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The effects of heat treatment variables on the microstructure and properties of ultra-high strength steels differing in titanium content

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The influences of different austenitizing and tempering temperatures on the microstructure and properties of three experimental ultra-high strength steels (UHS) have been investigated. The steels had different Ti content and were subjected to austenitizing treatment at 900, 1000, 1100 and 1200 °C followed by oil quench and tempering at 200, 300, 450 and 600 °C. It has been found that the high temperature (1100 and 1200 °C) austenitizing treatments, alter both microstructure and properties, and depending on the subsequent tempering temperature, may have a beneficial or detrimental influence upon the mechanical properties. Addition of up to 0.011 wt% Ti to the steel composition improves hardness, toughness and tensile strength. This improvement in mechanical properties is obtainable with any subsequent heat treatment. For higher Ti content (0.089 wt%), although some further improvement in hardness and tensile strength was obtained, significant degradation in toughness was achieved, particularly when the steel was subjected to high temperature austenitizing and tempering treatment.

1. Introduction

Ultra-high strength steels are a class of constructional steels with very high strength level. One of the steels in this class, with a nominal composition (wt %) C 0.38–0.43, Mn 0.75–1.00, Cr 0.80–1.10, Mo 0.15–0.25, is designated as AISI/SAE 4140. Conventional heat-treatment for this steel is austenitization at 830–870 °C followed by oil quench and tempering at two different temperature ranges, 175 to 230 °C and 370 to 675 °C for at least 0.5 h. The goal pursued through the development of this class of steels was a combination of toughness, strength and ductility. For this purpose many heat-treatment procedures have been proposed. Some of them are double austenitizing, i.e. high temperature followed by low temperature austenitizing treatment [1–4], high temperature austenitizing [5, 6], rapid heating austenitization [7] and duplex treatment consisting of two stages; first austenitizing and tempering at high temperature then applying the same treatment at conventional temperature [8]. In fact the desirable combination of strength, toughness and ductility can be achieved by establishing an accurate structure–property relationship [2, 4, 9, 10].

Mintz and co-workers [9, 10] have studied the relationship between the structure and properties of some ferrite–pearlite steels and recommended some new equations for determining the impact transition temperature.

Martensitic structure and its relation to properties, particularly toughness, has been extensively studied by many investigators [1, 11–20]. The effect of microstructural change on the mechanical properties of a specific type of high strength steel was investigated by Chang *et al.* [1]. They used various heat treatment methods, particularly the double austenitizing treatment, to find the relationship between impact, toughness and microstructure and found how the double austenitizing treatment helps to dissolve VC carbides and change the morphology of the other types of carbides in the form of M_3C, consequently altering the toughness of the steels investigated.

Grain boundary segregation of impurities such as P, S, Sb and their influence on temper embrittlement of low alloy steels was studied by Song and Xu [21]. They proposed a new model for a combined equilibrium and non-equilibrium segregation mechanism of temper embrittlement. Low temperature tempering of martensitic structures in the range of 200–400 °C was usually performed to achieve higher ductility and toughness and to keep desirable hardness, but in many cases, loss of toughness was observed at temperatures around 300 °C [22–24]. This embrittlement was defined as tempered martensite embrittlement (TME). Tempered martensite embrittlement was the research subject of many investigators and has been reported to be associated with two different transgranular and intergranular fracture modes [23–27]. Its mechanism was studied by Horn and Ritchie [24] for 4340 and (300-M) ultra-high strength steels. They questioned the claimed increase in toughness due to the presence of the retained austenite in as-quenched steels, and expressed the opinion that the evidence regarding this issue is inconclusive. They explained that the severity of tempered martensite embrittlement is directly related to the volume fraction of interlath retained austenite and this was found to be the source of interlath cleavage in microstructure during mechanical fracture. A new mechanism of TME involving thermal decomposition of interlath retained austenite and precipitation of interlath cementite was also proposed by some researchers [14, 24–26]. The precipitation of brittle cementite on grain and lath boundaries was found to be the essential feature of tempered martensite embrittlement [24]. It was also reported that for the steels containing a low level of impurities and retained austenite, the fracture mode is transgranular cleavage but for high amounts of impurities and retained austenite, the fracture mode is intergranular [24] and this is because the impurities tend to segregate at pre-existing austenite grain boundaries during austenitization and tempering treatment, particularly in the range of tempered embrittlement. Retained austenite also decomposes and produces Fe_3C, so the combination of impurities and cementite precipitated at pre-existing austenite grain boundaries seems to be the main reason for intergranular fracture.

