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DOI 10.1016/j.msea.2020.139331

Publication date 2020 **Document Version** Final published version

Published in Materials Science and Engineering A

Citation (APA) Verdiere, A., Castro Cerda, F., Béjar Llanes, A., Wu, J., Crebolder, L., & Petrov, R. H. (2020). Effect of the austenitizing parameters on the microstructure and mechanical properties of 75Cr1 tool steel. *Materials* Science and Engineering A, 785, Article 139331. https://doi.org/10.1016/j.msea.2020.139331

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Contents lists available at ScienceDirect

Materials Science & Engineering A



journal homepage: http://www.elsevier.com/locate/msea

Effect of the austenitizing parameters on the microstructure and mechanical properties of 75Cr1 tool steel

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ARTICLE INFO

Keywords: 75Cr1 tool steel Austenitizing Carbide dissolution Strength Toughness

ABSTRACT

The austenitizing heat treatment of the 75Cr1 tool steel was optimized in order to obtain high yield strength and toughness. Samples were austenitized at different temperatures and soaking times, and subsequently quenched and tempered. The phase transformation characteristics during heating and quenching were studied by dilatometry. Optical microscopy, electron backscatter diffraction, x-ray diffraction and transmission electron microscopy were used to characterize the microstructure, while tensile tests and toughness tests were employed to determine the mechanical properties after various heat treatments. It was found that both yield strength and toughness decrease with increasing austenitizing temperature. Thermodynamic and kinetic calculations with Thermocalc and Dictra software were used to study the movement of the austenite-carbide interface and the compositional gradients in the microstructure at different austenitizing conditions. Based on the calculations of chromium and manganese between carbides and austenite. The compositional gradients in the microstructure form the Dictra calculations were confirmed by energy dispersive spectrum (EDS) measurements in transmission electron microscope. No significant changes in mechanical properties and microstructure (carbide fraction) could be observed after more than 15 min soaking. However a significant prior austenite grain size growth and as a consequence a larger martensite grain (block) size is observed for long soaking times.

1. Introduction

Tool steels refer to a variety of hard and tough steels that are used to make tools for cutting, forming or shaping. They typically contain a high amount of carbon (often hyper-eutectoid) and a combination of alloying elements, aiming to control quenchability, hardness, toughness, wear and fatigue resistance, etc. The final microstructure typically consists of untransformed cementite/carbides in a matrix which contains mainly martensite and some retained austenite. Due to the chemical composition of the steel and the applied heat treatment, the martensite finish temperature M_f is often below room temperature [1]. Therefore, a significant fraction of untransformed (retained) austenite can remain in the final microstructure and may alter the mechanical and functional properties. The amount of retained austenite can be minimized by cryogenic quenching. However, the selection of the steel grade and its heat treatment depend on the application and the desired material properties. In this study, the maximum potential of a DIN 75Cr1 tool steel, with main alloying elements Cr, Mn and Si, is explored for applications where a combination of high yield strength, high fatigue resistance, wear resistance and toughness is required. Examples of applications for this grade are the body of circular saws and so-called elements in the pushbelt of the continuously variable transmission (CVT) in cars, however many similar applications exist.

The microstructure of the as-received 75Cr1 grade, after spheroidization annealing, is a mixture of soft ferrite and hard spheroidized (Fe, Cr)₃C carbide (or Cr-alloyed cementite), having good machinability and formability. After forming, the steel is subjected to the classical heat treatments, consisting of austenitization, quenching and low temperature tempering. It is well known that the chemical composition and the microstructure of the parent austenite have a strong influence on the martensitic transformation (M_s and M_f temperatures) and the hardness and microstructure formed by subsequent quenching. During

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https://doi.org/10.1016/j.msea.2020.139331

