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THE APPLICATION OF LINEAR ELASTIC FRACTURE MECHANICS TO OPTIMIZE THE VACUUM HEAT TREATMENT AND NITRIDING OF HOT-WORK TOOL STEELS

UPORABA LINEARNE ELASTOMEHANIKE LOMA PRI OPTIMIRANJU VAKUUMSKE TOPLOTNE OBDELAVE IN NITRIRANJU ORODNIH JEKEL ZA DELO V VROČEM

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Linear elastic fracture mechanics was used to optimise the vacuum-heat-treatment procedure for conventional hot-work AISI H11 tool steel. The fracture toughness was determined with non-standard, circumferentially notched and fatigue-precracked tensile-test specimens. The fracture-testing method is sensitive to changes caused by variations in the microstructure due to the austenitizing and tempering temperature as well as the homogeneity of the material itself. The combined tempering diagram – Rockwell-C hardness, fracture toughness, K_{Ic}, tempering temperature – was used to help choose the vacuum-heat-treatment parameters, aimed at obtaining the best properties for a given application with respect to the investigated steel. Nitriding treatments are established methods for improving the wear performance of tools and dies from H11 hot-work tool steel. However, the understanding of the relationship between the nitriding process parameters, on the one hand, and the microstructure and fracture behaviour of the surface layers, on the other, is far from complete. Vickers hardness indentations can generate radial fractures in brittle surface layers, and it has been shown that these cracks can be used to provide reliable information about the fracture toughness of these layers. The results suggest that for a sufficiently thick compound layer, this method has the potential to be applied as a pseudo non-destructive method of monitoring the fracture properties of treated surfaces on actual tool parts. The use of Vickers indentations for fracture toughness would give a¸useful insight into the fracture properties of nitride layers and their likely response to the application conditions involving high shear or impact loading. This could be very useful for investigations and quality control tool for industrial surface engineering applications.

Key words: hot-work tool steel, vacuum heat treatment, fracture toughness, hardness, microstructure, nitriding, indentation, cracks

Pri optimiranju vakuumske toplotne obdelave konvencionalno izdelanega orodnega jekla za delo v vročem AISI H 11 smo uporabili princip linerne elastomehanike loma. Lomno žilavost smo določili z nestandardnimi cilindričnimi nateznimi preizkušanci z zarezo po obodu in utrujenostno razpoko v konici zareze. Metoda je občutljiva za spremembe v mikrostrukturi, ki so povezane s temperaturo avstenitizacije in popuščanja, kot tudi na homogenost samega jekla. Za izbiro parametrov vakuumske toplotne obdelave smo uporabili kombiniran diagram popuščanja – trdota Rockwell-C, lomna žilavost K_{Ic}, temperatura popuščanja – z namenom, da bi dobili najprimernejše lastnosti preiskovanega jekla za specifično uporabo. Z modificiranjem delovnih površin povečamo orodju iz jekla za delo v vročem AISI H11 obrabno odpornost. Nitriranje v pulzirajoči plazmi je uveljavljen postopek s katerim delovnim površinam takemu orodju povečamo odpornost proti obrabi. Vendar pa odnosi med parametri nitriranja in mikrostrukturo ter lomnimi lastnostmi modificiranih plasti še niso dovolj raziskani. Posledica vtiskovanja Vickersove piramide v trde krhke površinske plasti so radialne razpoke, na osnovi katerih lahko pridobimo zanesljive informacije o lomni žilavosti teh plasti. Iz rezultatov je razvidno, da lahko uporabljeno neporušno metodo uporabimo za ugotavljanje lomnih lastnosti modificiranih površin orodij, če je spojinska plast dovolj debela. Uporaba vtiskovanja Vickersove piramide za določevanje lomne žilavosti omogoča boljše razumevanje lomnih lastnosti nitridnih plasti in njihov verjeten odziv na specifične pogoje uporabe pri velikih strižnih in/ali udarnih obremenitvah. To bi lahko bila tudi zelo uporabna raziskovalna in kontrolna metoda, ki bi jo uporabljali pri aplikacijah inženirstva površin v industrji.

