THE INFLUENCE OF MICROSTRUTURE ON FRACTURE TOUGHNESS OF VACUUM HEAT TREATED HSS AISI M2

VPLIV MIKROSTRUKTURE NA LOMNO ŽILAVOST VAKUUMSKO TOPLITNO OBDELANEGA HITROREZNEGA JEKLA M2

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The microstructure of AISI M2 high-speed steel can be substantially modified by vacuum heat treatment in order to optimize the ratio between hardness and fracture toughness, which is, however, significantly affected by the volume fractions of retained austenite and undissolved eutectic carbides, as well as the mean distance between these carbides. Calculated fracture toughness values, which were obtained using a newly developed semi-empirical equation, based on the stress-modified critical strain criterion and the quantified microstructural parameters, agreed well with the experimental results.

Key words: vacuum heat treatment, high speed steel, quantified microstructural parameters, hardness and fracture toughness

1 INTRODUCTION

The vacuum heat treatment of high speed steels for cold working applications must satisfy ever greater demands regarding their exploitation, particularly in respect of greater toughness while maintaining or even increasing hardness, and in respect of the smallest possible dimensional changes of such tools. A high fracture toughness \( K_{IC} \) means that these tools will be more resistant to shock loadings as well as to the propagation of fatigue cracks.

The microstructure of hot worked high speed steel, which has been vacuum quenched and tempered after annealing, consists of relatively large eutectic carbides in a martensitic matrix, hardened with finer secondary carbides. In the matrix, in which the eutectic carbides are distributed more or less in stringers, there is also some retained austenite. The fracture toughness of such steel is determined by the stress concentrators in the microstructure (e.g. carbides in stringers, carbide clusters, individual larger carbides, and non-metallic inclusions). When tools are subjected to loads, local stress concentrations occur next to the above-mentioned microstructure features which, if these stresses cannot be released through microyielding of the matrix, accelerate tool breakage. By means of heat treatment, the microstructure of high speed steel can be changed, and, within fairly wide boundaries, the properties of the matrix, too. The microstructure of high speed steel can be changed, and, within fairly wide boundaries, the properties of the matrix, too. By means of heat treatment, the microstructure of high speed steel can be changed, and, within fairly wide boundaries, the properties of the matrix, too. The microstructure of high speed steel can be changed, and, within fairly wide boundaries, the properties of the matrix, too. By means of heat treatment, the microstructure of high speed steel can be changed, and, within fairly wide boundaries, the properties of the matrix, too. By means of heat treatment, the microstructure of high speed steels having the same hardness but different microstructures and consequently different fracture toughnesses can be obtained, so that the optimisation of the heat treatment of high speed steels is a worthwhile task.

2 THEORY

The Rockwell C hardness as determined by a normal indentation test is primarily a feature of the matrix of high speed steel, provided that the indentation is not made at a position where carbide size or quantity is excessive. In the as-quenched condition, hardness may give some indication of the temperature from which the specimen has been quenched. In the tempered condition, hardness is essential from the user’s viewpoint, although this value alone is not capable of differentiating between specimens hardened and tempered by different routes –
for example, similar hardness may be obtained by varying quenching and tempering temperatures, or merely by taking a tempering temperature either side of the peak secondary hardness value. For that reason beside of hardness an additional mechanical property such as fracture toughness \( K_c \) could be used for differentiation concerning the influence of vacuum heat treatment. Namely, fracture toughness tests on high speed steels show a better differentiation concerning the influence of heat treatment by the fracture toughness \( K_c \) than by the data of bend test 1.

An overview of the literature has shown that several different methods are used for measuring the fracture toughness of high speed steels. These include both standard methods, with the use of CT and SENB test specimens 2, and non-standard methods 3-5. Furthermore recently was, by the authors 6-7, developed semi-empirical equation where the fracture toughness of the high speed steel was quantified on the basis of microstructural parameters and several other material properties:

\[
K_c = 1.363 \left( \frac{HRc}{HRc - 53} \right) \left[ E \cdot d_p \cdot (f_{carb})^{0.2} \cdot (1 + f_{aust}) \right] \quad (1)
\]

Since the above correlation is a semi-empirical one, and has been derived by taking into account, on the one hand, the critical strain criterion (McClnctock 8, Mackenzie et al 9, Bates 10, and Ritchie et al 11) and, on the other hand, the experimentally determined effects of the microstructural parameters and Rockwell C hardness, it is necessary to take all due care with units. The constant 1.363 was obtained by assuming that the modulus of elasticity \( E \) is expressed in MPa, the mean distance between undissolved eutectic carbides \( d_p \) in m, the Rockwell C hardness in units of HRC, and \( f_{carb} \) and \( f_{aust} \) as volume fractions of undissolved eutectic carbides and retained austenite. In this case the fracture toughness \( K_c \) is obtained in units of MPa\( \cdot \)m\( ^{-0.5} \).

