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ICM11

# Influence of heat treatment on the fracture toughness and crack propagation in 5% Cr martensitic steel

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## Abstract

The high strength of 5% chromium steels at elevated temperature is directly related to their complex microstructure generated by specific heat treatments that consist of thoroughly controlled austenitizing, quenching and tempering. Fracture toughness and fatigue crack propagation are studied for various heat treatments. It is shown that changing the austenitizing temperature, i.e. the grain size and morphology of primary carbides, does not change the fracture characteristics. However, modifications of tempering, directly impacting the alloy hardness, result in drastic changes of fracture toughness and crack propagation rates. As no change in grain or martensitic lath morphology is observed, the associated modification of the Paris law coefficients is attributed to a change in the distribution and volume fraction of nanometric carbides investigated by Small Angle Neutron Scattering.

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Keywords: Fracture toughness; Fatigue crack propagation; Hardness; Austenitizing temperature; Microstructure

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## 1. Introduction

Outstanding properties of martensitic steels used for applications at high temperature result from their complex microstructure obtained by dedicated heat treatment including thoroughly controlled austenitizing, quenching and tempering. Depending on the final application, the parameters of the heat treatment will be adjusted to seek for appropriate mechanical strength and toughness. Optimizing the compromise between these two mechanical characteristics remains of utmost concern and requires controlling the microstructure at different pertinent scales from the nanometric precipitation of hardening carbides to the microscopic features including grain size, primary carbides distribution and martensitic laths morphology.

Whereas the microstructural parameters controlling the yield strength and the fatigue limit at high temperatures are relatively well identified, the relevant characteristics that influence the fracture toughness and crack propagation are not well understood. Namely, it is not clear whether the mechanisms of crack propagation are controlled by nanometric or micrometric scale structural features.

In previous studies [1, 2] dealing with fracture toughness of tool steels, the results obtained show that the large numbers of inclusions within the highly strained region (plastic zone) in front of the crack affect the fracture toughness. From other respects, the increase of the austenitizing temperature results in the fracture toughness improves [1, 2, 3]. H.Nilsson [3] studied the influence of the austenitization temperature and the cooling rate on the toughness of the hot-work tool steels H11 and H13.

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The results obtained are correlated with the corresponding modifications of microstructure. The impact toughness and the elevated temperature ductility are affected by the formation of grain-boundary carbides during cooling.

The influence of austenitizing and tempering temperatures on the hardness and fracture toughness of hot-work H11 tool steel was also investigated in [4, 5]. It is shown that the microstructure can be substantially modified by vacuum heat treatment. In the range of tempering temperatures from 580 to 610°C, the toughness increases up to 25% and a slight decrease is observed at 620°C. In contrast, the hardness values remain constant for the whole range of tempering temperature. In other words, the fracture testing method used is sensitive to changes due to variations in the microstructure as a consequence of different austenitizing and tempering temperatures as well as of the homogeneity of the steel.

The literature concerning the effect of heat treatment on the crack propagation rates is abundant. More research works are focused concentrated on the effect of various testing conditions (load, temperature) or the geometry of the specimen test (thickness) on the fracture toughness. Conversely, the heat treatment effect is not so oftenly investigated. Experimented results concerning the influence of the hardness level and austenitizing temperature on the values of fracture toughness and crack propagation rate are presented and discussed. Three levels of hardness, namely 42, 47 and 50HRC and two austenitizing temperatures, 990 and 1050°C, were studied for the hot work tool steel X38CrMoV5 (AISI H11).

## 2. Experimental details

### 2.1. Material and microstructure

X38CrMoV5 is a martensitic steel with hardness in the range 40- 56 HRC depending on the parameters of the heat treatment, in particular the tempering temperature and time. A satisfactory hardness is generally retained up to relatively high temperature, namely 600°C. High mechanical strength and good resistance to thermal shock are provided by the presence of chrome, molybdenum and vanadium. Steel, whose composition is given in table 1, is supplied by Aubert & Duval.

Table 1. Chemical composition of X38CrMoV5 ( weight %).