Received 8 December 2019; Received in revised form 28 March 2020; Accepted 30 March 2020 Available online 5 April 2020 0921-5093/© 2020 Elsevier B.V. All rights reserved. austenitization heating above the Ac₃ temperature, all ferrite and a fraction of carbides transform cooperatively into austenite (stage 1) and then the remainder of the carbides dissolve once all ferrite is transformed to austenite (stage 2) [2]. Firstly, austenite nucleates preferentially at the alloyed cementite-ferrite interface or at ferrite-ferrite grain boundaries, which are energetically preferential nucleation sites [3]. The austenite grows towards ferrite until the carbides are completely enveloped [4]. It is reported that the average diffusion rate of carbon in austenite at temperatures of 800–850 $^\circ\text{C}$ is approximately 2.34 μm per second [5], therefore the carbon concentration in the austenite region formed from the decomposition of ferrite and carbides can be assumed to be nearly uniform during heating and soaking. The extent of the alloyed cementite to austenite transformation affects the effective carbon concentration in the austenite and in the subsequent martensite. Since carbon accounts for the major contribution to the martensite strength, the dissolution of carbides influences the mechanical properties. Meanwhile, the hard untransformed carbides can increase the wear resistance of the tool steels. Thus the rate of dissolution of pre-existing carbides during austenitization is critical in balancing between the mechanical (strength) and the functional (wear resistance) properties. Moreover a large volume fraction of fine carbides can effectively suppress the austenitic grain growth and improve both the strength and toughness [1]. To optimize the balance between the properties, the tool steels are often alloyed with various combinations of carbide forming elements such as Nb, V, Mo, W and Cr, with a decreasing carbide forming ability from Nb to Cr [6,7].

The dissolution of carbides into austenite requires partitioning of both carbon and other alloying elements during the austenitizing treatment. The stability of the spheroidized carbides depends on how the alloying elements are partitioned between the alloyed cementite and the matrix. Cr, Mn, Mo and W have the highest ratios (in descending order) of wt.% of the element contained in the cementite to wt.% in the matrix [8]. Therefore, Cr is often used as a cementite stabilizer. Liu et al. [9] studied carbide dissolution during austenitizing of an Fe-2.03Cr-3.91C (at.%) alloy and confirmed that the undissolved carbides were enriched in Cr. They suggested that the main part of the reaction for the studied alloy is controlled by Cr diffusion. Hillert et al. [10] concluded that the dissolution rate of the carbides is controlled by the rate of diffusion of carbon in the very early stages and of the alloying element in the later stages. Alloying elements such as Cr or Mn lead to a longer austenite nucleation incubation period and a lower nucleation rate of austenite compared to Fe-C alloys, which can be explained by the alloying element affecting both carbide stability and diffusion fluxes [3]. However, such delaying effect is less significant at elevated temperatures. The partitioning of the alloying elements between the austenitic matrix and the undissolved carbides determines the volume fraction of the carbides as well as the chemical composition of the austenite.

The tempering treatment is usually the last step to release the thermal residual stress and to further strengthen the martensite in the tool steel. During the tempering step, carried out at 180–190 °C in oil baths, carbon atoms are released from the martensite dislocations in the carbon supersaturated matrix to form carbide precipitates [11]. The carbon diffusion causes a decrease of the tetragonality of the martensite lattice and the formation of transition epsilon or eta carbides at tempering temperatures between 100 and 200 °C [12]. In some cases, a part of the carbon-depleted retained austenite can transform into tempered martensite at this temperature range, because the M_s temperature increases [13]. After the final heat treatment, the microstructure is tempered martensite with untransformed carbides and possibly retained austenite. Changes in the microstructure have a large influence on the mechanical properties and give the possibility to find a good balance between toughness and strength.

The goal of this study is to find a clear link between the heat treatment parameters and the final properties of 75Cr1 steel used for elements of the pushbelts for the CVT, aiming for high yield strength and toughness. Having in mind the importance of the austenitization stage for the microstructure formation and properties, the influence of the austenitizing parameters, namely austenitizing temperature and soaking time, on the microstructure and correspondingly on the strength and toughness of the steel will be investigated. In order to quantitatively describe the process of carbide dissolution during austenitization, the distribution of alloying elements in the alloyed cementite and austenite during the heat treatment is simulated and measured experimentally.

