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Effects of Deformation on the Behaviour of Chromium Carbides in Tool Steel Studied by Use of Semi-Solid Forming

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Abstract: Conventional technology is mainly used for processing parts where high hardness, although conventional treatment of tool steels is ordinarily used in industrial practice, engineers continue to seek new procedures to rid tool steels of objectionable primary sharp-edged chromium carbides, which impair toughness. Fortunately, research into metal forming yielded new methods of modifying the microstructure of hypereutectoid steels. Using these methods, mechanical properties can be improved by virtue of eliminating objectionable sharp-edged carbides. These carbides resist dissolution and their size and shape make them undesirable microstructural constituents. Although they do improve wear resistance of the matrix, they also impair toughness and may act as stress concentrators. The microstructures produced by a sequence involving semi-solid processing and subsequent forming operations were different from conventional semi-solid-processed microstructures. In the former microstructures, the prior carbide network was broken up, dispersed, and became a strengthening constituent. Brittleness which plagues materials with prominent carbide networks was thus removed.

The experimental material used in this study was X210Cr12 tool steel. Two semi-solid processing temperatures were used: 1240°C and 1260°C. There were two holding times: 30 minutes and 60 minutes. Another variable was the number of reductions. The resulting microstructures were examined with respect to individual sequences and reductions applied. Detailed microstructure analysis was carried out using a scanning electron microscope (SEM). Chemical compositions of carbides were determined by means of EDS (Energy Dispersive X-ray Spectroscopy). Microhardness was measured in order to gather comprehensive materials data. The purpose of the study was to identify trends, if any, in microstructural property evolution in response to the above-described processing sequence.

Keywords: semi-solid processing, primary chromium carbides, tool steel, carbide refinement

1 Introduction

Unconventional treatment of material by heating to the semi-solid state has been known since the 1970s. At that time, it was primarily used for low-melting materials. Most often, they were aluminium and magnesium alloys [1, 3]. Years later, the process was adopted for higher-melting materials, typically steels [2-4]. For the desired microstructure to be obtained, a liquid fraction interval of 10%-60% must be maintained. Microstructures produced by semi-solid processing typically consist of polyhedral austenite grains embedded in a ledeburite network. In terms of the effect of the network on mechanical



properties, this configuration is undesirable as the network is very brittle. It is therefore necessary to crush, refine and disperse its fragments uniformly across the part. The microstructure obtained in this manner will be fine-grained and homogeneous, containing a fine dispersion of carbides and showing improved mechanical properties.

Semi-solid processing involves heating to high temperatures. This was one of the reasons for using this process for eliminating large sharp-edged chromium carbides. These carbides greatly improve wear and creep resistance but reduce toughness of the material due to their large size. Typically, they are found in high-chromium tool steels. Tool steels produced by a conventional metallurgical route contain mostly primary carbides of the M_7C_3 type. These are very difficult to eliminate by conventional processes. Consequently, some steels must be made by powder metallurgy [5-7]. To overcome these difficulties, a sequence was developed which combines semi-solid processing and subsequent forming. It can eliminate sharp-edged chromium carbides by converting them into a very fine dispersion within a martensitic matrix. This sequence was tried on X210Cr12 tool steel. It was first applied to small specimens 30 mm in diameter and 55 mm in height [8-10]. Following the verification of all key parameters, the sequence was carried out again on stock which was almost three times larger. Specimens of this size were expected to provide enough material for manufacturing real-world products

2 Experimental program

The material which was studied in these experiments was X210Cr12 tool steel, a typical representative of a group of steels with large sharp-edged chromium carbides. Given its chemical composition and a relatively wide solidification range, this steel is suitable for semi-solid processing (Table 1). It was obtained in the form of annealed bars. The as-received microstructure contained primary sharp-edged chromium carbides and very fine cementite in a ferritic matrix (Figure 1). In order to be able to specify the values of key variables for this sequence, one has to identify the relevant processing window, i.e. the solidification range and liquid fractions at particular temperatures [1, 3]. Another variable was the temperature at which chromium carbides dissolve. As the rate of cooling from a forming temperature controls the decomposition of metastable austenite, it becomes another variable of importance. The heating temperatures and other parameters of the experimental sequence were identified using JMatPro software [11].