The purpose of the present investigation was to reassess the effect of austenitizing and tempering temperature on both the microstructure and mechanical properties of ultra-high strength steels and also to find out how the structure and properties can be affected by titanium as an alloying element.

2. Materials and experimental procedures

2.1. Materials

The materials investigated were three steels designated as C-4140, T1-4140, T2-4140, and of chemical composition as shown in Table I. The steels were prepared by vacuum arc remelting and received as hot-forged in the form of 70 mm square bars. Specimens for mechanical testing were prepared in a manner to examine the mechanical properties in the transverse direction of the bars.

2.2. Heat treatments

Different austenitizing temperatures, namely, 900, 1000, 1100 and 1200 °C, were used for heat treatment of specimens prepared from all the investigated steels. Each specimen was austenitized followed by oil quench then tempered at 200, 300, 450 and 600 °C for 3 h. All specimens were about 2 mm oversized from the final form and were finally machine-finished after heat treatment.

2.3. Mechanical testing

DIN-50125 standard tensile tests were carried out for measurement of yield stress (YS) and UTS. Yield stresses were measured by 0.2 per cent offset and the extension rate was $2\ \mathrm{mm\,min^{-1}}$.

ASTM TGL 11225 standard Charpy 2 mm V-notched (CVN) impact and Rockwell C scale hardness tests were carried out for measurement of impact energy and hardness values. At least five measurements for each tensile and CVN and six measurements for hardness were carried out at room temperature to determine the YS, UTS, CVN energy and hardness values of each specimen.

2.4. Metallography

Light optical microscopy was utilized for microstructural examination, particularly for observation of pre-existing austenite grain size and grain boundaries and also martensitic structures. The etching solution was a mixture of 100 ml picric acid, 80 mg cupric chloride and 60 ml wetting agent [28]. Etching time was almost one minute for all specimens.

Scanning electron microscopy (SEM) together with energy dispersive spectrometry (EDS) and wavelength dispersive spectrometry (WDS) were performed for fractography and analysing carbides and inclusions observed in the microstructures and at fracture surfaces.

3. Results and discussion

3.1. The influence of heat treatment variables on the microstructure

Microstructures of steels austenitized at different temperatures are illustrated in Fig. 1. As can be seen austenite grain sizes are significantly increased with increasing austenitizing temperature, as expected. Increasing austenitizing temperature not only increases the grain sizes, but also dissolves most of the dispersions, particularly secondary carbides inherited from the processing history of the steel; so by raising the austenitizing temperature the alloying elements remain in solution with austenite and decrease the M_s temperature leading to more retained austenite in the microstructure [13, 29]. Dissolution of carbides and other dispersions by applying high temperature austenitizing treatments is based upon the thermodynamic stability of such compounds. Particles with low thermodynamic stability such as $M_{23}C_6$ carbides are completely decomposed during high temperature austenitization and those with high thermodynamic stability slightly dissolved and only their morphology

TABLE I The chemical compositions (wt %) of the experimental steels

	C	Si	Mn	Cr	Mo	Ti	Ni	P	S	Al	V
C-4140	0.450	0.260	0.660	1.100	0.240	0.000	0.049	0.016	0.008	0.008	0.005
T1-4140	0.380	0.250	0.670	1.110	0.250	0.011	0.050	0.014	0.007	0.007	0.006
T2-4140	0.420	0.230	0.650	1.080	0.230	0.089	0.052	0.018	0.009	0.008	0.007