2. Experimental procedure

The chemical analysis of the DIN 75Cr1 steel is given in Table 1. The as-received steel sheets have a thickness of 1.8 mm and are in the so-called "soft annealed" condition with a microstructure of ferrite and spheroidized carbides (Fig. 2).

A Bähr DIL 805 A/D dilatometer was first used to determine the austenitization kinetics of the 75Cr1 steel specimens, with rectangular shape of 10 x $4.5 \times 1.5 \text{ mm}^3$. Samples were heated at 20 °C/s to 800, 820, 860, 900 or 1000 °C and soaked for 2 min or 10 min. The specimens were subsequently quenched to room temperature with a cooling rate higher than 200 °C/s to form martensite. The heating rate was chosen to be close to the heating rate in the salt bath. According to preliminary calculations with JMatPro® software [14], the critical quench rate for a steel with this composition varies between 60 and 80 °C/s, depending on the austenite grain size [15].

The steel was subjected to 40 different heat treatments in a salt bath with various austenitizing temperatures and soaking times and subsequently oil quenched to form martensite. The austenitizing temperatures were 800, 820, 840, 860, 880 and 900 °C, and at each temperature the samples were soaked for 2, 4, 6, 8, 10, 12, 15, 20, 25 or 30 min before oil quenching (30 °C). The minimum cooling rate of oil in the temperature range of 850 to 750 °C is 20 °C/s and in the temperature range of minimum stability of austenite for this steel (600–400 °C) it is \sim 70–80 °C/s [16]. Afterwards all samples were tempered at 190 °C for 45 min in an oil bath and air cooled in order to keep the hardness after quenching, minimizing slightly the post quenched stress.

The initial microstructure was studied by optical microscopy, after etching with 4 vol% HNO3 in ethanol (4% nital) for 5 s. The carbide fraction and size were quantified by means of ImageJ Software on SEM images of samples etched with 4 vol% picric acid in ethanol (picral 4%) to reveal the edges of the carbides. Electron backscatter diffraction (EBSD) was performed with a FEI Nova 600 Nanolab at 20 kV and beam current 2.2 nA. The sample preparation for EBSD consisted of mechanical and electrolytic polishing with A2 Struers electrolyte, followed by 40 min OP-U polishing with 5 N pressure to remove the remaining surface relief after electropolishing. In all EBSD measurements, a step size of 50 nm in hexagonal scan grit was employed and the points with confidence index lower that 0.1 were removed from the maps. The scans were post-processed by applying the grain confidence index correlation procedure, with grain definition as follows: min 8 pixels/grain and 5° misorientation. The prior austenite grains were reconstructed with ARPGE software [17], by assuming Kurdjumov-Sachs orientation relationship between the parent and product phases. The calculated average grain diameter (area fraction) is used as a representative number for the grain size. XRD analyses were performed by means of a Siemens Diffractometer D5000 with a molybdenum X-ray tube. The angular range of 25° – 40° was scanned with a step size of 0.05° and 5 s/step. Using the empirical formula of Cullity [18], the fraction of retained austenite was calculated from the diffraction data for different heat treatments. TEM-samples were prepared by means of a twin-jet electropolishing setup (Struers Tenupol-5) using a mixture of 4 vol% perchloric acid and 96 vol% acetic acid at room temperature. A JEOL JEM-2200FS FEG TEM operating at 200 kV was used to study the general microstructure and the compositional gradients in the microstructure by integrated EDX-STEM.

