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Modeling of the Steel Microstructure Gained after the Application of an Ultra-Fast Heat Treatment

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Abstract

The current work elucidates the phase transformation and carbide dissolution during rapid heating followed by short austenization and quenching to room temperature. For this work, dilatometry experiments were carried out in medium carbon low alloy steel containing chromium and molybdenum (42CrMo4).The thermal cycle of the sample was analyzed with the use of Thermocalc® and Dictra. The simulation results indicate that the high heating rate and short austenization time limits diffusion and the alloying elements segregate at the interfaces between carbides and ferrite. In addition, the segregation of alloying elements at the interfaces in conjunction with the rapid heating rate and the short dwelling time leads to incomplete dissolution of carbides and the formation of austenite with varying sizes and different carbon content, which in some cases reaches 1% wt; this of course can be of significance as it affects phase transformations and lead in retaining the austenite during quenching. The carbide morphology, size and type could be revealed only after Transmission Electron Microscopy (TEM) analysis. The microstructure consists of bainite, martensite and undissolved carbides confirming the results of simulation. Further investigation in the effect of chemical composition and size of austenite during bainitic and martensitic transformation is necessary and thorough study on the effect of the alloying elements on the transformations mentioned above.

Keywords: Ultra – fast heating, Ultra – fast annealing, Phase transformations, TEM, Undissolved carbides, Martensite, Bainite, Thermocalc®, Phase diagrams, DICTRA simulation, 42CrMo4, Automotive, Austenization, Kinetics, Moving boundaries.

Introduction

Within the last three decades advanced high strength steels (AHSS) have been fully implemented by the automotive industry as they fulfill the requirements for improved fuel efficiency while not compromising the safety of the passengers. The advanced high strength steels include dual phase (DP), transformation induced plasticity (TRIP), complex phase (CP), partially martensitic steels (PM), twining induced plasticity steels (TWIP) and quenched and partitioning steels (Q&P). These steels show good formability while maintaining high strength values [1,2]. The enhanced mechanical properties are attributed in multiphase microstructure consisting of ferrite, bainite, martensite and austenite. However, complex and energy consuming heat treatments must be implemented to obtain this microstructures or even expensive alloying additions (e.g. Mn). For this reason, the need for producing low – cost and advanced steel development is imminent. Nowadays, steel development does not rely on ferrite grain refinement alone. Strengthening mechanisms such as solid solution, carbide precipitation causing dislocation generation and slip, transformation or twinning during deformation are well established and fully incorporated in industry. Current attempts focus on obtaining a good combination of strength and ductility. This is achieved via ultra – fine grained nano – precipitated hardened phases mainly ferritic and bainitic, mixtures of retained austenite with martensite and distribution of dislocations. In addition, modifications in martensite morphology and microstructures containing bainite and martensite have been reported to increase both toughness and ductility. Nonetheless, mixed microstructures with martensite and bainite require austempering treatments at 125 – 350 °C up to 240 h [3-9]. A promising approach [1] to achieve high strength with increased ductility has led to the development of flash processing i.e. rapid heating, short holding time at the austenisation temperature and quenching which results in mixed microstructure of martensite and bainite exhibiting superior mechanical properties than advanced high strength steels (e.g. 1,4 – 1,5GPa for yield strength (YS), 1,8 – 2,0GPa for tensile strength (TS) and 7 – 8% for total elongation). Using the unconventional approach [1,10-12] to reheat steel rapidly leads to chemical heterogeneity, which ends up in complex microstructures in one step. Microstructure evolution under ultra-fast (UF) cycles if understood and exploited can result to a new alloy design approach. The current work focuses on the identification of the microstructural mechanisms that lead to these mechanical properties. For this purpose, we use dilatometry to apply rapid thermal cycles, STEM to characterize the ultra-fine microstructures (phases, type and morphology of carbides), EDS to define the composition of precipitates and the

chemical heterogeneity within the microstructure. Thermocalc [13] and DICTRA [14] serve our efforts to rationalize and closely monitor phase transformations that require time for diffusion when this time is not available and to explain the microstructural evolution under rapid thermal treatment.