Ključne besede: jeklo za delo v vročem, vakuumska toplotna obdelava, lomna žilavost, trdota, mikrostruktura, nitriranje, vtiskovanje, razpoke

1 INTRODUCTION

The process parameters, the work material and the tool material determine the dominant damage mechanism. For this reason, improving the tool's performance requires a detailed knowledge of the relevant damage mechanism. It is also clear that the tool material itself plays a very important role, and that the properties' profile of the tool material greatly influences its lifetime. The variety of tooling operations is enormous; however, there are some basic properties of tool materials that are very significant for almost all applications. These properties are toughness, which prevents the instantaneous fracture of the tool or tool edge due to local overloading, and hardness, which must be sufficiently high to avoid local plastic deformation. Hardness and toughness are in general mutually exclusive properties, which means the prevention of instantaneous tool failures is often connected with a critical hardness level that must not be exceeded for a specific application. The hardness and the toughness of hot-work tool steel depend a lot on the vacuum-heat-treatment procedure. Hardness is closely related to the ductility and the toughness, in particular to the latter. In this paper the influence of the austenitizing and tempering temperatures on the hardness and fracture toughness of conventional hot-work AISI H11 tool steel is investigated and discussed.

2 THEORY

According to ref. [1], toughness and ductility are the most relevant properties in terms of resistance to total failure as a result of overloading. Toughness and ductility are two different material properties, even though both – unfortunately – are sometime denominated as toughness. The opposite of both properties is, however, the same, i.e., brittleness. No standardised tests for the determination of toughness or ductility are in general use. Often, data determined with different test methods are available, which makes them difficult to compare and can lead to misunderstandings. Toughness and ductility are different characteristics, and for this reason it is necessary to distinguish between them [1]. Their importance for tool-steel performance depends a lot on the tool geometry [1]. In the case of un-notched specimens or specimens with smooth notches, the ductility and fracture stress are the relevant material properties. However, if sharp notches or cracks are present, fracture toughness is the most relevant property. The conclusion is that the tool steel should be optimised in terms of ductility and fracture stress for un-notched regions, and in terms of fracture toughness for notched regions.

The toughness depends a lot on the hardness, and the hardening mechanism is different in as-quenched and fully-heat-treated tool steels. In the as-quenched tool, work-hardening and solid-solution hardening, mostly due to carbon in solid solution, affect mainly the steel's hardness. Tempering leads to the precipitation of carbide particles and to a significant decrease of the carbon content in solid solution, and to an increase of the dislocation density. The hardness of fully-heat-treated tool steels is therefore mainly the result of precipitation hardening and, to a small extent, of solid-solution hardening. The work-hardening and grain refinement seem to play only a minor role [2].

The most reliable measure of toughness is the plain-strain fracture toughness. The minimal size of specimens used for the testing depends on the yield stress and the fracture toughness of the tested material, both essential for the plane-strain deformation. A fatigue crack of a defined length is propagated from a mechanical notch in the specimens ensuring that the notch effect is a maximum and equal for all tests. The same value of fracture toughness should be found for tests on specimens of the same material with different geometries and with a critical combination of crack size and shape, and fracture stress. Within certain limits, this is indeed the case, and information about the fracture toughness obtained under standard conditions can be used to predict the failure for different combinations of stress and crack size, and for different geometries [3].

