Semi-solid processing of high-chromium tool steel to obtain microstructures without carbide network

H Jirková¹, D Aíšman¹, K Rubešová¹, K Opatová² and B Mašek¹

University of West Bohemia, Research Centre of Forming Technology, Univerzitní 8, 306 14 Pilsen, Czech Republic

University of West Bohemia, Regional Technological Institute, Univerzitní 8, 306 14 Pilsen, Czech Republic

E-mail: hstankov@kmm.zcu.cz

Abstract. Treatment of high-alloy tool steels that involves transition to the semi-solid state can transform the sharp-edged primary carbides which usually form during solidification. These carbides severely impair toughness and are virtually impossible to eliminate by conventional treatment routes. Upon classical semi-solid processing which dissolves these carbides, the resulting microstructure consists of polyhedral and super-saturated austenite embedded in lamellar austenite-carbide network. This type of microstructure reflects in the mechanical properties, predominantly in material behaviour under tensile loading. Such a network, however, can be removed by appropriate thermomechanical treatment.

In the present experiment, various procedures involving heating to the semi-solid state were tested on X210Cr12 tool steel. The feedstock was heated to the temperature range of 1220 – 1280 °C. The heating was followed by procedures involving either water quenching to the forming temperature, room temperature or temperature from the range from 500 °C to 1000 °C followed by reheating to the forming temperature. It was found that the development of the lamellar network strongly depends on the temperature of heating to semi-solid state. Thermomechanical treatment produced microstructures in which the matrix consisted of a mixture of polyhedral austenite grains and M-A constituent. In addition, the initial lamellar eutectic network was partially or even completely melted and substituted with a mixture of very fine recrystallized austenite grains and precipitates of chromium carbides. Some fine M₇C₃ carbides were present in the austenitic-martensitic matrix as well. When appropriate processing parameters were chosen, very good mechanical properties were obtained, among them a hardness of 860 HV10.

1 Introduction

The high-alloy tool steels of the X210Cr12-type produced by conventional metallurgy contain large sharp-edged M₇C₃ carbides which remain stable even at high temperatures [1]. Consequently, there is virtually no way of to modify these carbides by means of conventional heat treatment to a more favourable morphology, i. e. to the microstructure with finer and more uniformly dispersed carbides. Since large sharp-edged primary carbides impair toughness, this kind of steels must be produced by powder metallurgy which can eliminate the risk of formation of large chromium carbides.

Primary M₇C₃ chromium carbides can dissolve at temperatures above the solidus. Therefore, a method based on semi-solid-processing has been proposed for transition of these carbides leading to the microstructure refinement of high-alloyed tool steels. During semi-solid processing, the material
exists as a mixture of liquid and solid phases. If the material in the semi-solid state exhibits thixotropy, it can be shaped by thixoforming. Thixoforming is a technique that can produce intricate-shape parts using a single forming cycle [4]. During thixoforming, microstructures characterized by polyhedral islands of super-saturated austenite surrounded by lamellar austenite-carbide-eutectics are developed [2, 3]. Hence, no large sharp-edged primary carbides remain in the structure. In these structures, austenite exhibits extraordinary thermal stability. Its thermal decomposition begins only at temperatures higher than 500 °C. The decomposition finishes when the material is annealed at 550–600 °C [5]. Austenite itself is ductile but the carbide network cannot sustain sufficient plastic deformation at room temperature. Nevertheless, these structures can be formed successfully by compressive deformation, with the achievable plastic deformation being no less than 35%. It has been verified experimentally that they can also be formed at high temperatures between 1000 °C and the solidus temperature [6, 7]. At an appropriate temperature, the austenite-carbide network can be broken up by forming. With the right amount and intensity of deformation, carbides can be dispersed uniformly throughout the austenitic matrix. After cooling, these carbides can remain dispersed, and contribute to the strength of the resulting structure. In addition, they prevent austenite grain growth upon high-temperature deformation. Applied deformation and temperature can also cause partial dissolution of these carbides. After reprecipitation, they can contribute to the strengthening of the matrix. To achieve optimal properties, the matrix can be altered by additional heat treatment, such as quenching and tempering or even quenching and partitioning of carbon. If mechanical working finishes under appropriate conditions, a structure with fine martensite is obtained.