Materials and Methods

In order to examine the evolution of microstructure under rapid thermal cycles, typical soft annealed medium carbon chromium molybdenum steel was used with spheroidized carbides (Table 1 shows its chemical composition). Ultrafast heat treatment was applied using dilatometry. Transmission electron microscopy (TEM) measurements were conducted with a high resolution JEOL JEM-2100, operating at 200 kV, equipped with an Oxford energy-dispersive X-ray spectroscopy (EDS) microanalysis system. Dilatometric plate and round samples (plate sizes length 10mm with width 2mm; round sizes: length 10mm with diameter of 3mm) derived from cross sections cut out of hot rolled bars with dimensions 95x49x5500 mm^3 (width x thickness x length). Chemical composition analysis was performed in 30x30 mm^2cross sections of the bars, transverse to rolling direction by means of Optical Emission Spectroscopy (OES). Cylindrical as well as non-Standard dilatometric plate specimens were machined using Wire Electro-Discharge Machining (EDM) with dimensions 2x4x10 mm3. The nonstandard specimens allow higher heating and cooling rates to be achieved, critical for the present heat treatment cycle. EDM was used to assure that the dimensions have low tolerance and at the same time the microstructure remains intact. The dilatometry tests were performed in a Bähr 805A Quench dilatometer. The apparatus uses an induction coil to heat the sample and detects the length change of the sample with a Linear Variable Displacement Transducer (LVDT). The specimens were placed in the dilatometer with a thermocouple spot welded at the middle in order to control the temperature. Experiments with two thermocouples were also performed, one at the edge and the second at the middle in order to check the temperature gradient and, thus, to make sure that the transformations occur in the whole sample and not just in the middle. It was found, that for the nonstandard specimens, even though the sample cross section was not cylindrical, the temperature variation margin was within 10oC. All samples were heated at 200 °C/sec to peak temperature (950 °C) under vacuum and then quenched with helium at the same rate to room temperature. The treatment included no isotheral holding time at the austenitization temperature.

Table 1: Chemical analysis of CrMo-steel (in wt.-%)

C	Mn	Si	P	S	Cr	Mo
0.407	0.79	0.21	0.009	0.010	0.97	0.187

Results

TEM results indicate that the microstructure after the ultra – fast heat treatment consists of bainitic ferrite, martensite and homogeneously distributed spherical carbides ranging from 5nm to 500nm. In the microstructure two types of carbides have been reported: chromium enriched cementite and chromium carbides (Figure 1). In addition, high dislocation density, dislocation pile-up was found in the grain boundaries of ferrite and Moire fringes were mostly found in carbide/ferrite boundaries. In addition, the EDS analysis (Figure 2) shows that chemical heterogeneity prevails in the microstructure. Both chromium and manganese tend to increase at carbides while silicon is homogeneously distributed.

Modeling

In order to rationalize the TEM results of dilatometry, thermodynamic and kinetic calculations have been performed using thermodynamic database TCFE8 of Thermocalc and kinetic database MOBFE3 of DICTRA. At the beginning, the phase diagram of the CrMo steel in order to obtain the stable phases expected at the equilibrium. Thus, the initial microstructure consists of ferrite and three types of carbides: cementite, M_7C_3 and $M_{23}C_6$.

The A_1 and A_3 at equilibrium were not used in the calculations as they tend to shift at higher values with the rapid heating. For this reason, the A_1 and A_3 estimated from the dilatometry curves were used which are 810 and 900 °C respectively. In order