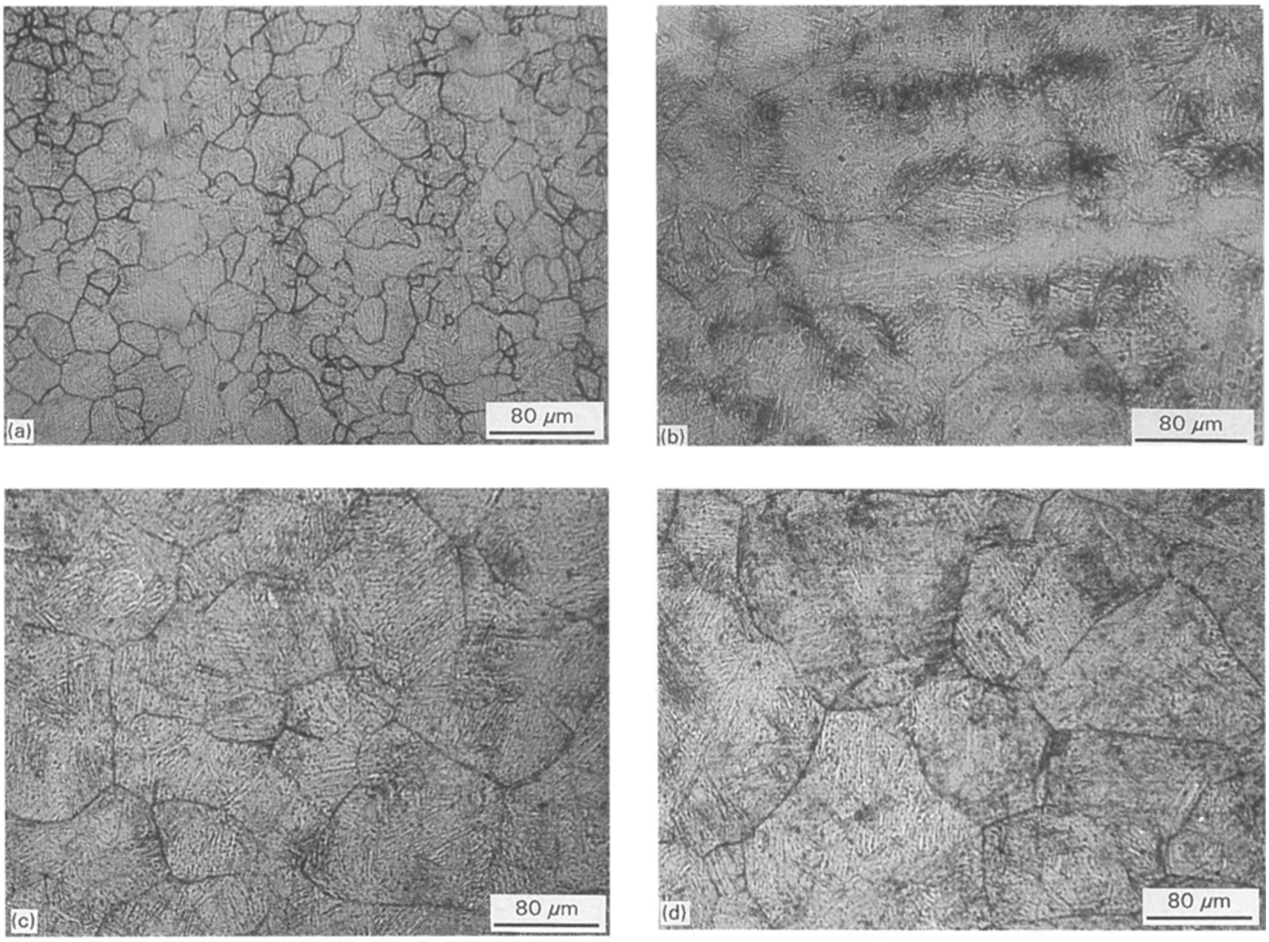

Figure 1 Initial austenite grain size of the C-4140 steel austenitized at: (a) 900 °C, (b) 1000 °C, (c) 1100 °C, and (d) 1200 °C.

may be changed to spherical during post austenitizing (long time austenitizing) treatment [30].

Partial or complete dissolution of eight primary or secondary dispersions by means of raising the austenitizing temperature can lead to substantial alteration of the microstructures as well as the properties of the steel. This may be due to phenomena such as void nucleation resistance, grain-refining, particle spacing and distribution in the microstructure of the steel. It has been found that the fine-scale secondary carbides serve as grain-refining dispersions as well as suitable sites for the nucleation of microvoids [30].

