre, i.e. the region which was subjected to the largest deformation. Procedure 1 (heating at 1240 °C, five deformation steps and water quenching) led to a very fine microstructure with a hardness of 864 HV10 (**Table 2**). The matrix consisted of recrystallized austenite grains with sizes of 0.5 to 1  $\mu$ m, which transformed to martensite during cooling (**Figures 4, 5**). Using X-ray diffraction analysis, this material was found to contain 17 % retained austenite. Chromium carbides precipitates were found in the matrix as well. Thanks to the presence of the liquid phase



after heating, the microstructure contained distinct remnants of the carbide network along prior austenite grain boundaries. Most of this network was fragmented. Primary chromium carbides were almost completely removed. EBSD analysis confirmed that the matrix consists predominantly of martensite and Cr<sub>7</sub>C<sub>3</sub> carbides.



Figure 4 Procedure 1 - Martensitic-austenitic matrix with fragmented ledeburite network and fine chromium carbides precipitates



Figure 5 Procedure 1 - Detail of carbides in the fragmented ledeburite network in fine recrystallized austenite grains that transformed to martensite

Procedure 2 (cooling in still air) led to a notably different microstructure. As it involved longer cooling time, a portion of austenite decomposed into very fine pearlite, both lamellar and spheroidised (**Figure 6**). The matrix consisted of fine recrystallized ferrite grains and contained very fine secondary chromium carbides precipitates. All these features were reflected in the resulting hardness of 331 HV10 which was lower than in the previous procedure (864 HV10). The eutectic network which had formed due to the presence of the liquid phase was substantially broken-up.



**Figure 6** Procedure 2 – Ferritepearlite structure with fine chromium carbides precipitates **Figure 7** Procedure 3 – Recrystallized grains of M-A constituent, fine Cr carbides

**Figure 8** Procedure 4 - Ferritic matrix with chromium carbides and spheroidised cementite

Procedure 3 (oil quenching) involved a cooling rate which was higher than in still air, which is why no pearlite was found in the microstructure (**Figure 7**). The type of microstructure is very similar to the specimen which was quenched in water. However, its hardness was lower: 778 HV10 (**Table 2**).



The largest difference from the other workpieces was found in the workpiece which cooled in a closed furnace (procedure 4). Its matrix was ferritic with some pearlite (**Figure 8**). Pearlite was mostly globular and only small amount of lamellar pearlite was found. Due to long cooling time, chromium carbides had coarsened. The hardness was 229 HV10. The procedure which involved water quenching and subsequent tempering at 250 °C for 1 hour led to a very fine microstructure with chromium carbides precipitates. Here, the matrix consisted of prior austenite which transformed to martensite and showed strong effects of tempering. Ferrite was found as well (**Figure 9**). However, even the long tempering time did not cause any grain coarsening. The hardness of 677 HV10 matched the hardness value obtained upon tempering of conventionally-treated steel [11].

Procedure 6 (short-time conversion to semi-solid state, reheating to 1080 °C and water quenching without any plastic deformation) led to a microstructure which is typical of semi-solid processing. Polyhedral austenite grains with a size of approximately 50  $\mu$ m were embedded in a lamellar network which consisted of a mixture of austenite and carbides (**Figure 10**). Fine precipitates of chromium carbides did not form and austenite did not transform to martensite, which is why the resulting hardness was low: 388 HV10. Even reheating to 1080 °C has not led to austenite refinement.



Figure 9 Procedure 5 – Tempered martensite microstructure with Cr carbides precipitates Figure 10 Procedure 6 -Polyhedral austenite grains embedded in a lamellar ledeburite network Figure 11 Procedure 7 -Recrystallized austenite grains after transformation to martensite, chromium carbides precipitates

The effect of repeated recrystallization was studied using procedure 7 which involved three-step deformation and reheating to the forming temperature of 1080 °C. The procedure produced a matrix of recrystallized austenite grains which were partially transformed to martensite. Chromium carbides precipitates were globular, mostly located along prior austenite grain boundaries (**Figure 11**). This procedure led to the highest hardness: 899 HV10.