![Image](image.png)

From equation (1) it follows that the fracture toughness \( K_c \) of high speed steel depends not only on the latter’s mechanical properties (apart from the modulus of elasticity, the fracture ductility and yield stress - both defined in terms of Rockwell C hardness, which means that the fracture ductility is inversely proportional to the hardness of the matrix and that the yield stress of the matrix is approximately proportional to hardness \( ^{7,8} \)) but also on several microstructural parameters (the volume fraction of undissolved eutectic carbides \( f_{carb} \), the volume fraction of retained austenite \( f_{aust} \) - which affects the strain hardening exponent \( n \) - and the mean distance between the undissolved eutectic carbides, \( d_p \), all of which depend on the vacuum heat treatment conditions. In the case of AISI M2 high speed steel, which contains a significant fraction of undissolved eutectic carbides, the mean distance between these carbides \( d_p \) can be calculated 12 from the following equation:

\[
d_p = D_p \cdot (1 - f_{carb}) \cdot \frac{2}{\sqrt{3} \cdot f_{carb}} \quad (2)
\]

where \( f_{carb} \) is the volume fraction of undissolved eutectic carbides, and \( D_p \) is their mean diameter. However, importantly, the calculated fracture toughness values, which were derived using a newly developed semi-empirical equation, agreed well with the experimental results obtained by the authors, as well as with results obtained by other authors 13.

### 3 EXPERIMENTAL PART

#### 3.1 Choice of material and vacuum heat treatment.

In the experimental work, the ESR high speed steel AISI M2, delivered in the shape of rolled, soft annealed bars \( \Phi \) 20 mm x 4000 mm, was used. This steel had the following chemical composition (mass content in %): 0.89 % C, 0.20 % Si, 0.26 % Mn, 0.027 % P, 0.001 % S, 3.91 % Cr, 4.74 % Mo, 1.74 % V, and 6.10 % W. The \( K_c \) test specimens, i.e. circumferentially notched and fatigue pre-cracked tensile test specimens made from these bars, were heat treated in a horizontal VTTC-324R vacuum furnace, with uniform high-pressure gas quenching, using \( N_2 \) at a pressure of up to 5 bars abs. An overview of the quenching and tempering temperatures, which were used in the experimental work described in this paper, is presented in Table 1.

<table>
<thead>
<tr>
<th>Group of ( K_c ) test specimens</th>
<th>Vacuum heat treatment conditions</th>
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<tbody>
<tr>
<td>A</td>
<td>1050/80/2x500 °C</td>
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<tr>
<td>B</td>
<td>1050/80/2x540 °C</td>
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<tr>
<td>C</td>
<td>1100/80/2x500 °C</td>
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<td>D</td>
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<td>H</td>
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<tr>
<td>I</td>
<td>1230/80/2x540 °C</td>
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<tr>
<td>J</td>
<td>1230/80/2x550 °C</td>
</tr>
<tr>
<td>K</td>
<td>1230/80/2x570 °C</td>
</tr>
<tr>
<td>L</td>
<td>1230/80/2x600 °C</td>
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For each set of vacuum heat-treatment conditions from A to G and I, at least 20 \( K_c \) test specimens were tested, the Rockwell C hardness and fracture toughness values being determined as described below. For each group of \( K_c \) test specimens from H and J to L, two \( K_c \) test specimens were tested.
3.2 Hardness and fracture toughness tests

The measurements of Rockwell C hardness were performed on the individual groups of the $K_{IC}$ test specimens using a Wilson 4JR hardness machine. In the case of the experimental work performed by the authors, circumferentially notched and fatigue pre-cracked tensile test specimens were machined, with the dimensions indicated in Figure 1.