In the present investigation it has been found that during austenitizing and tempering treatment, particles containing Ti have different effects on the microstructure and properties of ultra-high strength steel. It has already been demonstrated that dispersions containing Ti are initially formed within the molten steel before solidification starts [31]. These particles will grow later and may interact with other particles and impurities throughout both freezing and solid state cooling sequences so they have been referred to as primary dispersions. Primary dispersions not only remain almost unchanged in conventional austenitizing, but also undissolved even at high temperature austenitizing, therefore high temperature austenitizing dissolves almost all carbides except primary dispersions which are thermodynamically stable [30]. Later it will be discussed that small amounts of titanium can alter the composition of sulfides in ultra-high strength steels, increase void nucleation resistance, and consequently improve the mechanical fracture properties of the steel.

Primary and secondary dispersions have both beneficial and detrimental effects on the microstructure and properties of ultra-high strength steels. It is understood that both of them pin migrating grain boundaries, preventing coarsening and acting as grain refining agents which result in the improvement of mechanical properties [30, 32, 33]. Conversely, these particles can be suitable sites for microvoid nucleation. This can be seen in the fracture surface presented in Fig. 2 which clearly demonstrates a particle within a void, documenting microvoid nucleation during mechanical fracture of the steel.

3.2. Mechanical properties

Figs 3 and 4 show the tensile properties of the C-4140 steel austenitized and tempered at different temperatures. It can be seen that UTS and YS are decreased with increasing austenitizing temperature up to 1100 °C which is believed to be due to the increasing grain sizes and decreasing grain boundaries as shown in Fig. 1. Apart from the steel austenitized at 1200 °C and tempered at low temperature, higher tensile properties, particularly UTS, were observed. The reason for this seems to be dissolution of the secondary dispersions, particularly chromium and molybdenum carbides ($Cr_{23}C_6$ and Mo_2C) which led to higher

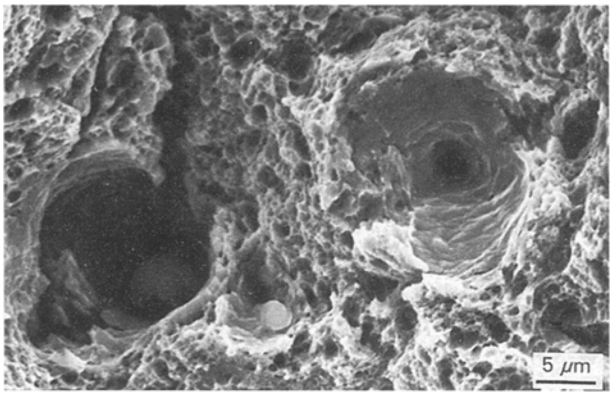

Figure 2 Fracture surface of T2-4140 steel austenitized at 1200 °C and tempered at 300 °C.

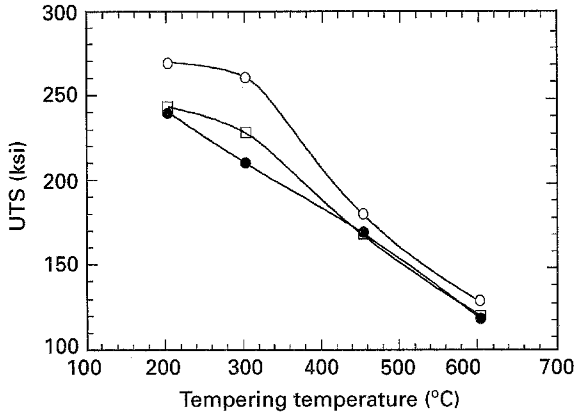

Figure 5 UTS versus tempering temperature for steels austenitized at 1100 °C. Key: -●- C-4140; -□- T1-4140; -○- T2-4140.

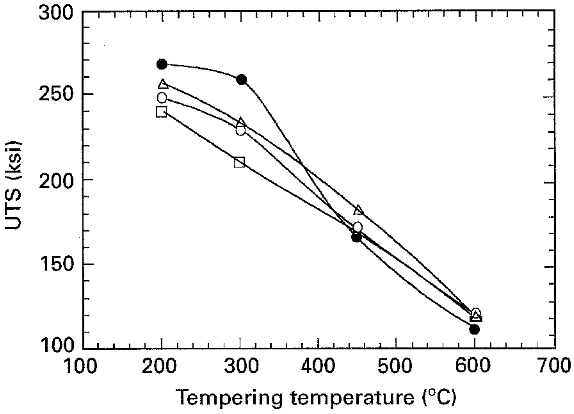

Figure 3 UTS versus tempering temperature. Key: austenitizing temperature, -●- 1200 °C; -□- 1100 °C; -○- 1000 °C; -△- 900 °C.