	C	Cr	Mo	Mn	Ni	Si	V
X38	0.36	5	1.2	0.3	0.1	0.34	0.5

Fig 1a shows the prior austenitic grain structure of the material. The average grain size is 22µm [6]. Within grains, martensitic laths, decorated by different types of precipitate, can be easily observed by TEM using the replica technique (Fig1b). Using both X-ray diffraction and electron micro-diffraction, these precipitates are identified as lengthened  $(Fe,Cr)_3C$  carbides, MC spherical carbides and  $M_7C_3$  angular carbides. Diameters of spherical and angular particles range respectively from 0.05 µm to 0.5µm and from a few nanometers up to 0.1µm [7].

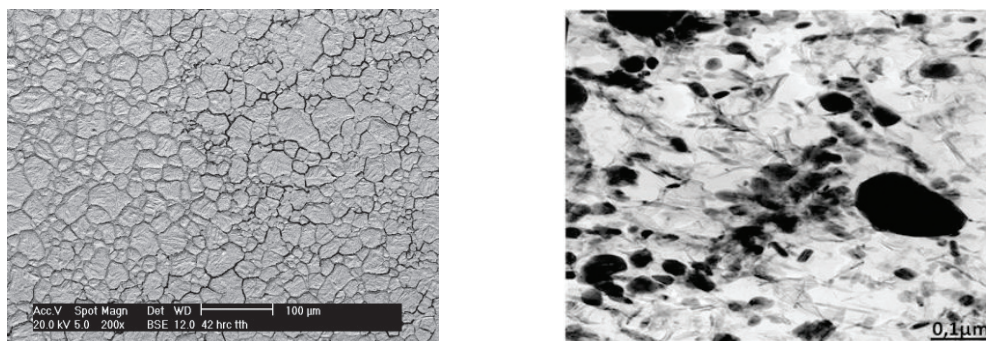


Fig. 1. (a) SEM image of grain structure in X38CrMoV5, (b) TEM micrograph showing carbide precipitation

## 2.2. Heat treatment

Subsequently to thermomechanical processing, X38CrMoV5 steel is annealed to homogenize the structure constituted of  $\alpha$  ferrite and carbides. Following quenching, a first tempering releases the residual stress and results in an extensive precipitation of secondary carbides. Different heat treatments and their influence on the fracture characteristic of the material are investigated. Details of the various austenitizing and tempering temperatures as well as the resulting hardness are given in table 2.

Table 2. The various investigated heat treatments and the resulting hardness

Austenitizing temperature (°C)	1 <sup>st</sup> temper (°C)	2 <sup>nd</sup> temper (°C)	Hardness (HRC)
990	550	625	42
990	550	603	47
990	550	595	50
1050	550	605	47

## 2.3. Procedures

To estimate the steel resistance to the propagation of a crack, toughness tests on standard CT specimens are generally used. CT specimens are machined from a cylindrical section with the principal strain parallel to the longitudinal axis of the cylinder. According to ASTM (E399-06 and E1820-01) standards, edges of specimens are machined along the circumferential and radial direction. These are C-R type specimens where the first letter stands for the perpendicular direction to the plane of crack and the second indicates the direction of crack propagation. Toughness and fatigue crack propagation tests are carried out in air at room temperature using a 250 KN servo-hydraulic machine MTS. Crack opening displacements are measured by an extensometer fixed at the notch of the specimen tested. Three tests are performed for each investigated set of parameters.

### 2.3.1. Fracture toughness test

Experiments are performed using standard compact CT25 specimens with width  $W = 50\text{mm}$ , thickness  $B=25\text{mm}$  and initial crack size  $a=20\text{mm}$  according to ASTM (E399-06 and E1820-01) standard [8, 9]. To avoid notch perturbation on crack tip plasticity, pre-cracking is performed in three steps with gradually lowered load. The initial load was calculated to initiate the pre-crack with a stress intensity factor of  $22 \text{ MPa}\cdot\text{m}^{1/2}$ . Fatigue loading is performed with a load ratio  $R$  of 0.1 and a frequency of 30Hz. The load at the end of pre-cracking ( $a/W=0.5$ ) corresponds to a stress intensity factor of  $15 \text{ MPa}\cdot\text{m}^{1/2}$ . After pre-cracking, a quasi-static tension up to fracture is applied to the specimens.