Thermodynamic calculations were carried out using Thermo-Calc with the TCFE6 database. The dissolution of the carbides after the

Table 1

Chemical analysis of the DIN 75Cr1steel (optical emission spectroscopy).

wt.%	Fe	С	Mn	Si	Cr	Cu	Ni	Мо	Al	Р
Comp.	Bal.	0.733	0.639	0.329	0.353	0.102	0.073	0.020	0.024	0.010

different austenitizing temperatures and times was simulated with Dictra [19-21] (version 25, TCFE6 database for thermodynamic data and MOB2 database for mobility data). The main goal of these calculations was to study the movement of the austenite-carbide interface and the compositional gradients in the microstructure. Since the advance of the austenite-ferrite interface is much faster than the austenite-carbide interface [22], it was assumed that the A_{CM} temperature corresponds to the full ferrite to austenite tranformation. Therefore, the nucleation process was not considered in the simulations. The presence of carbides and fully transformed austenite is assumed at the soaking temperature. The microstructural model with initial spherical representative volume and phase distribution is represented in Fig. 1. The initial radius of the carbides was set to be the mean radius and the total radius of the system was calculated from the area fraction (9 vol% of carbides), measured on the as-received microstructure. A local equilibrium is assumed at the interface and the reaction rate is calculated based on the solute flux of the alloying elements. The initial chemical composition and equilibrium fractions of ferrite and alloyed cementite were estimated with Thermo-Calc, at a temperature in the range of the ferrite and cementite metastable equilibrium. The starting chemical composition of austenite was considered to be equal to the composition of the initial ferrite at room temperature. Calculated volumetric phase fractions were estimated neglecting the effect of carbon. The alloyed cementite dissolution into austenite at the isothermal heat treatments were calculated at 800 °C, 820 °C, 840 °C, 860 °C, 880 °C and 900 °C for a maximum simulation time of 2000 s. Heating and cooling stages were discarded from the calculations.

Tensile specimens were prepared according to the ASTM standard E8M-04 (gage length 50 mm) with thickness 1.8 mm, samples were first machined and heat treated in the salt bath afterwards. Tensile tests were carried out on a Zwick Z250 testing machine in stress controlled mode with a speed of 20 MPa/s until the yield point, and 0.0067 mm/s cross head speed for strain controlled plastic deformation until fracture. The Izod impact tests were executed on a Zwick/Roell HT55P machine on sub-size samples with the geometry of elements of the CVT transmission described elsewhere [23]. The goal of the Izod impact test is to compare the impact toughness as a function of the parameters of the thermal treatment and to obtain a set of parameters that correspond to high values of both strength and impact toughness.



Fig. 1. Schematic representation of the austenite and carbide initial microstructure in the DICTRA simulations.



Fig. 2. Microstructure of the as-received material in the soft annealed condition: EBSD image quality (IQ) map and phase map superimposed.

3. Results

The microstructure of the as-received material in the soft annealed condition is visualized in Fig. 2. The EBSD map shows a bimodal distribution of spheroidized carbides (8 \pm 1%) in a ferritic matrix. Practically all carbides were indexed correctly as Fe₃C phase, which allows assuming that the carbide type in the steel is cementite alloyed with Cr i. e. (Fe, Cr)₃C. However optical microscopy displays a higher carbide fraction, see previous work [24]. The underestimation of the carbide fraction measured with EBSD in comparison to the optical microscopy data is most probably due to the smaller area of the EBSD scan.

The Ae₁ and Ae_{CM} equilibrium transformation temperatures calculated with Thermo-calc are 722 °C and 770 °C respectively and ferrite, cementite and austenite are in equilibrium in the temperature range between 722 and 734 °C, see previous work [24] for detailed information. According to the calculations, (Fe,Cr)₃C cementite is the stable carbide among the other complex carbides. At the annealing temperature of 734 °C, the equilibrium carbide fraction is 1.6%.