Table 1. Chemical composition of experimental steels.

X210Cr12	C	Cr	Mn	Si	Ni	P	S
Content [%]	1.8	12	0.3	0.35	max 0.5	max 0.03	max 0.035

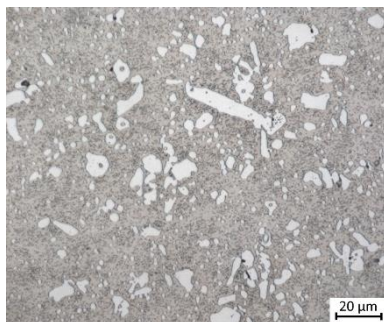


Figure 1. As-received condition of X210Cr12 steel, confocal micrograph.

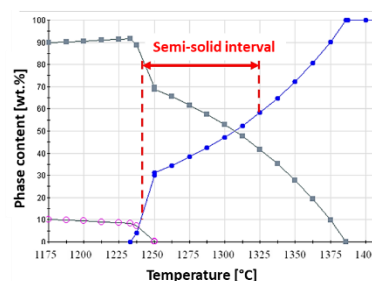


Figure 2. The calculated solidification range (semi-solid interval) for heating the stock.

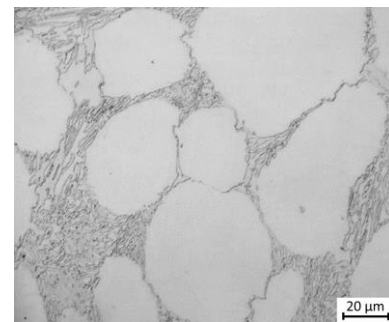


Figure 3. Microstructure of material which has been heated to semi-solid condition, no deformation was applied, light micrograph.

Calculations performed using JMatPro revealed that up to 758°C, the material retains a ferritic-cementitic microstructure. Incipient melting takes place at 1225°C (Figure 2). Melting is finished at 1373°C. Chromium carbides should dissolve completely at 1255°C.

In earlier experiments, the sequence was tried and fine-tuned on smaller specimens 30 mm in diameter [9, 12]. The aim of the present experimental programme was to transfer the procedure to larger stock in order to obtain a larger amount of uniform material. It was essential to bring the material into the semi-solid state in order to obtain an optimal liquid fraction. After cooling to forming temperature, appropriate deformation was to disintegrate and uniformly disperse ledeburite network (Figure 3), (which forms upon semi-solid processing) and initiate dynamic recrystallization of austenite [9, 12].

In this experimental programme, several heating temperatures and amounts of reduction were used. Specimens were heated in an air furnace with no protective atmosphere and formed in CKW 1000 hydraulic press.

The resulting microstructures were examined using optical (OM) and scanning electron microscopy (SEM). Mechanical properties were determined by means of hardness and microhardness testing.

2.1 Semi-solid processing and subsequent thermomechanical treatment

Experimental specimens were 75 mm in diameter and 150 mm in length. For ease of handling of partially-melted stock, the specimens had to be inserted into containers of SJ355 steel, which has a higher melting point.

A bar of X210Cr12 tool steel 65 mm in diameter was inserted into a low-carbon steel tube which was sealed with lids on both ends and welded closed. Four experimental sequences were designed, involving two different temperatures and several other variables (Table 2).

The heating temperature for the first two sequences was 1240°C and the time at temperature was 30 minutes. Soaking was followed by water quenching for 20 seconds, after which the stock was placed into a furnace at 1080°C and held for 10 minutes. Temperature of 1080°C is the recommended forging temperature, as set forth in the material data sheet for X210Cr12. After holding in a furnace, the stock was formed between flat dies in a hydraulic press. In sequence 1, three reductions were used. The first was a reduction of the diameter by 20 mm along the entire length of the workpiece. The second reduction was by 20 mm, but only along 2/3 of the workpiece length. The last reduction, by another 20 mm, only spanned one third of the workpiece length at its end. This produced three different regions for comparing the impact of deformation within sections reduced to different thicknesses on refinement of carbides and disintegration of the carbide network (Table 2).