The advantage of such test specimens over CT (compact tension) specimens lies in the former’s radial symmetry, on account of which they are particularly suitable for studying the influence of the microstructure of metallic materials on their fracture toughness. This is because, due to the radial symmetry of the heat transfer, the formation of the microstructure is completely uniform.

Due to the high notch sensitivity of hard and brittle metallic materials, such as the high speed steel AISI M2, it is very difficult, and sometimes almost impossible, to create a fatigue crack in the corresponding test specimens. However, a fatigue crack can be created on such test specimens under a rotating-bending regime, if this is done before final heat treatment is performed. The advantage of such test specimens is that plane strain conditions can be achieved while using specimens having smaller dimensions than is possible in the case of conventional CT test specimens. The critical stress intensity factor $K_{IC}$ was calculated from the equation developed by Parish and Sih as modified by Bueckner for a circumferentially notched round bar:

$$K_{IC} = \frac{P}{D^{3/2}} \left( -1.27 + 1.72 \frac{D}{d} \right)$$

where $P$ is the load at failure, $D$ is the outside diameter, and $d$ is the notched section diameter of the test specimen, i.e. the diameter of the ligament next to the crack. The relationship (3) holds true as long as the condition $0.5 < d/D < 0.8$ is fulfilled.

3.3 Microstructural and microfractographic tests

The microstructural tests were performed on the individual groups of $K_{IC}$ test specimens using, firstly, conventional optical metallographic techniques and a NIKON Microphoto-FXA optical microscope, and, secondly, a JEOL JSM-35 scanning electron microscope, which was also used for the micrographic tests of the fracture surfaces of the $K_{IC}$ test specimens. The microstructures of the test specimens of the investigated, vacuum heat treated AISI M2 high speed steel were quantitatively evaluated, using the following parameters: the mean diameter of the undissolved eutectic carbides, and the volume fractions of the individual microstructural phases (the undissolved eutectic carbides, the tempered martensite, and the retained austenite). The mean diameter $D$ and volume fraction of the undissolved eutectic carbides $f_{carb}$ were determined on unetched metallographic specimens. SEM images of the microstructures were obtained with reflected electrons (BE)17,18, at a magnification of M 1000 x. The images of 11 to 16 visible fields, obtained on each of the metallographic specimens of the investigated high speed steel, which had been vacuum quenched and tempered, were analysed using KS Lite V2.00 software for image analysis. From the images of the microstructure, obtained using the optical microscope at magnifications of M 600 x, of the same metallographic specimens, which had been etched for 2 to 3.5 minutes in a 5 % solution of nital with 10 % added HCl, and by means of image analysis using the KS Lite V2.00 software, the total volume fraction $f_{carb}$ of the undissolved eutectic carbides and of the retained austenite was determined. Eleven to twelve visible fields were analysed on each of the metallographic specimens of the investigated high speed steel. From the differences between the so determined total volume fraction of the undissolved eutectic carbides and the retained austenite (which appears white in the images obtained using the optical microscope) and the volume fraction of the undissolved eutectic carbides (SEM with reflected electrons), the volume fraction of the retained austenite in the investigated high speed steel was determined.

4. RESULTS AND DISCUSSION

It is well-known that the hardness of high speed steels varies according to composition, austenitizing temperature and time, and tempering temperature, and number of tempering operations. Different heat treatment processes (i.e. salt bath, fluidised bed or vacuum heat treatment) as well as microstructure has also an effect. Figure 2 shows the effect of tempering temperature on secondary hardness peak of the
investigated vacuum heat treated high speed steel after double tempering.

The key problem in measuring fracture toughness of investigated high speed steel using circumferentially notched and fatigue pre-cracked tensile test specimens is linked with the disturbing effect of larger carbides or carbide clusters, which represent the weak spots on or near the fracture surface.

Analysis of the results of measurements of fracture toughness showed that the relationship between the calculated value of $K_{IC}$ using equation (3) and the radial distance $x$ of the weak spot from the tip of the fatigue crack (see Figure 3) was a linear one (except for group G), so that the correct value of fracture toughness can be obtained simply, by linear extrapolation to $x = 0.5$.7.

Observation in the SEM of the weak spots on the fracture surfaces of the $K_{IC}$ test specimens, at high magnification, provided confirmation that such weak spots are characterised by the coalescence of microvoids (ductile regions) connected together by carbides or carbide clusters, as well as by characteristic tearing areas, where fracture in plastic shear had been initiated (see Figure 4).

