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

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ABSTRACT

Linear elastic fracture mechanics was used to optimise the vacuum-heat-treatment procedures 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 resulting from the austenitizing and tempering temperatures as well as the homogeneity of the material itself. The combined tempering diagram – Rockwell-C hardness, fracture toughness K_{Ic}, tempering temperature – was used for the choice of the vacuum-heat-treatment parameters necessary to obtain the best properties for a given application with respect to the investigated steel. Nitriding treatments are established methods of improving the wear performance of tool and die from H11 hot work tool steel. However, our understanding of the relationship between nitriding process parameters, and microstructure and fracture behaviour of the surface layers is far from complete. 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. The results suggest that where a sufficiently thick compound layer has formed, 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 test method such as Vickers indentations for fracture toughness would give useful insight into the fracture properties of nitride layers and their likely response to application conditions involving high shear or impact loading. This could be very useful as an investigative or quality control tool for industry-based surface engineering contractors.

KEY WORDS: conventional hot work tool steel, vacuum heat-treatment, fracture toughness, hardness, microstructure, nitriding, and indentation fracture, Palmqvist crack

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 mechanisms. 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. Despite the enormous variety of tooling operations there are some basic properties of tool materials that are common to almost all applications. These properties are the toughness, which prevents instantaneous fracture of the tool or tool edges due to local overload, and the hardness, which must be sufficiently high to avoid local plastic deformation. Hardness and toughness are more or less 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 ductility and toughness, in particular 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 common use; often, data determined with different test methods are available, which makes them difficult to compare, and this can lead to confusion. 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 geometry of the tool [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, therefore, 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 the solid solution, mainly affect the steel's hardness. Tempering leads to the precipitation of carbide particles and significantly decreases the carbon content in the solid solution and the dislocation density. The hardness of fully-heat-treated tool steels is therefore mainly affected by precipitates that cause precipitation hardening and, to small extent, to 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 minimum size of the specimens depends on the yield stress and the fracture toughness of the tested material, both of which are necessary for a 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 failure for different combinations of stress and crack size and for different geometries [3].

On the other hand, the compound layers arising from nitriding surface treatments have 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 conditions. Characterisation of the relationship between nitriding process parameters, microstructure and fracture behaviour of the nitride layers is therefore crucial to ensure these surface treatments can be adopted commercially with confidence. Unfortunately, the nature of these thin surface layers makes fracture testing by conventional means unviable. The use of indentation fracture testing has a number of advantages, since 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 analysis of relatively uniform bulk ceramic materials [4], many of the assumptions made in developing the equations that relate fracture toughness to observed cracking behaviour may not be entirely valid for materials where a thin brittle layer is supported by a relatively tough substrate material with varying properties by depth. Despite this, the theory and application of the method for fracture toughness testing of thin, hard coatings has more recently been considered [4].

3. EXPERIMENTAL

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

Conventional hot-work AISI H11 tool steel delivered in the shape of plates with dimensions 263 mm x 220 mm x 25 mm, cut from forged-and-soft-annealed master blocks with dimensions 263 mm x 220 mm x 4000 mm and the following chemical composition (mass content 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, e.g.: 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 minutes, gas quenched to a temperature of 100 °C. First temper was performed at 540 °C and second at different temperatures between 540 °C and 620 °C as shown in Fig. 2, each time for 2 hours, respectively. 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, metalographic specimens of 1.5 cm thickness and 2.5 cm diameter were manufactures for further pulse plasma nitriding and nitrocarburising treatment. The specimens were than plasma nitrided at 540 °C (Series 1), or nitrocarburized at 580 °C (Series 2), using 3.3 hPa pressure for nitriding and 4.3 hPa for a nitrocarburising and total gas flow rate of 75 l/h. The gas atmosphere was 75 vol. % of H_2: 25 vol. % of N_2 for nitriding and 87 vol. % of N_2: 2 vol. % of CO_2: 11 vol. % of H_2 for nitrocarburising. Heating to the process temperature took approximately 3 h and the duration of treatments for each condition was 8, 16, or 32 h.