2 Experimental programme
To obtain carbide precipitates with an optimal distribution, sizes, and morphology, and microstructures with optimal austenite grain sizes and martensite volume fractions, it is necessary to optimize a whole range of treatment parameters, starting with the temperature of heating into the semi-solid state. The X210Cr12 tool steel was selected for this pilot experimental programme.

2.1 Experimental material
X210Cr12 is a high-carbon and high-chromium steel (table 1). It has been developed for applications in punching machines and presses, mainly for heavy-duty punches and highly-complex progressive and combination tools. The initial annealed microstructure contained large sharp-edged primary chromium carbides and very fine cementite embedded in a ferritic matrix.

| Table 1. Chemical composition of X210Cr12 steel [wt. %]. |
|----------------|----------------|----------------|----------------|----------------|----------------|
| C   | Cr   | Mn | Si | Ni  | P   | S   |
| 1.8 | 11   | 0.2 | 0.2 | 0.5 | 0.03 | 0.035 |

Before definition of processing parameters, it was necessary to identify the freezing range and the dissolution temperature of chromium carbides. To this end, an approximate calculation using the JMatPro software was carried out [8]. It was found that up to 758 °C, the material retains a stable ferrite-cementite microstructure. It begins to melt at 1225 °C and finishes melting at 1373 °C. According to computation, the primary chromium carbides finish to dissolve at 1255 °C.

When the material is heated into the semi-solid state and subsequently cooled, its microstructure changes strongly. The resulting microstructure consists of grains of super-saturated austenite embedded in a lamellar ledeburitic network. This network comprises austenite and secondary M23C6 carbides.

2.2 Thermomechanical treatment
To break up the lamellar network and initiate recrystallization, the material had to be worked using a large amount of deformation. In the present case, open-die forging in a hydraulic press was used. As the material was to be heated into the semi-solid state, the workpiece was enclosed in a case made of low-
carbon steel. This simplified the handling of the partially melted material and made its temperature field more homogeneous. The case of 30 mm diameter, 6 mm wall thickness, and 55 mm length was made of the SJ355 low-carbon steel with the melting temperature above 1400 °C. The workpieces were heated in an air furnace. Flat dies were used for the forming operation. A total of 10 different treatment procedures were carried out (Table 2).

Table 2. Parameters of thermomechanical treatment procedures.

| Procedure no. | Heating temperature [°C] | Holding time [min] | Cooling temperature [°C] | Reheating temperature [°C] | Holding time at reheating temp.[min] | No. of forming steps [-] | HV10 [-] |
|---------------|--------------------------|--------------------|--------------------------|----------------------------|------------------------------------|-------------------------|---------|
| 1             | 1265                     | 15                 | 500                      | 1050                       | 5                                  | 1                       | 520     |
| 2             | 1265                     | 15                 | 500                      | 1100                       | 5                                  | 1                       | 487     |
| 3             | 1265                     | 15                 | RT                       | 1050                       | 12                                 | 1                       | 520     |
| 4             | 1220                     | 15                 | 600                      | 1050                       | 6                                  | 3                       | 788     |
| 5             | 1220                     | 15                 | 1100                     | –                          | –                                  | 4                       | 803     |
| 6             | 1225                     | 60                 | 900                      | 1080                       | 1.5                                | 5                       | 836     |
| 7             | 1200                     | 15                 | 1000                     | 1070                       | 2                                  | 4                       | 848     |
| 8             | 1240                     | 15                 | 900                      | 1080                       | 1.5                                | 5                       | 864     |
| 9             | 1240                     | 60                 | 900                      | 1080                       | 1.5                                | 5                       | 855     |
| 10            | 1280                     | 16                 | 900                      | 1080                       | 1.5                                | 5                       | 866     |

Procedures 1 through 3 involved the heating temperature of 1265 °C with a holding time of 15 minutes. This temperature had been tested in earlier experiments conducted in a thermomechanical simulator [3]. According to the calculations carried out using the JMatPro software, all primary chromium carbides should be dissolved, and the structure should consist of the melt and austenite at this temperature. The calculated proportion of the melt was 30 %. The treatment variants included quenching in water to the temperature of 500 °C (procedures 1 and 2) and to the room temperature (procedure 3), followed by material reheating to the forming temperature, either 1050 °C or 1110 °C, and holding for 5 minutes. The specimens were upset to a half height in a single operation.