### 2.3.2. Crack propagation test

Tests are performed using CT25 specimens with a reduced thickness  $B = 12\text{mm}$  and initial crack size  $a = 10\text{mm}$  according to ASTM (E 647-08) standard [10]. The specimens are pre-cracked at a high frequency of 20Hz with a load gradually lowered from 14KN and 6 KN up to a crack length of 3 mm. Fatigue crack propagation tests are performed with a constant load of 6 KN until final crack of about 40mm is obtained at a lower frequency of 5Hz.

## 3. Results and discussion

The objective of the paper is to study how the austenitizing and tempering temperatures influence the fatigue crack propagation and toughness. Indeed, these two parameters have a strong impact on the metallurgical structure of the steel and in turn, on the fracture properties. Particularly, they modify and control the structure at various levels from:

- The microstructural scale, e.g. the size of grains and martensitic laths, the morphology and volume fraction of primary carbides, for the austenitizing temperature.

- The nanostructural scale, e. g. the secondary carbides as well as the dislocation structure inherited from the quench, for the tempering temperature.

However, note that the austenitizing treatment can also change the volume fraction of secondary carbides by modifying the quantity of solute atoms in austenite prior to quenching. Consequently, austenitizing and tempering are somewhat interdependent and may have synergetic effects both on the microstructure and the fracture characteristics. Micro- and nanoscale investigations are performed using complementary techniques such as Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Small Angle Neutron Scattering (SANS).

### 3.1. Fracture surface morphology

SEM observations show that the crack surface consists of four different successive (Fig.2) zones, namely i) the region of pre-cracking ( $a/W=0.26$ ), ii) a zone without striations near pre-cracking, iii) a zone with clearly visible fatigue striations (from  $30\text{MPa}\cdot\text{m}^{1/2}$ ) and iv) the zone of final rupture.

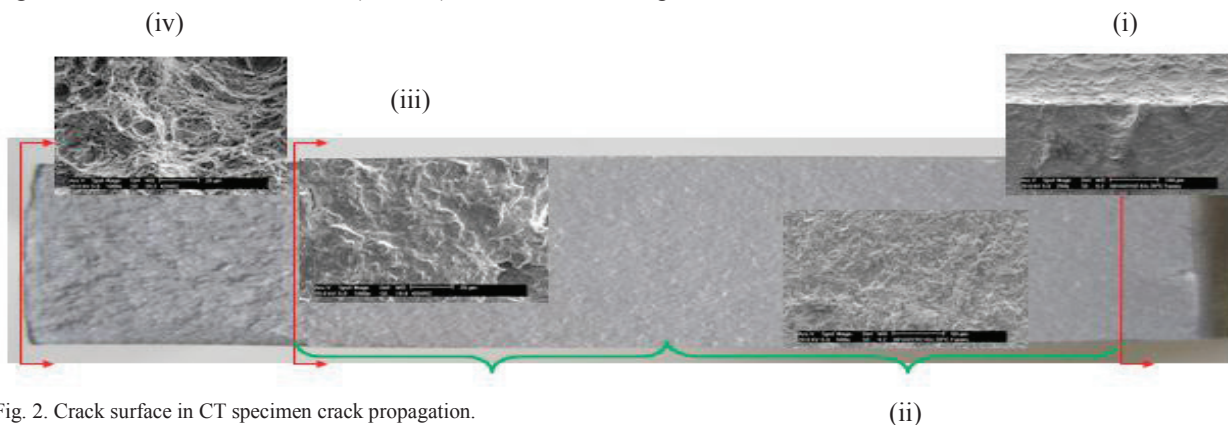


Fig. 2. Crack surface in CT specimen crack propagation.

The various zones were observed and measured. A significant difference is noticed for the length of the final rupture zone which is more important when hardness increases. Moreover, this zone corresponds to a more ductile fracture for 42HRC steel as compared to the 50 HRC grade (important concentration of cupules). The thorough observation of each zone does not reveal a significant difference between the various tested specimens.