Figure 2: Influence of tempering temperature on secondary hardness peak of the investigated high speed steel. Vacuum austenitized 2 mins at 1230 °C and double tempered for 1 h. (measured on $K_{IC}$ test specimens from G to L; Table 1)

Slika 2: Vpliv temperature popuščanja na vrh sekundarnega utrjevanja pri preiskovanem hitroreznem jeklu. Vakuumsko avstenitiziran 2 min na 1230 °C in dvakratno popuščen po 1 h. (Merjeno na $K_{IC}$ preskus-šancih G do L, Tabela 1)

Figure 3: SEM image of a fracture surface containing a weak spot ($K_{IC}$ test specimen from group G)

Slika 3: SEM posnetek lomne površine s šibkim mestom ($K_{IC}$ preiskušanec skupina G)

Figure 4: SEM image of an area of coalescence of microvoids at a weak spot of the $K_{IC}$ test specimen shown in Figure 3. Areas of transgranular fracture can also be seen ($K_{IC}$ test specimen from group G)

Slika 4: SEM posnetek področja s koalesenco mikropor na šibkem mestu $K_{IC}$ preiskušanja s slike 3. Vidno je tudi področje transgranalnega preloma ($K_{IC}$ preiskušenec skupina G)

Figure 5: The influence of the temperature of austenitization on the hardness HRc and fracture toughness $K_{IC}$ of the investigated high speed steel, for two selected temperatures of tempering, for $\varphi_p = 500$ °C and $\varphi_p = 540$ °C (measured on $K_{IC}$ test specimens from A to G and I; Table 1)

Slika 5: Vpliv temperature avstenitizacije na trdoto in lomno `ilavost preiskovanega hitroreznega jekla, po popuščanju 2 x 1h na temperaturi $\varphi_p = 500$ °C oziroma $\varphi_p = 540$ °C (Merjeno na $K_{IC}$ preiskušancih A do G in I; Tabela 1)
The results of the measurements of hardness and fracture toughness are shown, for the individual groups of $K_{IC}$ test specimens from A to G and I, in Figure 5.

From the diagram in Figure 5 it can be seen that the highest fracture toughness $K_{IC}$ of 17.7±1.4 MPa√m and belonging hardness of 60.4±0.5 HRc is achieved after vacuum quenching from austenitizing temperature of 1230 °C and double tempering for 1 hour at a temperature of 500 °C. In examining the course of tempering, it is clear that fracture toughness $K_{IC}$ is a very selective mechanical property with regard to the temperature of austenitization and of tempering.

The microstructures of the investigated high speed steel (set of vacuum heat-treatment conditions G, I, K and L, from Table 1) examined by scanning electron microscope are shown in Figure 6.

As can be seen from micrographs in Figure 6, the microstructure of the investigated high speed steel consisting of tempered martensite and undissolved eutectic carbides. There is also retained austenite in the matrix, tough less after double tempering at 570 °C (K), and more after double tempering at 500 °C (G). After double tempering at 600 °C (L) retained austenite in the matrix is no more visible. According to above micrographs it can be concluded that after vacuum quenching from 1230 °C and double tempering up to 570 °C retained austenite is very stable. In the case of the microstructure of the investigated high speed steel, which was quenched in the vacuum furnace from the temperature of 1230 °C, and double tempered for 1 hour at 500, 540, 570 and 600 °C, respectively, the volume fraction of the retained austenite $f_{av}$ being determined as described above. The results of the determination of the volume fraction of the retained austenite are shown in Figure 7.

From the diagram in Figure 7 it can be seen that in the case of the microstructure of the investigated high speed steel, which was vacuum quenched from 1230 °C, and double tempered at 500 °C, a relatively large volume fraction of stabilized retained austenite was obtained, amounting to approximately 25 to 30 vol. %, giving this steel its relatively high fracture toughness (see Figure 5). In this case a matrix hardness of 60.4 HRc still ensure a sufficiently high compressive yield stress $\sigma_{YS} = 1421$ MPa.