Subsequently, a reduced heating temperature of 1220 °C was tested. It is just below the calculated solidus temperature. At this temperature, the microstructure still contains about 8 % of M₂C₃ carbides. In procedure 4, quenching to the temperature of 600 °C was followed by reheating in a furnace to the forming temperature of 1050 °C. The specimen was first upset to a half height, then drawn-out to 50 mm and then upset again all the way to the height of 20 mm. Another variant (procedure 5) had the same heating temperature but involved cooling to no less than 1100 °C followed by forming: upsetting – drawing-out – upsetting – drawing-out. The forming procedure finished at the temperature below 800 °C. In order to explore the effects of the holding time, the procedure 6 had the holding time extended to 60 minutes. The aim was to ascertain whether the austenite grains coarsen, whether coarser grains affect the morphology of recrystallized grains after forming, and whether primary chromium carbides dissolve in any greater extent.

In another variant (procedure 7), the heating temperature was reduced even more, i. e. to 1200 °C. At this temperature, the material is not expected to contain any liquid phase, and therefore the effects of liquid fraction on microstructural evolution from previous experiments can be compared. Upon heating, the microstructure consisted of a mixture of austenite and 9 wt. % of carbides according to JMatPro calculation. The heating temperature of 1240 °C was combined with the holding times of 15 min (procedure 8), and 60 min (procedure 9). This temperature approaches the limit, at which primary chromium carbide are fully dissolved. Nevertheless, their fraction is still approximately 7 wt. % at this temperature. The temperature at the end of cooling was 900 °C, after which the material was reheated to the forming temperature of 1080 °C.
In order to be able to describe the impact of a larger liquid fraction on the microstructure evolution, procedure 10 was designed with the highest heating temperature of 1280 °C. It was expected to lead to complete dissolution of carbides and even to partial melting of austenite. According to calculations, the liquid fraction is 35%.

The microstructures were examined for their homogeneity using optical microscopy. More detailed analysis of microstructure was performed in a scanning electron microscope. The distribution of chromium was mapped by EDX, and microstructure constituents identified using EBSD. Mechanical properties were measured by HV10 hardness testing.

3 Results and discussion

Procedure 1 that involved the heating temperature of 1265 °C and a single upsetting operation led to a strongly non-uniform microstructure with a distinct diagonal cross pattern on the cross-section through the specimen. As expected, in the locations which had not been strongly affected by deformation a structure of polyhedral austenite grains embedded in a lamellar ledeburitic network was developed (figure 1). The location of the largest strain exhibited a heavily distorted structure in which austenite grains were elongated, and the austenite-carbide network was somewhat fragmented but still enveloping isolated austenite grains. There were very fine recrystallized austenite grains within the network (figure 2). Lamellar structure was found in some locations. The hardness was 520 HV10. Another slight increase in the forming temperature, from 1050 °C to 1100 °C, caused no considerable changes in the microstructure evolution.

After cooling all the way down to room temperature and reheating to the forming temperature of 1050 °C, the region along the axis of the specimen exhibited effects of recrystallization in austenite grains and formation of slip bands and subgrains (figure 3). The character of lamellar network was preserved in some areas.

If the heating temperature was reduced to 1220 °C and 1225 °C, i.e. just below the solidus curve, the primary chromium carbides did not dissolve (figure 4). The character of the microstructure depended on the subsequent thermomechanical treatment procedure. Unlike in the first three variants, there was a greater deformation intensity because the forming involved between 3 and 5 reductions. Quenching in water to the temperature of 600 °C and subsequent reheating to the forming temperature of 1050 °C (procedure 4) had produced a matrix consisting of a mixture of fine recrystallized austenite grains which transformed to martensite, resulting in the M-A constituent. This was only visible using large magnifications which confirmed the presence of martensite (figure 5). The units of the M-A constituent had an approximate size of 1 μm, with some units sized up to 3 μm. This matrix contained not only primary chromium carbides but also secondary chromium carbides present between the units of the M-A constituent. Since no melting had occurred during
heating, no ledeburite network formed. All these factors were reflected in the increase in hardness to 788 HV10 (table 2). When cooling took place to no less than 1100 °C and immediately followed by forming without any reheating, the character was similar to variant 5. The larger amount of deformation was another factor why the fraction of unrecrystallized austenite grains was smaller. The hardness was 803 HV10. When the heating time was extended from 15 minutes to 60 minutes (procedure 6), there was no considerable change in the morphology of primary chromium carbides. The microstructure configuration was therefore very similar. Nevertheless, the hardness increased to 836 HV10. A further reduction in the heating temperature to 1200 °C (procedure 7) led to no considerable microstructural changes. The hardness was 848 HV10.