In sequence 2, only two reductions were applied. The first one, to half thickness, i.e. to 37.5 mm, covered the entire length of the workpiece. The second one, to 18 mm, only spanned one half of the length.

Sequence 3 involved a longer time at the semi-solid process temperature 1240°C: instead of 30 minutes, it was 60 min. In addition, the time at 1080 °C, the forging temperature, was longer than in previous sequences. Deformation was imparted in a manner identical to sequence 1. In the last sequence, no. 4, the semi-solid process temperature was increased above the previous level, to 1260°C, and the time at temperature was extended to 60 minutes. The time at 1080 °C was extended as well: from 10 minutes to 30 minutes. Deformation was imparted in a manner identical to sequence 1 (Table 2).

Table 2. Heating temperatures, times, and deformation parameters.

Sequence	Semi-solid process temperature [°C]/ time [min]	Cooling method	Forming temperature [°C] /Time at temperature [min]	Number of reductions [-]	Mode of reduction	Cooling method
1	1240/30	Quenched in water/20s	1080/10	3		Quenched in oil to RT
2				2		
3	1240/60			3		
4	1260/60			1080/30	3	

3 Discussion and Results

Microstructures of the workpieces were examined on longitudinal metallographic sections, with reference to their portions which experienced different amounts of reduction.

Sequence 1 involved a temperature of 1240°C. The resulting specimens contained austenite and a fine dispersion of chromium carbides and cementite particles. It was thus clear that the temperature was too low and failed to cause dissolution of all primary chromium carbides. If melting occurred, it was probably limited to grain boundaries. In the single-reduction portion of the workpiece, the carbide network was already fragmented, with some fragments dispersed through the austenitic matrix (Figure 4). With increasing magnitude of deformation, austenite grains became finer. In the portion subjected to two reductions, the grain size was approximately 20 μm (Figure 5). Where the largest deformation was applied, the austenite grains were smaller than 10 μm (Figure 6). Larger strains dispersed chromium carbides and cementite more effectively.

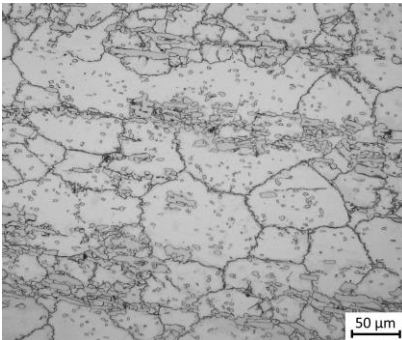


Figure 4. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 1 - reduction 1.

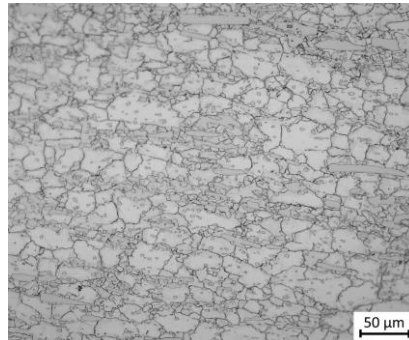


Figure 5. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 1 - reduction 2.

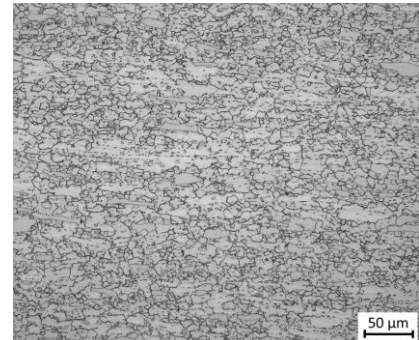


Figure 6. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 1 - reduction 3.

The same results were obtained with sequence 2 which involved two reductions. The portion of the workpiece which had been subjected to a single reduction from 75 mm to 37.5 mm contained an austenitic structure with dispersed chromium carbides (Figure 7). The portion which also received the second reduction by 19.5 mm to the final thickness of 18 mm contained a very fine microstructure thanks to dynamic recrystallization of austenite. The grain size was less than 10 µm (Figure 8).