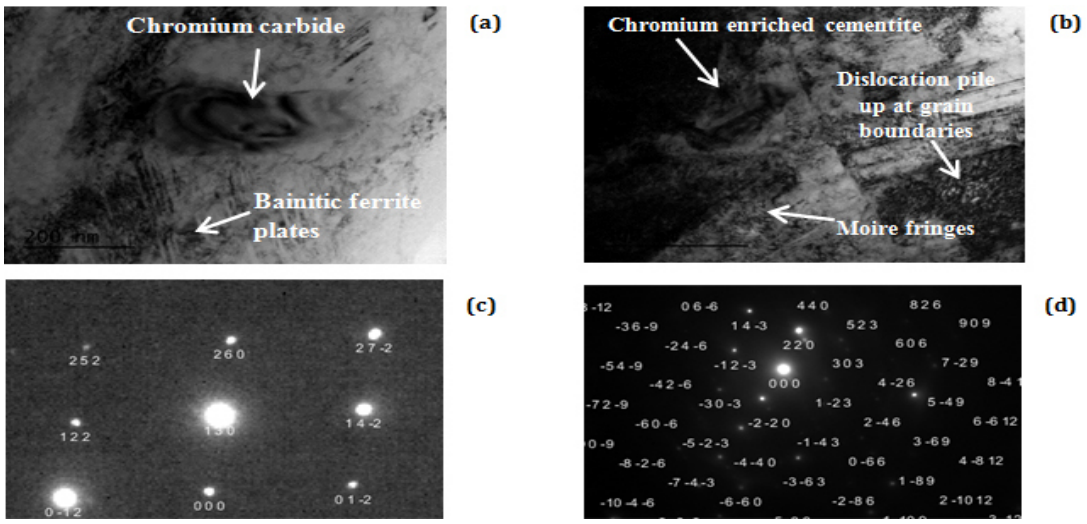

Figure 1: a) Spheroidizedchromium carbide surrounded by fine ferrite plates, **b)** Chromium enriched cementite surrounded by fine ferrite plates, **c)** Indexed pattern of chromium carbide, **d)** Indexed pattern of chromium enriched cementite (14)

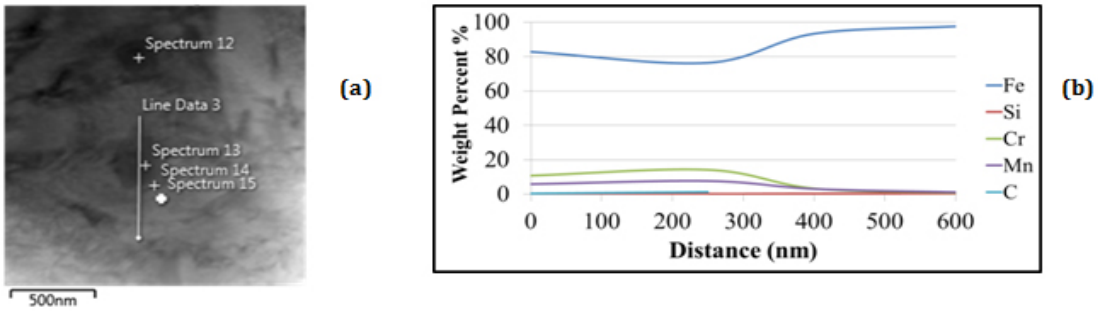

Figure 2: a) microstructure in which the EDS analysis was conducted,**b)**EDS analysis of the sample which indicates the chemical heterogeneity. The area in which chromium and manganese are increased represents the area of carbide and proves that both of these elements have the same tendency.

to study the microstructural evolution during rapid heating in DICTRA six (6) thermal sections were studied from 513 to 950 °C. In each section, the diffusion of alloying elements at the interfaces of carbides/ferrite and carbides/austenite has been studied. Due to its limited solubility in carbides silicon (Si) was excluded from the system, which is limited to Fe – C – Cr – Mn -Mo. The starting point for the simulation was set at 513 °C and the chemical composition of each phase was calculated with Thermocalc®. The dilatometry results (heating rate, cooling rate and dwelling time) are used as input data in DICTRA simulations in each section. For brevity, the results from 728 °C to 950 °C as indicated in Table 2 will be shown and discussed.