The experimental research continued by increasing the heating temperature to 1240 °C. Two procedures were tested with the only difference in the heating time (procedures 8 and 9). Thanks to five-step deformation, the resulting microstructure was very fine. The matrix consisted of recrystallized austenite grains which had mostly transformed to martensite and had the form of the M-A constituent. Their mean size was between 0.5 and 1 μm (figure 6). Chromium carbide precipitates were found in the matrix. Thanks to the presence of liquid phase after heating, the microstructure contained distinct remnants of carbide network along prior austenite grain boundaries. This network was mostly fragmented. EBSD analysis confirmed that the matrix consists predominantly of martensite and Cr7C3 carbides. The hardness was 864 HV10. The extension the
holding time from 15 minutes to 60 minutes caused no considerable changes in the microstructure. The hardness was 855 HV10.

Thanks to a higher amount of melted phase present at the heating temperature of 1280 °C (procedure 10), there was a larger fraction of the ledeburite network. Its removal by forming was not as successful as in the previous cases (figure 7). Despite that, a very high hardness of 866 HV10 was obtained, thanks to a very fine austenitic-martensitic matrix with chromium carbide precipitates.

4 Conclusion

An unconventional thermomechanical treatment route was used to remove large sharp-edged primary chromium carbides in the X210Cr12 steel. These normally form during solidification at the metallurgical stage of production, and it is impossible to remove them by conventional heat treatment. The underlying principle was to use material heating to the semi-solid state to transform the initial microstructure to polyhedral austenite embedded in carbide-austenite network, and then use forming to break up this network and thus produce a fine microstructure of the M-A constituent and fine chromium carbide precipitates. The semi-solid processing stage was necessary in order to achieve the temperature at which the primary sharp-edged chromium carbides dissolve. By this means, the carbides can be converted to an austenitic-carbidic structure which can be hot-formed. Using plastic deformation, the carbide network can be broken up, and the carbides can be uniformly dispersed.

The results showed that the temperature of 1240 °C must be exceeded in order to obtain sufficient amount of dissolved primary chromium carbides. When lower heating temperatures were used, i.e. 1220 °C and 1200 °C, the microstructure became much finer thanks to dynamic recrystallization of austenite, high hardness was achieved, but the primary chromium carbides remained. They can be melted completely at heating temperatures higher than 1255 °C followed by multi-step deformation below the solidus temperature. Such a procedure can lead to microstructures in which a matrix of the M-A constituent prevails, the prior austenite grains are smaller than 1 μm, and fine chromium carbide precipitates are present. The hardness of such structures exceeds 850 HV10.

Acknowledgement

This paper includes results achieved within the project SGS-2015-028 Semi-solid Processing and New Structures without Carbide Net. The project is subsidised from specific resources of the state budget for research and development.

References

[1] Wang J, Guo W, Sun H, Li H, Gou H, Zhang J 2016 Plastic deformation behaviours and hardening mechanism of M7C3 carbide Materials Science and Engineering A 662 88-94
[2] Rassili A, Atkinson H V 2010 A review on steel thixoforming Trans. Nonferrous Met. Soc. China 20 1048-54
[3] Mašek B, Aišman D, Behulová M, Jirková H 2010 Structure of miniature components from steel produced by forming in semi-solid state Trans. Nonferrous Met. Soc. China 20 1037-41
[4] Rogal L, Dukiewicz J, Szklarz Z, Krawiec H, Kot M, Zimowski S 2014 Mechanical properties and corrosion resistance of steel X210CrW12 after semi-solid processing and heat treatment Materials Characterization 88 100-110
[5] Jirková H, Aišman D, Mašek B 2010 Unconventional structure of X210Cr12 steel obtained by thixoforming Journal of Alloys and Compounds 504 S500–S3
[6] Aišman D, Jirková H, Kučerová L, Mašek B 2011 Metastabulka structure of austenite base obtained by rapid solidification in a semi-solid state Journal of Alloys and Compounds 509 S312–S15
[7] Aišman D, Jirková H, Mašek B 2012 The influence of deformation and cooling parameters after transition through semi-solid state on structure development of ledeburite steel Journal of Alloys and Compounds 536 S204-S7
[8] JMatPro, Release 9.0, Sente Software Ltd., 2016