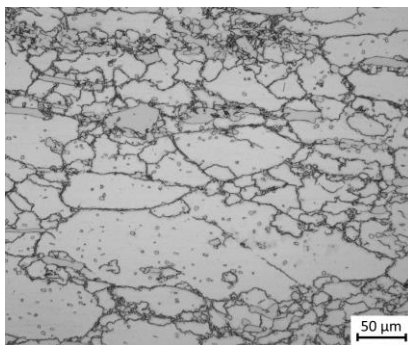


Figure 7. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 2 - reduction 1.

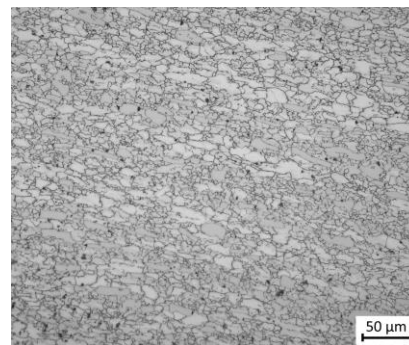


Figure 8. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 2 - reduction 2.

Holding time was extended in sequence 3 from 30 minutes to 60 minutes. Microstructural evolution initiated by this sequence was similar to those in the previous sequences. In addition, the time at the forging temperature of 1080°C was also increased from 30 min to 60 min. The workpiece portion which had been reduced from 75 mm to 55 mm contained a rather coarse microstructure. It was obvious that the long time at 1240°C caused partial dissolution of primary chromium carbides. The resulting carbide particles are smaller and uniformly distributed throughout the material (Figure 9). The second reduction from 55 mm to 37 mm reduced the austenite grain size. In the relevant portion of the specimen, the grain size was around 20 µm (Figure 10). Where additional strain was introduced, it continued to refine the grain. The third reduction, from 37 mm to 17 mm, produced a grain size of less than 10 µm (Figure 11).

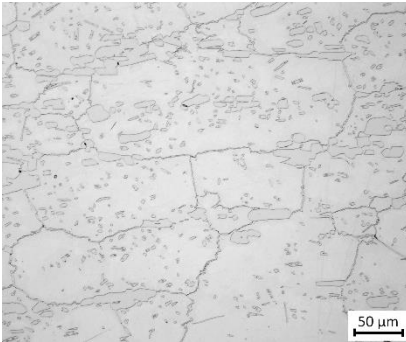


Figure 9. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 3 - reduction 1.

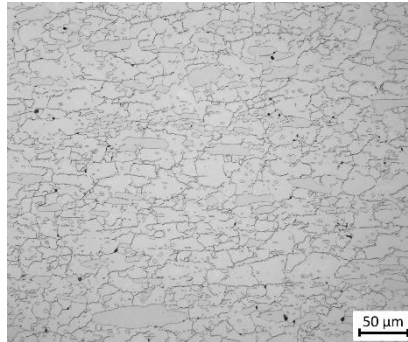


Figure 10. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 3 - reduction 2.

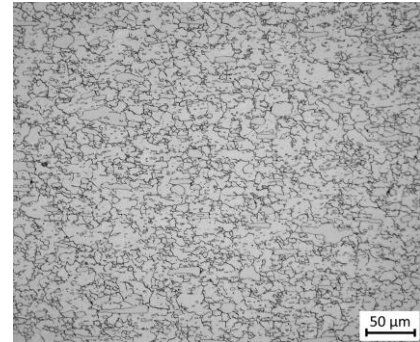


Figure 11. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 3 - reduction 3.