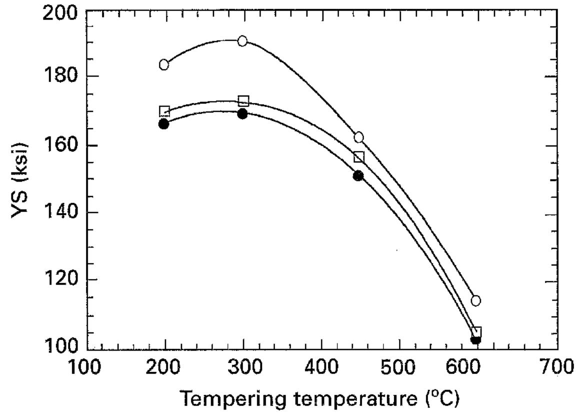

Figure 6 YS versus tempering temperature for steels austenitized at 1100 °C. Key: -●- C-4140; ; -□- TI-4140; -○- T2-4140.

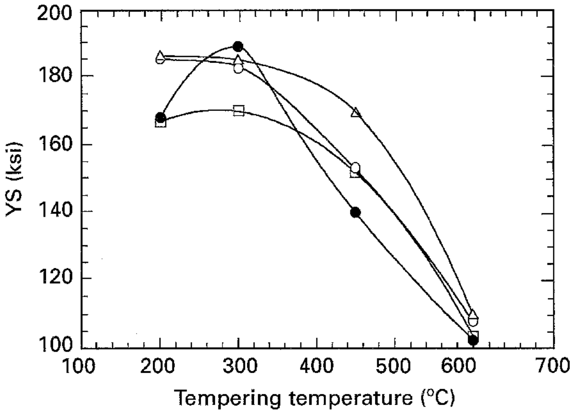

Figure 4 YS versus tempering temperature. Key: austenitizing temperature, -●- 1200 °C; -□- 1100 °C; -○- 1000 °C; -△- 900 °C.

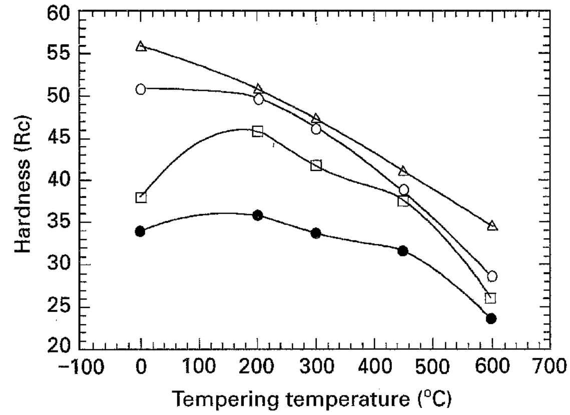

Figure 7 Hardness versus tempering temperature. Key: austenitizing temperature, -●- 1200 °C; -□- 1100 °C; -○- 1000 °C; -△- 900 °C.

amounts of retained austenite, enriched in C and other alloying elements. UTS and YS for all investigated steels austenitized at 1100 °C and tempered at different temperatures are illustrated in Figs 5 and 6. For T1-4140 steel containing small amounts of titanium, the tensile properties are slightly increased, but for T2-4140 steel, higher YS and UTS were observed, these are due to the presence of the strong titanium carbides within the microstructure of the steel.