### 3.2. Influence of austenitizing conditions

The grain size and the martensitic lath size straightforwardly increase as the austenitizing temperature increases. This is illustrated in Fig. 3 showing that grain size increases from  $22\ \mu\text{m}$  to  $127\ \mu\text{m}$  when the austenitizing temperature is enhanced from  $990^\circ\text{C}$  to  $1050^\circ\text{C}$ .

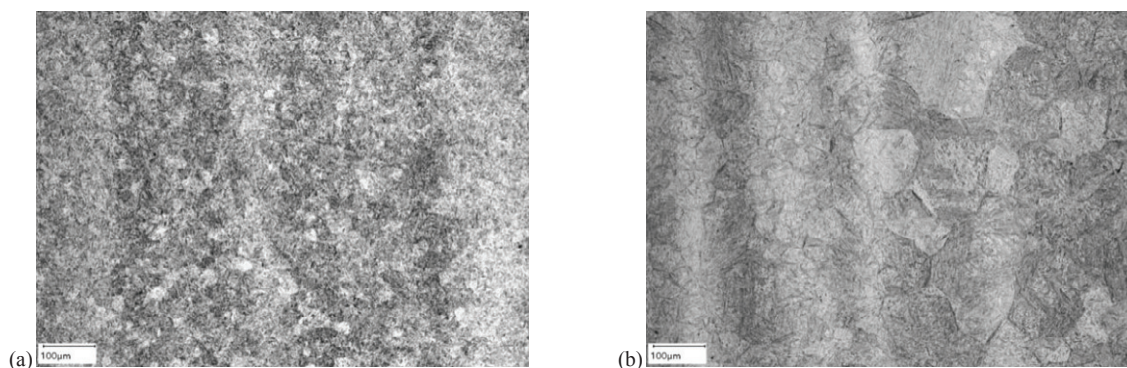


Fig. 3. Grain size resulting from an austenitization at  $990^\circ\text{C}$  (a) and  $1050^\circ\text{C}$  (b).

Fig. 4 details XRD profiles for steel austenitized at 990°C and 1050°C. Austenitizing at 990°C leads to the formation of primary carbides  $V_4C_3$ ,  $Cr_{23}C_6$  and  $Cr_7C_3$ , Austenitization at higher temperature, typically 1050°C, results in an enhanced dissolution of carbides in the austenitic matrix.  $Cr_{23}C_6$  and  $Cr_7C_3$  dissolve completely and only  $V_4C_3$  carbides, very slightly dissolved due to their high thermal stability, are present.

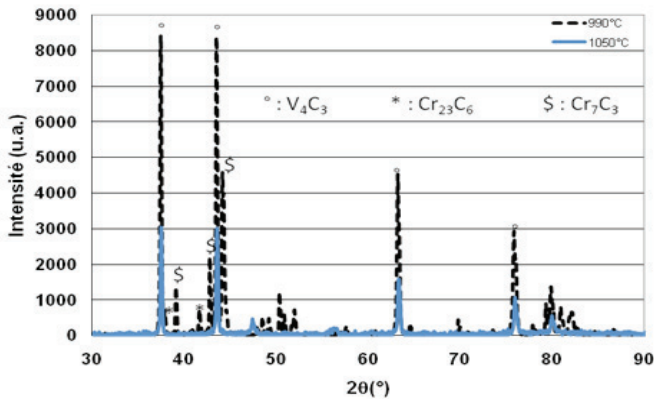


Fig. 4. The different primary carbides identified in the AISI H11 steel austenitized at 990°C and 1050°C.

As a matter of fact, the main metallurgical changes due to the modification of the austenitization temperature, i.e. the grain size and the morphology and type of primary carbides, does not impact the fracture toughness and crack propagation. Indeed, toughness tests give very close values for specimens austenitized at 990°C ( $84 \text{ MPa}\cdot\text{m}^{1/2}$ ) and for specimens austenitized at 1050°C ( $82 \text{ MPa}\cdot\text{m}^{1/2}$ ). Very similar results are also obtained for fatigue crack propagation rates which are particularly close for low  $\Delta K$  values, e. g. 20, 30 and 40  $\text{MPa}\cdot\text{m}^{1/2}$  (Fig.5).