The kinetics of the transformation from the initial ferrite and spheroidized alloyed cementite to austenite during the austenitizing treatments and the subsequent quenching were studied with dilatometry. Note that the tensile and impact toughness samples were heat treated in a salt bath under the same conditions. The relative changes in length versus temperature for several heat treatments are shown in Fig. 3a. In the range of 750–770 °C, a slight deviation from linear behavior in the length versus temperature curve is observed during heating. This could be explained by the Curie-point or the precipitation of secondary carbides [25]. At 785 °C, the distinct reduction in length starts, indicating the start of the transformation of ferrite (and part of the carbides) to austenite. This transformation is completed at around 840 °C, therefore the samples heated to 800 and 820 °C had an incomplete ferrite to austenite transformation during the heating stage. The measured transformation temperatures by dilatometer are higher than



Fig. 3. Dilatometry curves: (a) relative change in length versus temperature for the austenitization and quenching treatment with holding time 2 min, (b) M_s versus austenitizing temperature for holding times 2 and 10 min, (c) relative change in length against holding time during austenitization.

the equilibrium temperatures determined with Thermocalc. Fig. 3b shows the measured M_s temperatures in function of soaking time and temperature. It can be observed that the M_s temperature decreases with increasing soaking time and austenitizing temperature. An

approximately linear decrease in M_s temperature with austenitizing temperature was also observed in Ref. [5]. This decrease can be explained by the more complete carbide dissolution at prolonged times and elevated temperatures, and hence increased carbon content in



Fig. 4. Reconstruction of the prior austenite grains with ARPGE software: 75Cr1 annealed (a) at 820 °C for 4 min and (b) at 900 °C for 30 min, quenched and tempered: EBSD color coded phase map. HAGB's > 15 °C are indicated with black lines. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article).

austenite which leads to a reduced M_s temperature. For all conditions, M_f is below room temperature. Accordingly, more retained austenite can be expected with increasing soaking time and temperature. Fig. 3c shows the relative change in length versus soaking time at different temperatures. For austenitizing temperatures of 800 and 820 °C, first a contraction can be observed, opposed to a clear expansion for the other temperatures. The contraction corresponds to the further transformation of ferrite to austenite, since austenite has a smaller molar volume than ferrite. The further expansion in the dilatometric curve is a delayed expansion due to temperature homogenization while the material is still expanding (typical for high heating rates). During austenitization this phenomenon is hidden due to large material contraction. The observed contraction after the expansion plateau at temperatures of 860 °C and higher, could be explained by austenite grain growth.

Fig. 4 displays the reconstruction of the prior austenite grains with ARPGE software. It is clear that an austenitizing temperature of 900 °C and a long soaking time lead to significant austenite grain growth. Fig. 5a and b shows the EBSD image quality map superimposed by the color-coded phase map of a sample annealed at 800 and at 900 °C for 4 min, followed by oil quenching and tempering. The carbide content and size is significantly higher for the material annealed at 800 °C. In comparison with the as-received material (Fig. 2), less and smaller carbides can be observed in these samples. For the material annealed at 900 °C, the carbide is very often surrounded by retained austenite, while this is rarely observed for the sample annealed at 800 °C. Some small regions of austenite in between martensitic plates (interlath retained austenite) could be distinguished for both measurements, however the austenite content is more than 10 times higher for the sample annealed at 900 °C.

The retained austenite fractions of selected samples were quantified from X-ray diffraction measurements and are plotted in Fig. 6a. In general, higher austenitizing temperatures and longer soaking times lead to a larger retained austenite fraction. Fig. 6b shows the average prior austenite grain size (PAGS), determined both with the linear intercept method and with ARPGE software (Fig. 4). The combination of high temperature and time leads to a large PAGS. As a consequence, the martensite grain (or block) size is also larger, Fig. 6c. A quantitative description of the dependence of carbide dissolution on time and temperature was obtained from Dictra simulations. The calculated volume fractions of the carbides are plotted in Fig. 7 and are compared with the ones quantified on SEM images. According to the model, the carbide fraction decreases with increasing austenitizing time and temperature. The same tendency is observed in the experimentally derived carbide fraction that decreases faster with increasing soaking time than the one predicted by the model until the annealing time of ~ 10 min. The experimental data show that the dissolution rate of the carbides, represented by the slope of the curves, decreases after 10 min.