Fig. 7 plots the hardness of C-4140 steel austenitized at different austenitizing temperatures as a function of tempering temperature. This figure shows a significant decrease in hardness due to the high temperature austenitizing (HTA) treatment. In the as-quenched condition (0 °C tempering temperature in the figure) the hardness of steel austenitized at high temperature (1200 °C) is much less than that for low temperature austenitization (900 °C). The influence of HTA on the hardness reduction can be better documented by comparing the differences between the hardness of as-quenched steels austenitized at 900, 1000 and 1100 °C. By raising the austenitizing temper-

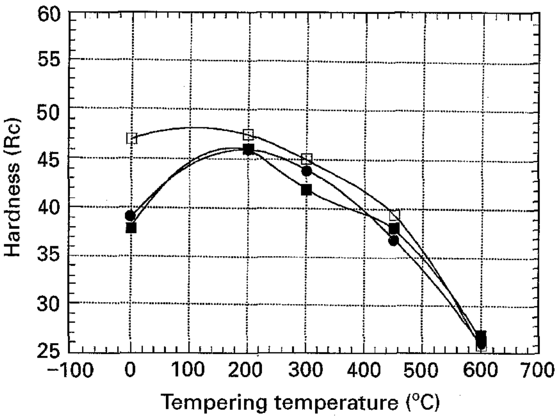

Figure 8 Hardness versus tempering temperature for steels austenitized at 1100 °C. Key: -■- C-4140; -●- T1-4140; -□- T2-4140.

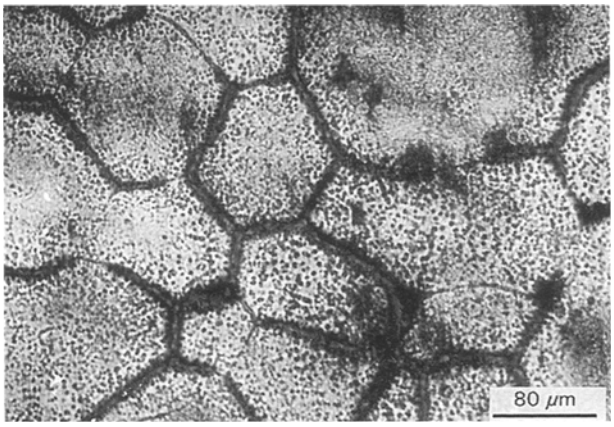

Figure 10 Grain boundary carbide distributions in T2-4140 steel austenitized at 1200 °C and tempered at 300 °C.

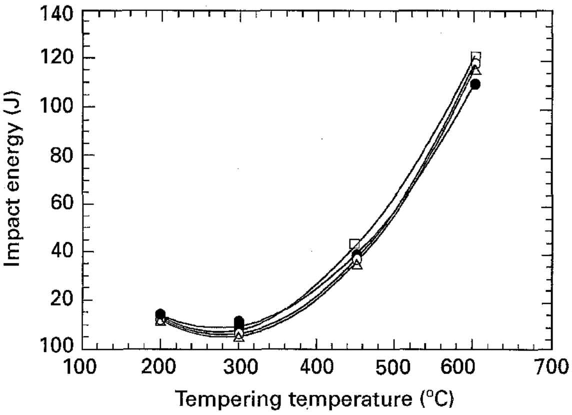

Figure 9 Charpy impact energy versus tempering temperature. Key: austenitizing temperature, -●- 1200 °C; -□- 1100 °C; -○- 1000 °C; -△- 900 °C.

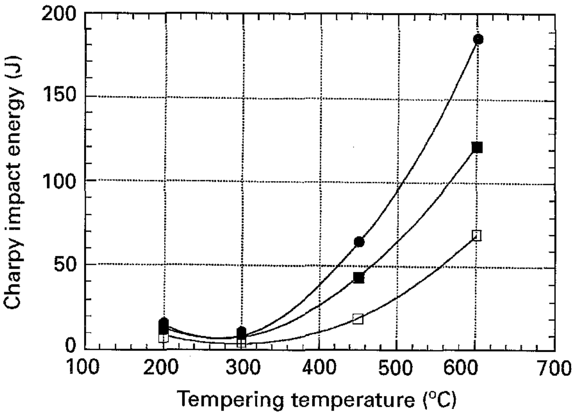

Figure 11 Charpy impact energy versus tempering temperature for steels austenitized at 1100 °C. Key: -■- C-4140; -●- T1-4140; -□- T2-4140.