Table 2: Thermal sections from 728 to 950 °C with each interface in conjunction with heating rate and dwelling time obtained from dilatometry

Thermal sections	**728 – 810 °C**	**810 – 900°C**	**900 – 950°C**	**950**°C Isothermal
Examined Systems	Cementite / Ferrite M_7C_3 / Ferrite $M_{23}C_6$ / Ferrite	Cementite/ Austenite/ Ferrite M_7C_3 / Austenite / Ferrite Austenite/ Ferrite	Cementite/ Austenite M_7C_3/ Austenite Austenite	Cementite / Austenite M_7C_3 / Austenite Austenite
Heating rate	191,3°C/s	238,94°C/s	187,59°C/s	0
Dwelling time	0,43 s	0,376 s	0,266 s	1,8 s

The initial size of carbides was set to 5nm with spherical geometry and symmetric mesh. At the interfaces of carbides with ferrite or austenite, thermodynamic equilibrium can be assumed to locally hold (local equilibrium hypothesis). The DICTRA simulation is in good agreement with TEM analysis (Figure 3, Figure 4). The results indicate the segregation of alloying elements at interfaces of carbides with ferrite and austenite. Furthermore, the results from DICTRA prove that undissolved carbides exist at the austenization temperature and chemical heterogeneity prevails in the microstructure leading to austenite regions poor (~0.4% wt) and rich (~1% wt) in carbon. The chemical heterogeneity controls phase transformations i.e.bainite and martensite in the stage of quenching while the rich austenite regions might lead to retained austenite.

Discussion

The initial microstructure of the sample is soft annealed with spheroidized carbides. Slow heating can support the diffusional progress of manganese and chromium positioning at favorable locations within the microstructure. In case of rapid heating rate (>200 °C/s) the diffusion of aloying elements is restricted especially those with larger atomic radius such as chromium (Cr) and manganese (Mn), however, a partitioning of alloying elements is observed at 2 – D defects such as grain boundaries or boundaries [15,16]. The segregation of alloying elements at grain boundaries locally alters the chemical composition contributing to chemical heterogeneity. The gradient of alloying elements such as chromium (Cr) and manganese (Mn) limits the dissolution of carbides. The undissolved carbides act as nucleation sites, thus, leading to an exceptionally refined microstructure. Moreover, the undissolved carbides as well as the segregation of alloying elements at grain boundaries affects phase transformations such as bainite and martensite during quenching. Segregation depends on misorientation between grains and dislocations in the microstructure [12].The higher the misorientation angle, the more intense the segregation. Moire fringes and dislocations at the triple points of grain boundaries or in the proximity of undissolved carbides acts as additional sites for segregation thus enhancing micro – diffusion in these regions. Finally, the limited time at the austenization temperature not only restricts the austenite growth but also ensures the chemical heterogeneity due to limited time for diffusion.

Conclusions

As a conclusion to this work, it has been observed that rapid thermal cycle restricts diffusion of alloying elements thus leading to segregation of alloying elements in 2 – D defects such as grain boundaries - interfaces and defects in the microstructure. The local increase of alloying elements in the interface of carbides controls the dissolution of carbides and the chemical heterogeneity in the microstructure. The undissolved carbides and the limited time at peak temperature prevent grain growth of austenite, thus, leading to refined microstructure. The chemical heterogeneity in austenite, in terms of carbon, strongly