The calculated carbon concentration profiles and the movement of the austenite/carbide interface in Fig. 8a and b shows the kinetics of carbide dissolution into austenite during various heat treatments, with 0 μ m corresponding to the initial interface and distance <0 to the initial carbide. It can be seen that the model predicts homogeneous distribution of carbon in the austenite while a small carbon spike is observed in the carbide close to the phase boundary. The concentration profiles of Cr at 860 °C for different holding times are presented in Fig. 8c. A significant chromium spike is noticed in the carbide close to the interphase, e.g. a Cr spike concentration of 25 wt% compared to the average of 3 wt% in the carbide. This simulation is confirmed by the Cr and Mn profiles across the interface measured with STEM-EDX (Fig. 9).

The effect of the soaking time and temperature on the yield strength of the tool steel is shown in Fig. 10. There is a general tendency of yield strength decrease when the austenitizing temperature increases. On the other hand, variations of the yield strength are observed for all austenitizing temperatures as a function of soaking time. In general, an increase of the yield strength is observed at short soaking times below 10 min. The Izod impact test results (Fig. 11) show that elevated austenitizing temperatures and prolonged soaking times lead to a decrease in toughness. The toughness decreases almost twice for all temperatures if the soaking time increases from 2 to 15 min.

4. Discussion

4.1. Austenite formation

The austenite start and finish temperatures obtained with dilatometer (cf. Fig. 3, resp. 785 and 840 °C) are higher than the ones calculated with Thermo-Calc (resp. 722 and 734 °C), as long-range diffusion of substitutional alloying elements is required to achieve full equilibrium conditions. Other factors affecting the kinetics of austenite formation temperatures can be linked to the heterogeneous chemical composition of the initial ferrite/alloyed cementite system and the diffusion controlled dissolution of the (Fe,Cr)₃C cementite. At around 840 °C, the transformation from ferrite to austenite is complete according to the dilatometer measurements, while the dissolution of alloyed cementite leads to a further change in length (cf. Fig. 3). In case of austenitizing temperatures below 840 °C (800 or 820 °C), still ferrite is present at the end of the heating stage, however even for the shortest holding time and lowest austenitizing temperature, all ferrite is transformed to austenite during isothermal holding.

4.2. Alloyed cementite dissolution

Dictra simulations show the movement of the cementite/austenite interface during austenitization at different temperatures. The simulations show very fast carbide dissolution at the early stages of heat treatment, which is related to the accumulation of Cr and to a smaller extent of Mn in the carbides (cf. Figs. 8c and 9). The Cr and Mn accumulation affects the carbon activity. The experimental data display



Fig. 5. Microstructure of 75Cr1 annealed (a) at 800 °C for 4 min and (b) at 900 °C for 4 min, quenched and tempered: EBSD image quality map with color coded phase map. K-S OR is indicated with white lines. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article).



Fig. 6. (a) Retained austenite fraction after different heat treatments, calculated based on XRD data, (b) average prior austenite grain size (PAGS) and (c) average martensite grain size (3 points at 4 min nearly coincide, therefore the points are represented at 4-/+a min) after different heat treatments based on EBSD measurements.



Fig. 7. Carbide fraction measured with ImageJ software on samples etched with picral 4% (points) and calculated with Dictra simulations (lines). For colors, please refer to the online version. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article).

similar tendency, although there is a discrepancy between experiments and simulations at longer soaking times (Fig. 7). Possibly, the contribution of smaller carbides cannot be accounted for and might partially explain the larger deviation at longer soaking times. In Ref. [26] it is reported that the size of carbide particles is log-normally distributed, and during the later stage of dissolution, the size and particle density do not decrease simultaneously, which is not incorporated into the model of this paper.