ature from 900 to 1000 °C, hardness dropped only 5 RC but this reduction in hardness is more than double (13 RC) for increases in austenitizing temperature from 1000 to 1100 °C which is believed to be due to the increasing amount of retained austenite as well as the austenite grain sizes. The slope change of the curves can be related to the transformation of retained austenite to ferrite and cementite which is reported to be in the form of thin films located at the interface between the martensite laths [13, 14, 23]. For those steels containing more retained austenite, transformation of austenite produces more cementite and other carbides which prevent sharp reduction in hardness during tempering in the range of 300 to 600 °C. This is clearly demonstrated in Fig. 7 where the slope change of the curve for steel austenitized at 1200 °C (representing HTA) is less than that for low temperature austenitization (900 °C) (compare the hardness reduction for tempering temperature 0 and 600 °C). Fig. 8 shows hardness values versus tempering temperature for the steels containing different amounts of Ti and austenitized at 1100 °C. The hardness is increased slightly for T1-4140 and more for T2-4140 steel compared to C-4140 steel. This is due to the presence of strong and very hard titanium carbides and other titanium content compounds, which are hardly affected by heat treatment even by the high temperature autenitizing treatment.

Fig. 9 shows Charpy V-notch impact energy for C-4140 steel heat treated at different austenitizing and tempering temperatures. Except for 1200 °C, the impact energy is increased by increasing austenitizing temperature. This can also be related to increasing grain size and decreasing grain boundaries (see Fig. 1) which lead to higher toughness values, except for steels austenitized at 1200 °C due to the high amount of retained austenite which, in fact, is enriched in carbon and other alloying elements. During tempering at 600 °C more carbon is available for extensive carbide formation along the pre-existing austenite grain boundaries, similar to those shown in Fig. 10, as well as the lath boundaries [14]. This can eventually lead to degradation in toughness, hardness and UTS as shown in Figs 9, 7 and 3, respectively.

Fig. 11 plots the Charpy impact energy of all investigated steels austenitized at 1100 °C versus tempering temperature. It can be seen that for steel containing smaller amounts of titanium (0.011 wt %) the Charpy impact energy is more than that for C-4140 and also increases with increasing tempering temperature. For steel that has more titanium (0.089 wt %), the impact energy is not as great as that for C-4140 steel. This difference in toughness could be the result of one or more variables and phenomena such as volume fraction of dispersions (carbides, carbonitrides, carbosulfides), particle size, particle spacing and distribution, interaction between alloying elements and impurities during heat treatment which can alter the microstructure as well as the mechanical properties, particularly the toughness of steel.

According to the data obtained from the present investigation and the results presented by other researchers [30, 34] primary dispersions (carbides, nitrides, sulfides and oxides) inherited from the solidification, and secondary dispersions (carbides and other compounds) inherited from the solid state processing history of the steel, have significant influence on the mechanical properties, particularly toughness due to the void nucleation and grain refinement phenomena. Different toughness observed in Fig. 11 as a result of different Ti content can be related to the void nucleation and the effect of Ti on inclusions such as MnS in the microstructure of steel. Garrison and his coworkers [34, 35] have pointed out that gettering the sulfur as Ti_2CS rather than other ordinary sulfides such as MnS, CrS and La_2O_2S (which are usually found in ultra-high strength steels) can substantially improve the fracture toughness and this is attributed to the characteristics of Ti_2CS particles which are more resistant to void nucleation compared to other sulfide particles. In the present work improvement of toughness due to small amounts of Ti addition, is in good agreement with their results. Although the reason is not so clear, some assumptions in this regard can be summarized as; due to the high activity of Ti, the cohesive strength of the particle–matrix interface for particles containing Ti is much more than that for ordinary metallic sulfides so particles containing Ti in steel with small amounts of Ti (less than 0.011 wt %) are more resistant to void nucleation phenomenon. Other factors that are believed to affect the toughness are particles size, and particle spacing and distribution. Since Ti is a strong carbide forming element and tends to form MC-type carbides which are finer than M_2C carbides [31, 35], for low amounts of Ti (less than 0.011 wt %) the finer and more evenly spaced MC-type carbides can result in the improvement of toughness, but for higher Ti content the coarser, closely spaced and sometimes heterogeneous distribution of Ti-containing particles such as TiC, Ti(CN) and Ti(CS), decrease void nucleation resistance, weakening the particle–matrix interface and favour microcrack initiation and growth, consequently decreasing the steel toughness.