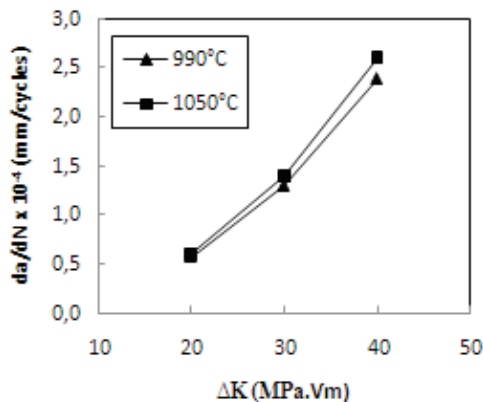


Fig. 5. Crack propagation rates for low  $\Delta K$  values, 20, 30 and 40  $\text{MPa}\cdot\text{m}^{1/2}$  in the AISI H11 steel austenitized at 990°C and 1050°C

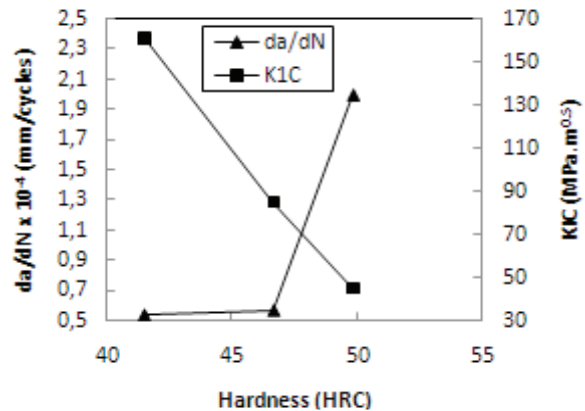


Fig. 6. Crack propagation rates for considered at  $\Delta K=20 \text{ MPa}\cdot\text{m}^{1/2}$  and toughness according to the hardness 42, 47 and 50 HRC

The modification of the structure at the micrometer scale (grains and martensitic laths) has no effect on the fracture toughness and the crack propagation rate. For the investigation of the second tempering influence (see section 3.3), only the specimens austenitized at 990°C, with the 3 different hardness conditions (42, 47 and 50HRC) are considered.

### 3.3. Influence of tempering conditions

The influence of second tempering on the crack propagation and fracture toughness is shown in Fig.6 where both  $K_{Ic}$  and  $da/dN$  are plotted against the hardness, mainly driven by the temperature of the heat treatment.

Results show that the toughness decreases and the crack propagation rate increases as the hardness increases (tempering temperature decreases). This confirms the expected embrittlement of the material as it hardens. The

variation of toughness and hardness is essentially linear whereas the crack propagation rate remains first almost constant between 42 and 47 HRC and dramatically jumps for 50 HRC.

Regardless of their hardness, the grain and martensitic lath characteristics are very similar for the three materials. For this reason, the investigation scale has been modified in order to thoroughly study the influence of hardness on the fracture toughness and crack propagation at the nanoscale.

SANS analysis were carried out at the Léon Brillouin Laboratory (CEA- CNRS) on specimens with different hardness (42, 47 and 50 HRC) to estimate the volume fraction, the size and the distribution of the nanometric secondary carbides. The experimental conditions are presented in [11, 12]. Results obtained are detailed in table 3 in terms of volume fraction  $f_v$ (%), equivalent radius  $r_p$  (nm) and inter-particle distance  $d$  (nm) of the secondary carbides promoted by the second tempering. Note that  $r_p$  is not hardness (tempering) dependent whereas  $f_v$  and  $d$  respectively increases and correlatively decreases as the material is hardening by appropriate tempering.