# 4. CONCLUSION

A process for removing primary chromium carbides was carried out on X210Cr12 steel with the use of unconventional thermomechanical treatment routes. These primary chromium carbides form during solidification and are impossible to remove by standard heat treatment routes. Therefore, a new route with short-time conversion to semi-solid state was developed. It causes chromium carbides to re-dissolve into austenitic matrix, and the appropriately-chosen deformation parameters lead to fragmentation of the lamellar ledeburite network. Concurrently, dynamic recrystallization of austenite and precipitation of chromium carbides take place. In this case, great attention was paid to the rate of cooling from the deformation temperature to



room temperature. It was found that final quenching in water produces a very fine structure with grains of the M-A constituent of  $1\mu$ m size and with very fine chromium carbides precipitates, which has a hardness of 864 HV10. At the same time, the ledeburite network that had formed due to the presence of liquid metal was broken up to a great extent. Slower cooling rates led to pearlite. Several-hour cooling in a closed furnace led to cementite spheroidisation and a drop in hardness to 229 HV10. It was confirmed by this experimental programme that in order to refine prior austenite grain, initiate dynamic recrystallization and promote chromium carbides precipitation, it is necessary to introduce plastic deformation in a two-phase region.

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#### REFERENCES

- [1] PAN, F. et al. Phase transformation refinement of coarse primary carbides in M2 high speed steel. *Progress in Natural Science: Materials International*, 2011, vol. 21, no. 2, pp. 180-186.
- [2] WANG, J., et al. Plastic deformation behaviors and hardening mechanism of M<sub>7</sub>C<sub>3</sub> carbide. *Materials Science and Engineering A*, 2016, vol. 662, pp. 88-94.
- [3] RASSILI, A., ATKINSON, H. V. A review on steel thixoforning. *Trans. Nonferrous Met. Soc. China*, 2010, vol. 20, pp. 1048-1054.
- [4] KOPP, R., KALLWEIT, J., MÖLLER, T., SEIDL, I. Forming and joining of commercial steel grades in the semi-solid state. *Journal of Materials Processing Technology*, 2002, vol. 130-131, pp. 562-568.
- [5] CEZARD, P., SOURMAIL, T. Thixoforming of Steel: A State of the Art from an Industrial Point of View. *Solid State Phnenomena*, 2008, vol. 141-143, pp. 25-33.
- [6] PÜTTGEN, W., B. HALLSTEDT, B., BLECK, W., UGGOWITZE, P. J. On the microstructure formation in chromium steels rapidly cooled from the semi-solid state. *Acta Materialia*, 2007, vol. 55, pp. 1033–1042.
- [7] AIŠMAN, D., JIRKOVÁ, H., KUČEROVÁ, L., MAŠEK, B. Metastable structure of austenite base obtained by rapid solidification in a semi-solid state. *Journal of Alloys and Compounds*, 2011, vol. 509, pp. S312–S315.
- [8] AIŠMAN, D., JIRKOVÁ, H., MAŠEK, B. The influence of deformation and cooling parameters after transition through semi-solid state on structure development of ledeburite steel. *Journal of Alloys and Compounds*, 2012, vol. 536, pp. S204-S7.
- [9] ROGAL, L., DUTKIEWICZ, J., SZKLARZ, Z., KRAWIEC, H., KOT, M., ZIMOWSKI, S. Mechanical properties and corrosion resistance of steel X210CrW12 after semi-solid processing and heat treatment. 2014, *Materials Characterization, vol.* 88, pp. 100-110.
- [10] UHLENHAUT, D. I., KRADOLFER, J., PÜTTGEN, W., J.F. LÖ ER, J. F., UGGOWITZER, P. J. Structure and properties of a hypoeutectic chromium steel processed in the semi-solid state. *Acta Materialia*, 2006, vol. 54, pp. 2727-2734.
- [11] <u>http://www.jkz.cz/cs/produkty/nastrojove-oceli/pro-prace-za-studena/w-nr-12080/</u>, 25 Apr. 2017 JMatPro, Release 9.0, Sente Software Ltd., 2016