3.3. Tempered martensite embrittlement (TME) and assessment of fracture surfaces

TME has been investigated by carrying out Charpy V-notch testing for steels tempered at 300 °C. As shown in Figs 9 and 11, the impact energy for all investigated steels decreased to a minimum level for a tempering temperature of 300 °C. The mechanisms of fracture associated with TME have been extensively discussed by many researchers [14, 23–27]. One of these mechanisms, describing the phenomenon as the segregation of impurities and formation of different types of carbides at prior austenite grain boundaries, causes intergranular fracture. The microstructure of C-4140 steel tempered at 300 °C for 3 h, which is presented in Fig. 10, clearly demonstrated that the carbides segregated at prior austenite grain boundaries. The fracture surface of this steel is shown in Fig. 12. The intergranular fracture mechanism is quite obvious in this micrograph, and is undoubtedly due to the segregation of different carbides at pre-existing austenite grain boundaries. As can be seen in Fig. 11, no significant change in TME (tempering temperature 300 °C in the figure) has been observed for the steel containing low amounts of Ti (0.011 wt %). This may be as a result of the paradoxical beneficial and detrimental influence of Ti on the toughness of steel tempered at 300 °C. The beneficial influences of Ti are; pinning migrating grain boundaries and depleting them from sulfur by forming Ti(CS), together with increasing void nucleation resistance which tends to increase toughness at 300 °C. The detrimental influence is; the formation of TiC, Ti(CN) and Ti(CS) particles which are suitable sites for void nucleation. These particles can also precipitate at pre-existing austenite grain boundaries lowering the toughness and causing the severity of TME. So this paradox in the influence of Ti, may be the reason for no change in the toughness of steel containing low amounts of Ti (0.011 wt %) compared to C-4140 steel. The detrimental influence of Ti is dominant and is more evident when steel has a higher amount of Ti and is tempered at 300 °C. In this case, microvoids nucleate on the coarse and brittle TiC, Ti(CN) and Ti(CS) particles present causing more severity of TME. This is clearly demonstrated in Fig. 2 which is a typical micrograph representing the microvoid nucleation phenomenon for the mechanical fracture of the steels investigated.

Figure 12 Fractograph surface of C-4140 steel austenitized at 1200 °C and tempered at 300 °C.

Although some aspects of the influence of Ti on the microstructure and properties of ultra-high strength steel are almost clear, much more work needs to be carried out to resolve all the issues regarding the presence of Ti in the structure of ultra-high strength steels.

4. Summary and conclusions

1. High temperature austenitizing treatment can drastically change both microstructure and properties of 4140 ultra-high strength steels.

2. By raising the austenitizing temperature austenite grain sizes are significantly increased, and carbon and other alloying elements remain in solution with austenite leading to more retained austenite. During tempering in the range of 450 to 600 °C, the retained austenite which is enriched in C and other alloying elements, provides more C for extensive carbide formation along the pre-existing austenite grain boundaries.

3. High temperature (1200 °C) austenitizing treatment followed by tempering at 600 °C, decreases UTS, hardness and toughness of steel. Degradation of hardness can be observed at any tempering temperature but improvement in tensile properties and toughness was observed for low tempering temperatures (less than 450 °C).

4. Ti poses a metallurgical paradox, having both beneficial and detrimental influences on both microstructure and properties of UHS. The beneficial influence of Ti is dominant particularly for toughness improvement, when small amounts of Ti (less than 0.011 wt %) is added. The mechanism of toughness improvement is not very well documented but it seems that Ti can interact with the inclusions present producing some new particles such as Ti(CN) and Ti(CS) which are more resistant to void nucleation.

5. More Ti addition (0.089 wt %) forms strong, hard and closely spaced TiC and other Ti containing particles throughout the microstructure, improving the hardness and tensile properties, but degrading the toughness of the steel. Degradation of toughness is believed to be due to the coarser, closely spaced and sometimes heterogeneously distributed TiC, Ti(CN) and Ti(CS) particles, which decrease void nucleation resistance, weaken the particle–matrix interface and favour microcrack initiation and growth.

6. Although the influence of Ti on the microstructure and properties of UHS is partially clear, many more aspects remain unresolved. Thus much more work is required to resolve all these issues regarding the role of Ti in UHS as an alloying element.

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