On the other hand, the compound layers formed with nitriding surface treatments have a substantially lower fracture toughness than the underlying substrate, and this can adversely affect the wear performance of components subjected to severe service environments involving high shear, compressive and/or impact loading, and therefore characterisation of the relationship between the nitriding process parameters, the microstructure and the fracture behaviour of the nitride layers is crucial to ensure that these surface treatments can be used commercially with confidence. The use of indentation fracture testing has a number of advantages: it relies on relatively inexpensive and unsophisticated test equipment, it can be used on a wide range of sample sizes and it requires minimal sample preparation. However, it should be noted that although the method is well known for the analysis of relatively uniform bulk ceramic materials [4], many of the assumptions made in developing the equations that relate fracture toughness to the observed cracking behaviour may not be entirely valid for materials where a thin brittle layer is supported by a relatively tough substrate material with depth-varying properties. The theory and application of the method for fracture toughness testing of thin, hard coatings has recently been extended [4].

3 EXPERIMENTAL

3.1 Material, vacuum heat treatment and pulse plasma nitriding and nitrocarburisig

Conventional hot-work AISI H11 tool steel delivered in the shape of plates with dimensions 263 mm × 220 mm × 25 mm, cut from forged-and-soft-annealed master blocks with dimensions 263 mm × 220 mm × 4000 mm and the following chemical composition (mass fractions in %): 0.39 % C; 1.06 % Si; 0.32 % Mn; 0.019 % P; 0.004 % S; 4.91 % Cr; 0.11 % Ni; 1.17 % Mo; 0.37 % V; and 0.011 % Ti was used. The K_{Ic}-test specimens, circumferentially notched and fatigue-precracked tensile-test specimens, were cut from these plates in the short transverse direction. A round notch with a fatigue crack at the notch root was at the same distance (60 mm) from the surface of the master block in all the K_{Ic}-test specimens. The specimens were heat treated in a horizontal vacuum furnace IPSEM VTTC-324R, with uniform high-pressure gas-quenching using nitrogen (N_2) at a pressure of 1.05 bar. After the last preheat (850 °C) the specimens were heated (10 °C/min) to the austenitizing temperatures 1000 °C, 1020 °C and 1050 °C, soaked for 20 min, and gas quenched to 100 °C. Then the first temper was performed at 540 °C, and the second at different temperatures between 540 °C and 620 °C, as shown in **Figure 2**, each time for 2 h. For each group of vacuum-heat-treatment conditions from A to C, five K_{Ic}-test specimens were tested for each second tempering temperature.

From the vacuum heat-treated test specimens, metallographic specimens of 1.5-cm thickness and 2.5-cm diameter were manufactured for a subsequent pulse plasma nitriding and nitrocarburising treatment. The specimens were then plasma nitrided at 540 °C (Series 1), or nitrocarburized at 580 °C (Series 2), using 3.3 hPa pressure for the nitriding and 4.3 hPa for the nitrocarburising, and a total gas flow rate of 75 L/h. The volume fraction of the gas atmosphere was 75 % H_2 : 25 % N_2 for nitriding and 87 % N_2 : 2 % CO_2 : 11 % H_2 for nitrocarburizing. The heating to the process temperature took approximately 3 h and the lengths of the treatments for each condition were (8, 16, or 32) h.

3.2 Hardness and fracture-toughness tests

The Rockwell-C hardness (*HRc*) was measured on the individual groups of K_{Ic}-test specimens using a Wilson 4JR hardness machine. Circumferentially notched and fatigue-precracked tensile-test specimens with the dimensions indicated in Figure 1 were used for this investigation [5].

The advantage of the test specimens used here over standardized CT specimens (ASTM E399-90) lies in the radial symmetry, which makes them particularly suited for studying the influence of the microstructure of metallic materials on fracture toughness. The advantage of these specimens is in the heat transfer, which is sufficient to provide a completely uniform microstructure over the specimen section. Due to the high notch sensitivity of hard and brittle metallic materials, such as the hot-work AISI H11 tool steel, it is very difficult – and sometimes almost impossible – to create a fatigue crack in the test specimen. However, with the used specimens the fatigue crack can be created with rotating-bending loading before the final heat treatment [5]; the second advantage of such test specimens is that plane-strain conditions can be achieved using specimens with smaller dimensions than those of conventional CT test specimens [6].