It is clear from the calculated concentration profiles in Fig. 8a and b that the homogenization of C in austenite is very fast. In contrast, the distribution of Cr (and Mn) in austenite and carbides is not homogeneous, indicating that the kinetics of carbide dissolution are controlled by the substitutional alloying elements. The diffusivity of C is around 1 \times 10⁶ times higher than the diffusivity of Mn at 900 °C [27]. The movement of the austenite-carbide interface requires extensive redistribution of Cr (and Mn) at the phase boundary during the carbide dissolution process. The enriching of these alloying elements is compared with Dictra calculations in Fig. 9. The measurements of the Cr content show good agreement with the calculations for bulk carbide. Nearby the interface, an increasing discrepancy is detected, probably partially caused by the electron beam size used for the composition analysis. The interacting volume of the TEM-slice and the electron beam nearby the interface includes a part of the matrix too, which averages the Cr content. The concentration of Mn measured by EDX is lower than



Fig. 8. Model of the position of the carbide austenite interface during carbide dissolution in austenite. (For colors please refer to the online version.) (a) C profile after 2 min and (b) C profile after 30 min at different temperatures, (c) Cr profile after austenitization at 860 °C with different holding times. 0 μ m corresponds to the initial interface (t = 0 min) for the 3 figures: <0 μ m initial carbide, >0 μ m initial austenite. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article).

expected both in the matrix and in the carbide, this might be a calibration error. Irrespective of the absolute measured values, it is clear that an accumulation of Mn nearby the interface was detected, concomitant with the general tendency of the calculated profile. Further accumulation of Cr and Mn in the carbides will continuously decrease the rate of carbide dissolution, as shown in Fig. 7. Most of the Cr diffuses into the carbides as the dissolution proceeds, which contributes to the enhanced stability of the carbides [28]. The decrease in the kinetics can be explained by the accumulation of Cr and Mn. It is reported that after complete dissolution of the alloyed cementite, the austenite will inherit a sharp Mn profile due to the initial Mn enrichment of carbide [27]. The carbide fraction in Fig. 7 shows that the most important changes take place during the first 10–15 min. Longer heat treatments might not produce significant variations in the microstructure, except for the austenite grain growth, Fig. 4.

4.3. Microstructure - mechanical properties relation

High austenitizing temperatures and long soaking times lead to a decrease in yield strength and impact toughness. Microstructural analyses of selected samples show that the retained austenite fractions are higher and the carbide fractions are lower when the austenitizing temperatures and soaking times increase. The retained austenite fraction after the final tempering as measured with XRD (Fig. 6a) is slightly higher than the one measured with EBSD (Fig. 5). This can be explained by several factors like the limited scanning area of the EBSD measurement, the mechanical polishing process that may transform part of the retained austenite into martensite and the limited resolution of the EBSD technique which does not allow detection of the interlath retained austenite. With increasing austenitizing temperatures and times, more carbides can dissolve in the austenite and correspondingly more carbon and alloying elements can go into solution. With an increasing amount of carbon in the martensite lattice due to longer soaking, one could expect stronger material but this is not the case; the higher fraction of retained austenite together with austenite grain growth, observed in Figs. 6b and 4, lead to a well-documented decrease in yield strength. For very short austenitizing times, full transformation to austenite of the entire sample cannot be guaranteed since the time at the austenitizing temperature corresponds to the time of contact in the salt bath at a certain temperature. For 800 and 820 $^\circ$ C, the dilatometer test showed that the transformation during heating is not yet complete but during soaking, ferrite further transforms to austenite (Fig. 3a, c).