In the last sequence, the semi-solid process temperature was increased to 1260°C, and the time at temperature was extended to 60 minutes. This higher temperature caused partial melting in the structure, and dissolved fine cementite and primary chromium carbide particles. The network which formed upon cooling was very fine and consisted of austenite and carbides. The first reduction led to no substantial distortion of austenite grains (Figure 12). After the second reduction, the strain imparted was manifested in elongated austenite grains and in stronger dynamic recrystallization in some regions. The mean grain size was around 20 μm (Figure 13). The last reduction resulted in notable elongation of austenite grains and alignment of the ledeburite network with the deformation direction. Recrystallization led to a number of very fine austenite grains. Along some austenite grain boundaries, austenite transformed to martensite. This occurred primarily in regions with a large proportion of carbide network. In these regions, carbon was bound in the carbide network, rendering austenite unstable during cooling to room temperature (Figure 14).

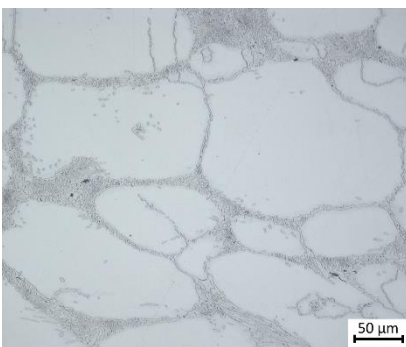


Figure 12. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 4 - reduction 1.

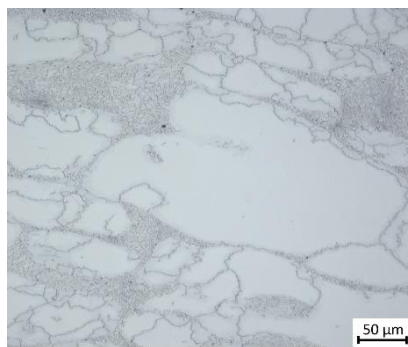


Figure 13. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 4 - reduction 2.



Figure 14. Microstructure produced by heating to semi-solid state and subsequent deformation - sequence 4 - reduction 3.

Detailed examination of the microstructure was conducted with the aid of scanning electron microscopy. Using high magnifications, the presence of martensite was even confirmed upon sequences involving the lower temperature, 1240°C. Martensite needles were found not only in the vicinity of reprecipitated fine carbides but also around the original chromium carbides (Figure 15). Close examination of ledeburite network in a specimen upon sequence 4, the sequence involving the higher temperature, 1260°C, revealed martensite even in those austenite grains within the network,

in addition to new austenite grains. Very fine secondary chromium carbides sized approximately 2 μm were found as well (Figure 16).

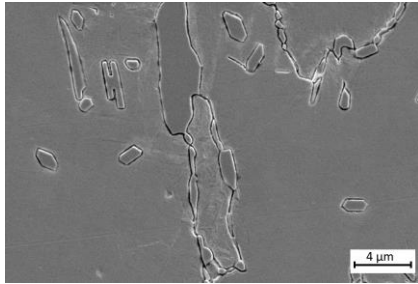


Figure 15. Sequence 4 - reduction 3 - strings of secondary carbides along grain boundaries, martensite needles within austenite, scanning electron micrograph.

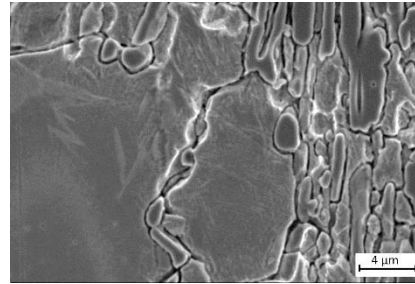


Figure 16. Sequence 3 - reduction 1 - undissolved primary Cr carbides, scanning electron micrograph.

EDS was employed for chemical analysis of carbides (Figure 17). It was found that both primary chromium carbides and newly-formed strings of secondary carbides were rich in chromium, whereas the matrix was depleted of this element.

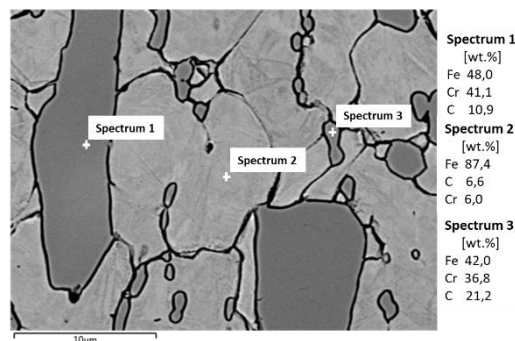


Figure 17. EDS analysis of primary carbides and the matrix – sequence 3 – reduction 3.