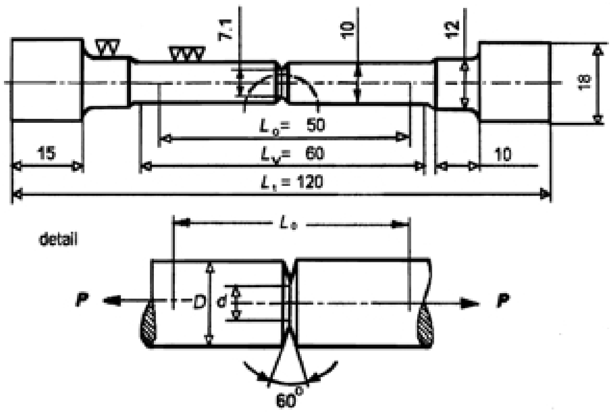


Figure 1: Circumferentially notched and fatigue-precracked K_{Ic}-test specimen. All dimensions are in mm

Slika 1: K_{Ic} preizkušanca z zarezo po obodu in utrujenostno razpoko v konici zareze. Vse dimenzije so v milimetrih.

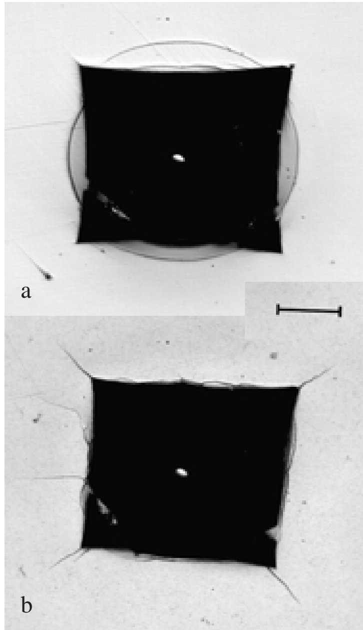


Figure 2: Optical micrographs showing typical (a) lateral (concentric ring) cracking in 8 h nitrided sample and (b) Palmqvist-type cracking in 16 h nitrocarburized sample. Both indentations made with a 50 kg load. The micron bar represents 75 µm.

Slika 2: Svetlobni posnetek prikazuje značilne (a) lateralne (koncentrični obroči) razpoke v vzorcu po 8 h nitriranja in (b) Palmqvistove razpoke v vzorcu po 16 h nitrocementiranja. Oba vtiska sta narejena z obremenitvijo 50 kg. Merilo predstavlja 75 µm.

Vickers hardness indentations generate radial fractures in brittle surface layers, and it has been shown that the length of these cracks can be used to provide valuable information about the fracture toughness of these layers [4]. There are two basic cracking modes possible from Vickers indentations in brittle materials, the radial-median and the Palmqvist cracking modes, **Figure 2**.

It has been shown that since the Palmqvist crack morphology is characterized by shallow cracks emanating from the corners of the Vickers indentation, it is appropriate to use the following relationship when characterizing the fracture toughness of nitride layers on tool steels [7];

$$K_{Ic} = 0.0319 \cdot \left(\frac{P}{a \cdot l^{1/2}} \right) \quad (1)$$

where P is the indentation load, a is the mean diagonal half length and l is the mean crack length. According to the Palmqvist theory, the fracture toughness K_{Ic} should be independent of the applied load. The most valid measure of K_{Ic} for the thin coating can therefore be obtained by extrapolating the K_{Ic} versus P data to $P = 0$, where the intrinsic fracture toughness of the coating, denoted by K_{Ic0}, can be deduced. Lightly polished treated surfaces were submitted to Vickers hardness testing in triplicate at loads of (5, 10, 15, 20, 30, 40, 50 and 60) kg. Indent and crack dimensions were measured using an optical microscope, and the measured values of the crack and indent half-diagonal lengths were used to calculate $K_{\rm Ic}$ according to equation (1). A mean value of $K_{\rm Ic0}$ is then derived by extrapolation to a zero load condition.