Quantitative measurements of the mean diameter $D_p$ and volume fraction of the undissolved eutectic carbides $f_{carb}$ were determined on unetched metallographic specimens. Statistical analysis of the experimental results has shown that, in the case of the investigated high speed steel, the mean diameter $D_p$ and volume fraction of the undissolved eutectic carbides mainly depend on austenitizing temperature and is practically independent of number of tempering cycles and tempering temperature. In this case - set of vacuum heat-treatment conditions G to L, from Table 1- the mean diameter of the undissolved eutectic carbides amount to $D_p = 0.94±0.1$ µm, and volume fraction of these carbides amount to $f_{carb} = 6.7±1.4$ %. The mean distance between these carbides $d_p$ was calculated by mean of equation (2), and amount to $d_p = 2.8±0.6$ µm.

From the results presented in ref. it can be seen that, for the investigated high speed steel, within the hardness range between 57 and 66 HRc, the measured and calculated values of fracture toughness $K_{IC}$ agree very well, the disagreement being less than 10 %, and...
In examining the course of tempering, it is found that there is a peak value of fracture toughness for a low tempering temperature ($\theta_a = 500 ^\circ C$) and a minimum corresponding to the hardness peak. This provides a strong indication that among others possible effects also differences in stabilised retained austenite (see Figure 7) cause the fracture toughness variations. Namely, the net effect of tempering is attributed to a combination of stress relief and a reduction in ductility due to the secondary hardening peak. From the diagram in Figure 8 it can be clearly seen that in the case of the same obtained hardness the under-tempered $K_t$ test specimens vacuum quenched from the same austenitizing temperature, achieve higher fracture toughness. For example, after vacuum quenching from 1230$^\circ C$ and double tempering for 1 hour at a temperature of 600 $^\circ C$ investigated high speed steel achieve hardness of 63.7 HRC and fracture toughness of $K_t = 9.9$ MPa\(\sqrt{m}\), the same hardness but for ca. 30 % higher fracture toughness i.e. $K_t = 12.8$ MPa\(\sqrt{m}\) could be obtained after double tempering for 1 hour at a temperature of 520 $^\circ C$ (see Figure 8). This could lead to the conclusion that high volume fraction of stabilized retained austenite in under-tempered investigated high speed steel (see Figure 7); significantly increase its fracture toughness.

Furthermore, good understanding of mutual interaction of mechanical and microstructural properties on fracture toughness $K_t$ of investigated high speed steel as shown with the developed semi-empirical equation (1) gave us an opportunity of optimal choice of composition and manufacture of high speed steel (HIP’ed, forged, sintered, ESR or conventional material) as well as the choice of optimal heat treatment process (salt bath, fluidised bed or vacuum heat treatment) in the possibility of obtaining an optimum combination of basic characteristic of tools of high speed steels, and of the given working part – tool combination.

5 CONCLUSIONS

On the basis of the results of extensive tests performed on the ESR high-speed steel AISI M2, it has been confirmed that the microstructure of the investigated steel can be substantially modified by vacuum heat treatment in order to optimize the ratio between the hardness and fracture toughness $K_t$ of this steel. It has also been experimentally proved that the volume fraction of retained austenite, the volume fraction of undissolved eutectic carbides, and the mean distance between the undissolved eutectic carbides all...
have a significant effect on the measured fracture toughness $K_{IC}$ of this steel.

The semi-empirical correlation equation (1) derived by the authors for calculating the fracture toughness of tool steels such as the investigated high speed steel demonstrate that beside the increased amount of retained austenite stable after tempering (steel initially vacuum austenitized at highest temperature), fracture toughness is significantly influenced also by the mean distance of undissolved eutectic carbides, and thus, at given composition, by the carbide size.

After vacuum quenching of the investigated high speed steel from the highest recommended austenitizing temperature the volume fraction of undissolved eutectic carbides amounting to 6.7 %, their mean diameter to 0.94 µm and the mean distance between them to 2.8 µm. When a crack propagates in a material with such large undissolved eutectic carbides separated by large mean distance, large ligaments are left between the voids, which form at the individual carbides or carbide clusters. The plastic deformation of these ligaments is responsible for the energy dissipation that determines the crack resistance of the material. Therefore large undissolved eutectic carbides, with correspondingly large mean distance, give higher fracture toughness than smaller carbides.

It was also experimentally confirmed that in such a way vacuum heat treated investigated high speed steel have superior fracture toughness, especially at hardness below peak. This allows greater freedom in selecting desired combinations of hardness and toughness, especially in applications not requiring peak hardness.

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