Measurement of Microhardness

Microhardness HV0.1 was measured in individual microstructure constituents: the matrix (Figure 18) and chromium carbides (Figure 19). These measurements were made for all sequences and for all combinations of reductions. In each region, three readings were taken and their arithmetic mean was calculated.

In the matrix, hardness values reflected the number of reductions performed. In regions subjected to a single reduction, most values were within the range 360–415 HV0.1. Three reductions led to higher hardness levels: 580–950 HV0.1. Another parameter of importance was temperature. Sequence 4 which involved a temperature of 1260°C, led to higher hardness in the matrix, up to 600 HV0.1 in the region subjected to a single reduction. Hardness levels of carbides were within 1200–1400 HV0.1.

Table 3. Mean values of microhardness (HV0.1) regardless of the number of reductions.

Sequence	Location	HV0.1
Sequence 1	Matrix	724
	Cr carbide	1272
Sequence 2	Matrix	373
	Cr carbide	1052
Sequence 3	Matrix	474
	Cr carbide	1085
Sequence 4	Matrix	640
	Cr carbide	1039

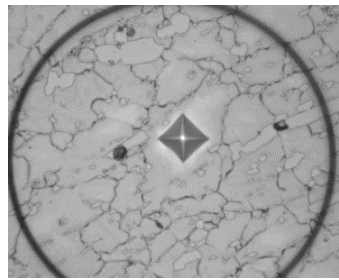


Figure 18. Sequence 3 - reduction 2 – an impression in the matrix – 415 HV0.1.

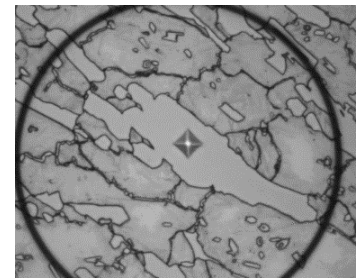


Fig. 19. Sequence 3 - reduction 1 – an impression in a primary chromium carbide – 1267 HV0.1.

4 Conclusion

Experimental semi-solid processing with subsequent forging in a press was carried out on X210Cr12 tool steel. The purpose was to identify the effects of temperatures of 1240°C and 1260°C and times at these temperatures on dissolution of primary chromium carbides and microstructural evolution. Where the temperature of 1240°C was maintained for 30 minutes, the specimens developed microstructures consisting of austenite with a fine dispersion of chromium carbides and cementite. In locations which experienced a single reduction, the carbide network became fragmented, with some fragments dispersed through the austenitic matrix. Higher strains led to further austenite grain refinement. Larger strains also dispersed chromium carbides and fine cementite particles more effectively. Sequence 3 involved holding at 1240°C for an extended period of 60 minutes but failed to produce any substantial changes in the microstructure. In the single-reduction region, the microstructure was rather coarse. It became clear that the longer time at 1240°C and added reductions had produced finer grains. Each reduction reduced the grain size by approximately 10 μm. After the third reduction, the grain size was approximately 10 μm. In the last sequence the semi-solid process temperature was increased to 1260°C, and the time at temperature was extended to 60 minutes. This higher temperature caused partial melting in the structure, and dissolved fine cementite and primary chromium carbide particles. The network which formed upon cooling was very fine and consisted of austenite and carbides. The last reduction resulted in notable elongation of austenite grains and alignment of the ledeburite network with the deformation direction. Recrystallization led to a number of very fine austenite grains. Along some austenite grain boundaries, austenite decomposed into martensite.

Distribution of individual phases in the microstructure was mapped using EDS. It was found that both primary chromium carbides and newly-formed strings of secondary carbides were rich in chromium, whereas the matrix was depleted of this element. Microhardness increased with the amount of strain, and finally ranged from 580 to 950 HV0.1. Hardness of carbides remained virtually constant for all sequences: 1200-1400 HV0.1.

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