4 RESULTS AND DISCUSSION

The average measured hardness and fracture-toughness data are shown for the normal range of working hardness (*HRc* 40–55) in a so-called combined tempering diagram (Rockwell-C hardness, Fracture toughness $K_{\rm Ic}$, Tempering temperature) in **Figure 3**.

It is clear that the highest fracture toughness $K_{\rm Ic}$ and the pertained hardness are achieved after vacuum quenching from the austenitizing temperature of 1020 °C and double tempering across the whole range of the used tempering temperatures. Considering the effect of tempering temperature, it is clear that the fracture toughness $K_{\rm Ic}$ is a very selective mechanical property with regard to the austenitization and tempering temperatures.

The influence of the temperature of austenitization on the fracture toughness $K_{\rm Ic}$ of the investigated tool steel is shown in **Figure 4** for selected tempering temperatures.

As shown in **Figure 4**, the influence of austenitizing temperature on the fracture toughness $K_{\rm Ic}$ of the investigated tool steel is practically negligible after double tempering at, or slightly above, the peak of secondary hardening, i.e., at 540 °C and 560 °C. At a higher tempering temperatures, especially in the range from 580 °C to 600 °C, that are generally applied for most hot-work applications, the influence of the austenitizing temperature on the fracture toughness $K_{\rm Ic}$, is significant. It is evident that by determining the hardness of the steel, the heat-treatment procedure also has a strong influence on the fracture toughness.

Figure 5 shows that hardness has a very strong influence on the fracture toughness of the investigated steel.

The correlation between the hardness and the fracture toughness is reasonably good for all three austenitizing temperatures. At a particular hardness, i.e., the normally used working hardness between *HRc* 45 and 48, considerable differences in the fracture toughness of the investigated steel due to the different vacuum-heat-treatment procedures are found (**Figure 5**). For this reason a thorough knowledge of the influence of the heat-treatment parameters used (**Figure 3**) on the hardness and fracture toughness is important for

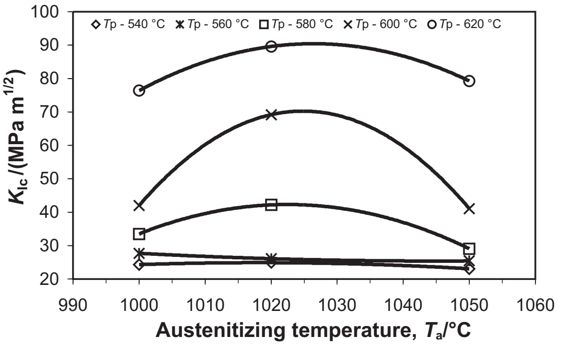
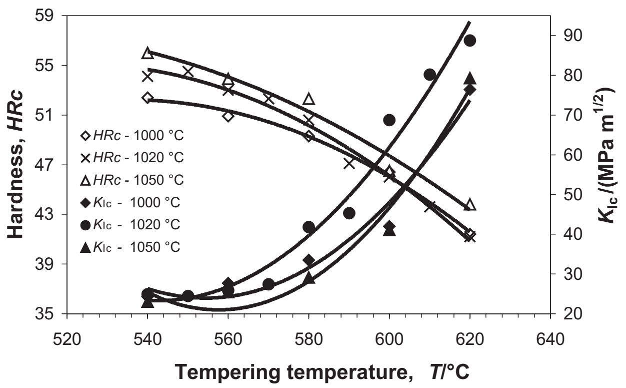


Figure 4: The influence of austenitization temperature on the fracture toughness $K_{\rm Ic}$ of the investigated tool steel for selected tempering temperatures. Vacuum-heat-treatment parameters are listed in **Figure 3**

Slika 4: Vpliv temperature avstenitizacije na lomno žilavost $K_{\rm Ic}$ preiskovanega jekla za izbrane temperature popuščanja. Parametri vakuumske toplotne obdelave so podani na **sliki 3**.