The toughness properties show the same trend as the carbide fractions, Figs. 11 and 7. The spheroidal carbides do not crack or exhibit decohesion at small strain, therefore the spheroidized steel can withstand substantial deformation before voids are nucleated and exhibits good ductility [29]. The decreased toughness with elevated temperature and longer soaking time can also be explained by the higher carbon content in the martensite. The enrichment of the austenite matrix with carbon and alloying elements leads to a lower M_s temperature (Fig. 3b) and therefore more untransformed austenite will retain at room temperature [30]. Retained austenite, depending on its distribution in the microstructure and carbon content, influences the mechanical properties. In general, retained austenite is unwanted in tool steels [31]. The retained austenite is soft and can be unstable, i.e. it can transform to brittle martensite under service conditions, leading to dimensional changes and distortions. On the other hand, the retained austenite can contribute to the impact toughness and ductility by stress/strain induced transformation, which is the transformation induced plasticity (TRIP) effect. Moreover the thin film-like morphology of retained austenite between martensite plates can increase ductility and toughness [32,33]. However, martensitic structures formed after low soaking temperatures and short soaking times lead to higher strength (Fig. 10) and toughness (Fig. 11). A lower yield strength can be observed for microstructures with a high retained austenite fraction. The toughness should not only be linked to the retained austenite fraction in such type of



Fig 9. (a) and (b) STEM-EDX around carbides of 75Cr1 annealed at 860 °C for 15 min, quenched and tempered. (c) Average alloying element profiles measured with EDX and Dictra simulations at 860 °C for 15 min (<0 cementite, >0 austenite).



Fig. 10. Yield strength versus soaking time for different austenitizing temperatures (error bars are displayed only for results at 860 °C but for the other temperatures the values are comparable).



Fig. 11. Results from the Izod impact tests for the different austenitizing condition (error bars are displayed only for results at 900 °C but for the other temperatures the values are comparable).

microstructure; In general the toughness is dominantly associated to the grain size. For the studied material, the expected increase in toughness associated to the increase of retained austenite (\sim 8% after austenitization at 900 °C) is not observed due to larger prior austenite grain size (Figs. 4 and 6b) and hence larger martensite grains (Fig. 6c).

Hence the combination of high yield strength and acceptably good ductility/impact toughness could be obtained via quenching from temperatures in the range between 820 and 840 °C after short soaking times up to 8 min. High austenitizing temperatures and long soaking times lead to lower strength and lower ductility.

5. Conclusions

The changes in the microstructure and mechanical properties of the tool steel 75Cr1 were studied as a function of the austenitizing temperature and time. It was found that:

- Low austenitizing temperatures between 820 and 840 °C in combination with soaking times up to 8 min lead to increased strength and ductility compared to high austenitizing temperatures. This effect is associated with limited dissolution of the initial spherical carbides. A lower austenitizing temperature also leads to less carbon saturation of the austenite and a lower retained austenite fraction.
- The accumulation of Cr and Mn acts as a barrier for the dissolution of the alloyed cementite during austenitizing. This is confirmed with Dictra simulations for the used temperatures and soaking times.
- The calculations of the cementite/carbide dissolution profiles show good agreement with experimental data at early stages of the carbide dissolution. The most significant changes in carbide fraction are at early stages of the austenitizing. This is consistent with the variation in mechanical properties with respect to the soaking time.
- Austenitizing temperatures above 860 °C and soaking times longer than 10 min lead to a decrease in both yield strength and toughness. Soaking times longer than 15 min do not produce significant variations in the carbide fraction. Although a significant prior austenite grain size growth is observed at high austenitizing temperatures and long soaking times, a significant deterioration in mechanical properties is not observed.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

A. Verdiere: Writing - original draft, Investigation, Conceptualization, Visualization. F. Castro Cerda: Investigation. A. Béjar Llanes: Investigation. J. Wu: Investigation. L. Crebolder: Validation. R.H. Petrov: Supervision, Writing - review & editing.

Acknowledgements

The authors acknowledge the financial support of the Dutch Materials Innovation Institute (M2i) under the contract M21.10.11442.

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