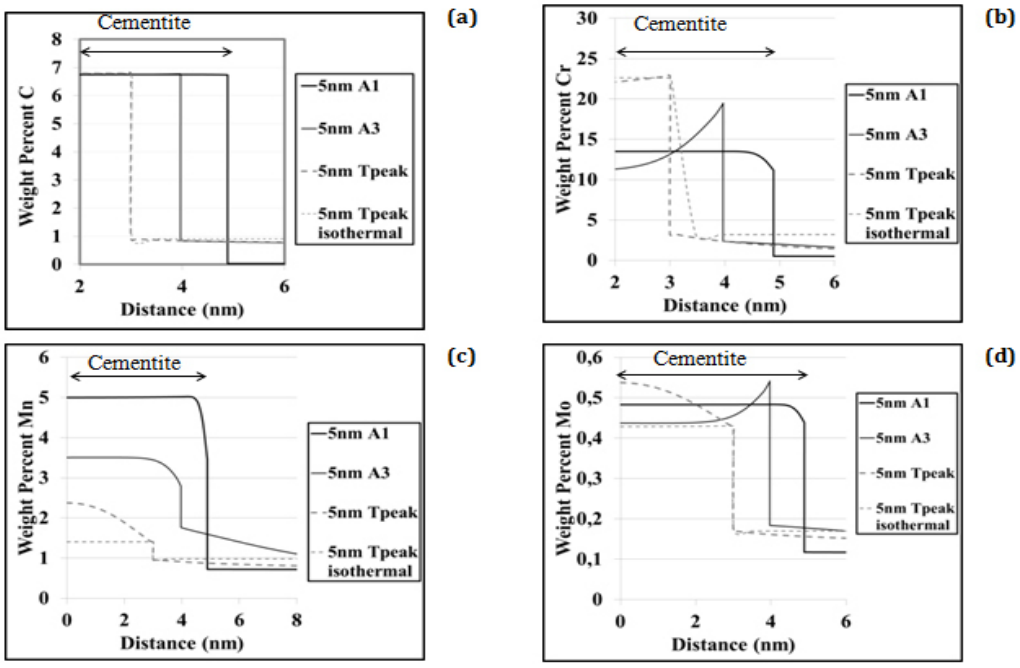

Figure 3: Variation of **a)** carbon, **b)** chromium, **c)**manganese and **d)** molybdenum at the interface of cementite at various critical temperatures for 5nm

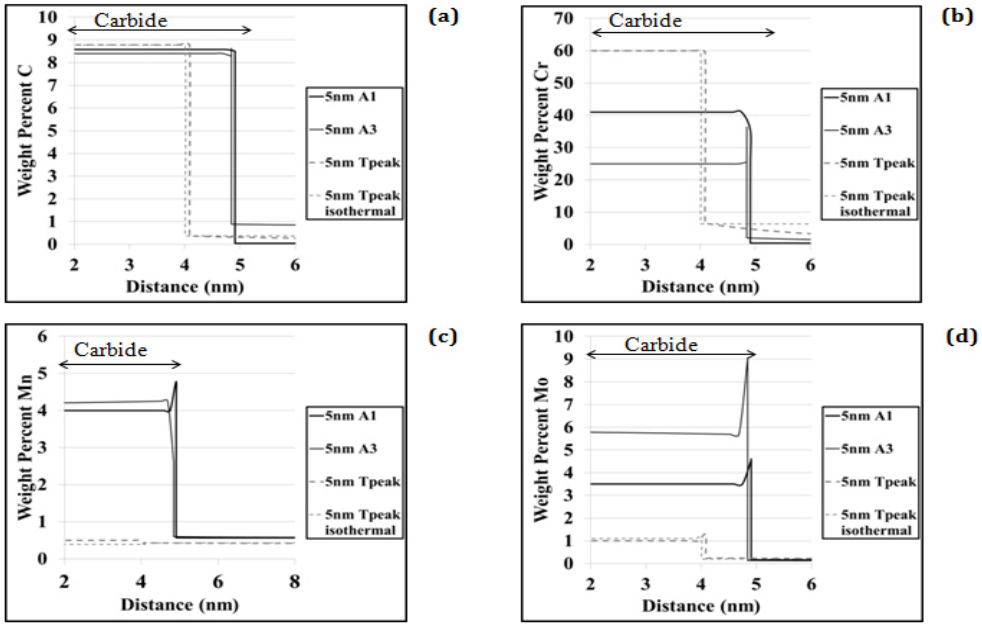

Figure 4: Variation of **a)**carbon, **b)** chromium, **c)** manganese and **d)** molybdenumat the interface of M7C3 carbide at various critical temperatures at 5nm

influences phase transformations in quenching thus leading in mixture of phases with bainite and martensite. However the simulation results show strong evidence for retained austenite in the final microstructure which was not found in TEM. Therefore, further investigation in the effect of chemical composition and size of austenite during and after the bainitic and martensitic transformation is necessary, as well as a thorough study on the effect of the alloying elements on the transformations mentioned.

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