$K_{\rm Ic}$ test specimens: circumferentially notched and fatigue-precracked tensile specimens ϕ (10 × 120) mm
Austenitization temperature: 1000 °C, 1020 °C and 1050 °C
Soaking time: 20 min
Quenching: gas quenching in N_2 at a pressure of 1.05 bar at 100 °C
Cooling parameter $\lambda_{800\text{-}500}$: 1.04; 1.02; 1.11
First tempering: 1 × 2 h at 540 °C
Second tempering: 1 × 2 h between 540 °C and 620 °C

Figure 3: Effect of austenitizing and tempering temperatures on the hardness and fracture toughness $K_{\rm Ic}$ of the investigated hot-work H11 tool steel

Slika 3: Vpliv temperature avstenitizacije in popuščanja na trdoto in lomno žilavost $K_{\rm Ic}$ preiskovanega jekla za delo v vročem H 11

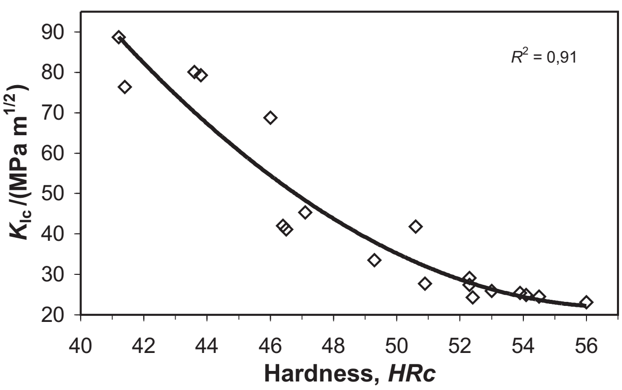


Figure 5: Relationship between the hardness and the fracture toughness of the investigated tool steel

Slika 5: Razmerje med trdoto in lomno žilavostjo pri preiskovanem jeklu

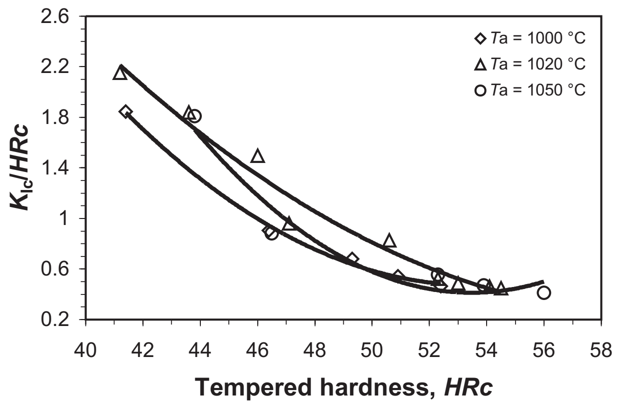


Figure 6: Ratio K_{Ic}/HRc of the investigated hot-work tool steels as a function of Rockwell-C hardness. Vacuum-heat-treatment parameters are listed in **figure 2**

Slika 6: Razmerje K_{Ic}/HRc pri preiskovanem jeklu za delo v vročem v odvisnosti od trdote Rockwell-C. Parametri vakuumske toplotne obdelave so podani na **sliki 3**.

optimising the ratio between the fracture toughness and the hardness for a given hot-work application.

The combined tempering diagram in **Figure 3** can be applied for selected heat-treatment parameters aimed at an optimal ratio between the fracture toughness and the hardness for specific hot-work tool steel, and for a given hot-work application. Thus, the properties of the investigated steel obtained with different parameters of vacuum heat treatment can be represented as a ratio of the fracture toughness and the hardness (K_{Ic}/HRc) versus the tempered hardness, (**Figure 6**).