Table 3. Size, volume fraction and inter-particle distance for secondary carbides in the AISI H11 steel with hardness 42, 47 and 50 HRC (SANS results)

	Hardness 42HRC	Hardness 47 HRC	Hardness 50 HRC
$r_p$ (nm)	1.87	1.64	1.7
$f_v$ (%)	0.36	0.59	1.01
$d$ (nm)	53	39	29

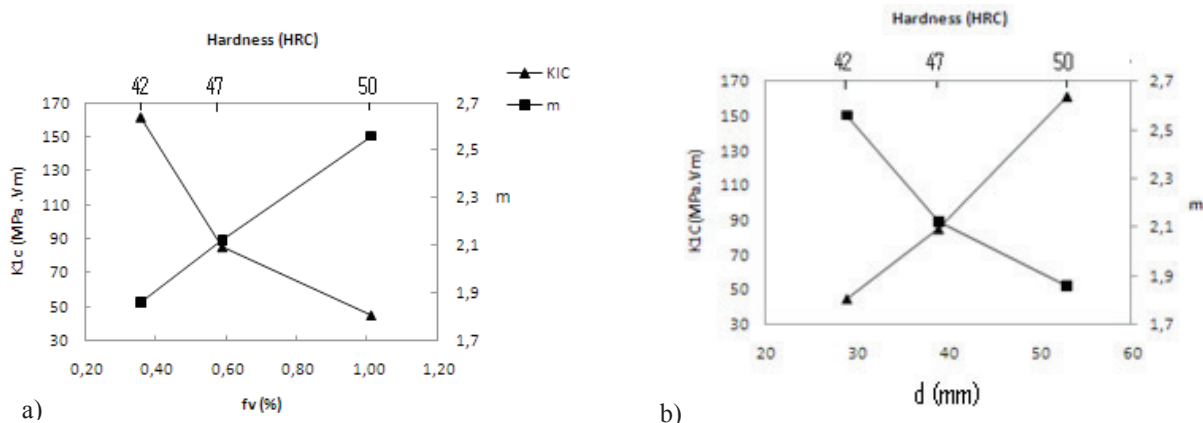


Fig. 7. Influence of (a) the volume fraction and (b) distance between nanometric carbides on the fatigue crack propagation and fracture toughness for hardness 42, 47 and 50 HRC.

Figure 7.a shows the influence of the volume fraction of secondary carbides (bottom scale) and the associated hardness (top scale) on the fracture toughness (left scale) and fatigue crack propagation (right scale). The toughness and the slope  $m$  of the Paris plot related to the variation of crack propagation rate show antagonist variations with an increasing volume fraction of hardening particles. As a result of an enhanced population of hard particles, toughness drops significantly following a quasi-linear decrease. Paris law exponent  $m$  strongly increases with the volume fraction of hardening particles. This indicates that the acceleration of the crack propagation with an increase in stress intensity factor is marked all the more as the material is hard. Figure 7.b confirms this general trend. The coalescence of secondary carbides as a function of the second tempering results in an increase of the distance inter-precipitates. For the lowest tempering temperature, the large quantity of non coalesced nanometric carbides reduces considerably the inter-particle distance and as a consequence increases the crack propagation rate. This result can be understood by transposing at the nanometric scale the mechanism proposed by Tomita [13] to explain the effect of microstructure on the fracture of martensitic steels. The mechanism, based on the interaction of cracking with large primary carbides can be divided in to three stages: i) damage initiation through the creation of voids around primary carbides due to plastic deformation, ii) increase in the size of voids, iii) coalescence and interconnection of the voids. The increase of the volume fraction of the nanometric precipitates has a detrimental effect on the fracture

toughness. As a consequence, the nanometric scale seems to be a relevant scale to be considered especially at the crack tip. However, complementary analyses are in progress to further investigate the precise nanostructural mechanism involved in fracture properties.

#### 4. Conclusion

Fracture toughness and fatigue crack propagation tests were carried out on various heat treatments of martensitic steels devoted to hot working operations. The analysis of the experimental results leads to the following main conclusions:

- the crack propagation rate increases and the toughness decreases as the hardness increases,
- an increase of the austenitizing temperature from 990°C to 1050°C has no influence on both the crack propagation rates and the toughness,

Conversely, modifications of tempering, which directly impact the alloy hardness, result in drastic changes of toughness and crack propagation rates. As no change in grain or martensitic lath morphology is observed, the associated modification of the Paris law coefficients is attributed to a change in the distribution and volume fraction of nanometric carbides investigated by Small Angle Neutron Scattering.

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