For the investigated steel hardened from three different austenitizing temperatures, 1000 °C, 1020 °C and 1050 °C, and double tempered to the same hardness *HRc* of 45, these ratios are 1.19, 1.52 and 1.4 and 0.73, 1.08 and 0.83, after hardening from the same austenizing temperatures and double tempering to the same hardness *HRc* of 48, respectively. The highest ratios, i.e., 1.52 and 1.08, are obtained in both cases after hardening and double tempering of the investigated steel from the austenizing temperature of 1020 °C.

Nitriding treatments are established methods of improving the wear performance of tools and dies from H11 hot-work tool steel. However, the understanding of the relationship between the nitriding process parameters, and the microstructure and fracture behaviour of the surface layers is far from complete. The nitride phases that arise from these surface treatments have a substantially lower fracture toughness than the underlying substrate, and this can adversely affect the wear performance of components submitted to severe service environments involving high shear, compressive and/or impact loading conditions. For this reason, the characterisation of the relationship between the nitriding process parameters, the microstructure and the fracture behaviour of the nitride layers is crucial to ensure that these surface treatments can be used commercially with confidence.

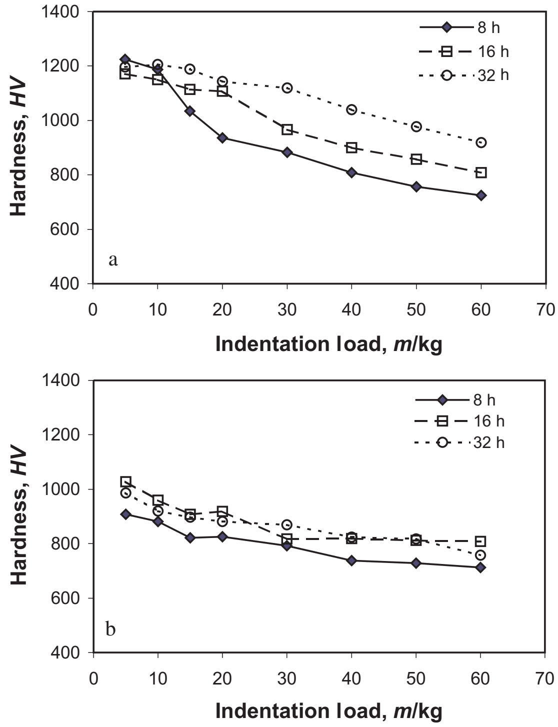


Figure 7: Surface Vickers hardness as a function of indentation load for (a) nitriding at 540 °C and (b) nitrocarburizing at 580 °C

Slika 7: Površinska trdota Hv po Vickersu v odvisnosti od obremenitve za (a) nitrirano pri 540 °C in (b) nitrocementirano pri 580 °C

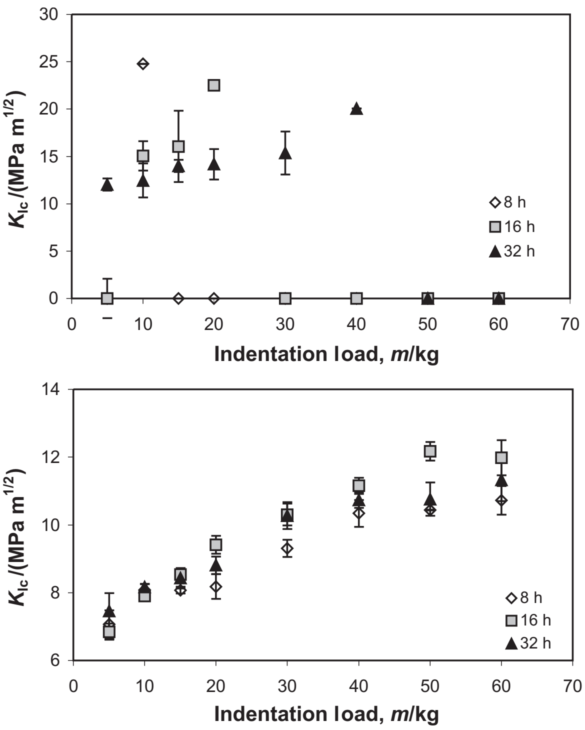


Figure 8: Mean K_{Ic} values calculated according to Equation 1 for (a) nitrided samples and (b) nitrocarburized samples. Error bars represent ± one standard deviation.

Slika 8: Srednje vrednosti K_{Ic}, izračunane z enačbo (1) za (a) nitrirane vzorce in (b) za nitrocementirane vzorce. Merilo za napako je ± en standardni odklon.

Graphs showing the surface hardness as a function of indentation load for the nitriding and nitrocarburizing treatments are presented in **Figure 7**. The 540 °C nitriding treatment produced a significantly higher hardness than the nitrocarburizing treatment at lower indentation loads for all processing times. For nitriding, increasing the process time from 8 h to 32 h appeared to produce a consistent increase in the surface hardness for a given indentation load. In the case of the 580 °C nitrocarburizing treatment, the surface hardness at all loads increased from 8 h to 16 h of treatment time; however, a longer treatment time (32 h) did not appear to result in a further increase in the surface hardness.

Graphs showing the calculated K_{Ic} values for each condition are presented in **Figure 8**. Where a condition showed no Palmqvist cracking, usually in the case when only the diffusion layer is present, a value of zero is presented for K_{Ic}. It can be seen that a valid estimate of K_{Ic0} for 8 h and 16 h of nitriding treatment (Series 1) is problematic, since Palmqvist cracking was observed only in a few cases for the longer processing time of 32 h. The nitrided sample shows consistent Palmqvist cracking at loads of 40 kg and less. In this case it may be possible to extrapolate a value for K_{Ic0} of 10–12 MPa $m^{1/2}$.

For the specimens of Series 2 the fracture toughness data allow the extrapolation to a K_{Ic0} value of 6–7 MPa $m^{1/2}$ (see **Figure 2b**). Some differences in the fracture toughness values for each condition and load do not show a consistent trend that could be correlated with the processing conditions. These variations are more likely due to the scatter of experimental measurements, and so provide an indication of the sensitivity of the test method.

The results suggest that where a sufficiently thick compound layer has formed, this method can be applied as a non-destructive method for monitoring the fracture properties of treated surfaces on actual tool parts [7].

5 CONCLUSIONS

The fracture toughness of the investigated steel can be determined using non-standard circumferentially notched and fatigue-precracked tensile-test specimens. Due to the high hardness and notch sensitivity of this type of steel, a fatigue crack can be created without affecting the measured fracture toughness in the soft-annealed specimen, i.e., before the final heat treatment, which also reduces the residual stresses in the K_{Ic}-test specimens.

On the basis of the results of extensive tests performed on conventional hot-work H11 tool steel it was confirmed that the microstructure can be substantially modified by vacuum heat treatment and the balance between the fracture toughness K_{Ic} and the hardness optimised. In other words, the fracture testing method used is sensitive to changes in the microstructure due to the different austenitizing and tempering temperatures as well as to the homogeneity of the steel.

The proposed combined tempering diagram – Rockwell-C hardness, Fracture toughness K_{Ic}, Tempering temperature – in **Figure 2** can be used for the selection of the proper vacuum-heat-treatment parameters to optimise the depth properties of the investigated steel for a given application. In particular, the combination of the theoretical method used (the concept of linear elastic fracture mechanics) with the sophisticated experimental and inspection techniques seems to be a suitable way to optimise the vacuum heat treatment of tool steels.

The ratio between the fracture toughness and the hardness (K_{Ic}/HRc) versus the tempered hardness of the investigated hot-work tool steels determined under specific vacuum-heat-treatment conditions is a reliable quantitative evaluation of the used vacuum heat-treatment processing route.

This investigation shows that the indentation fracture test method could be used to estimate the K_{Ic} fracture toughness in nitrided and nitrocarburized compound layers on tool steels, provided that the compound layer is at least 10 μm thick in order to facilitate the reliable generation of Palmqvist-type cracks at the Vickers indentations.

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