3.2 Hardness and fracture-toughness tests

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

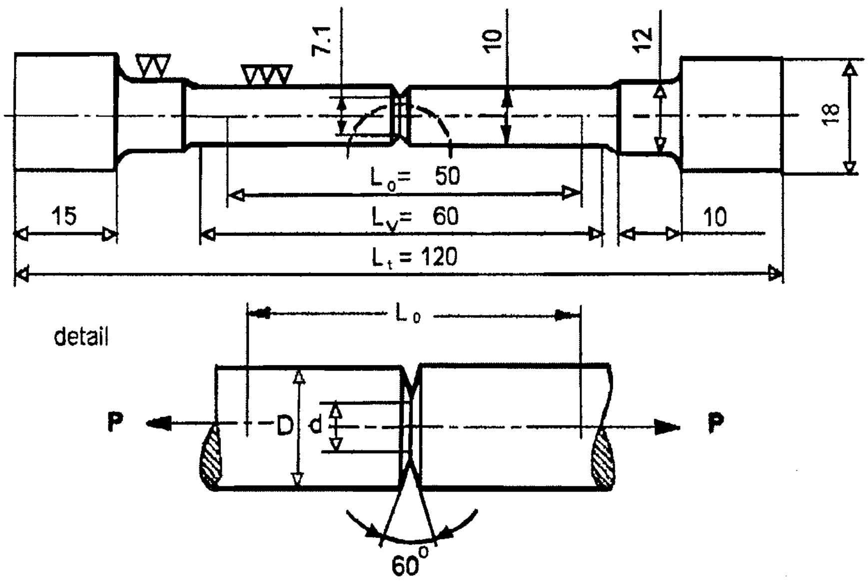
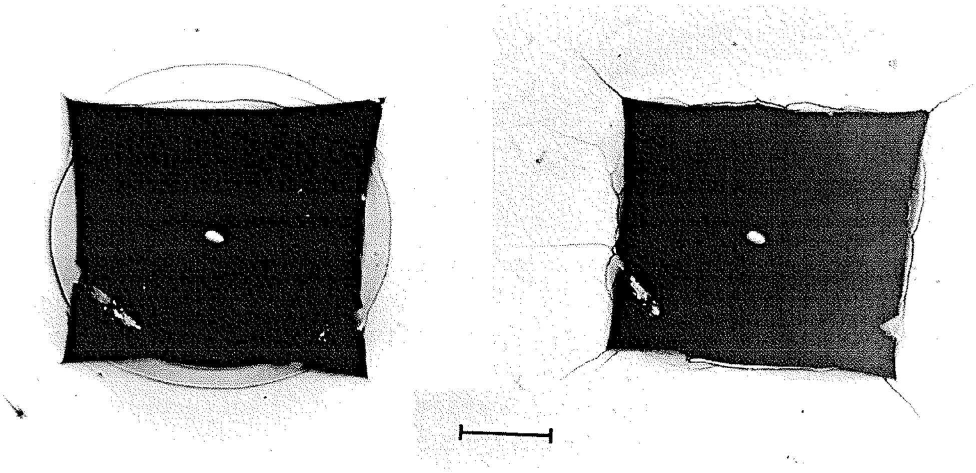


Figure 1. Circumferentially notched and fatigue-precracked K_{Ic}-test specimen; all dimensions are in mm.

The advantage of the test specimens used here over standardized CT specimens (ASTM E399-90) lies in the radial symmetry, which makes them particularly suitable for studying the influence of the microstructure of metallic materials on fracture toughness. The advantage of these specimens relates to the heat transfer, which is sufficient to provide a completely uniform microstructure. 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 our 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].

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 on brittle materials, the radial-median and Palmqvist cracking modes, figure 2.



a) b)

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

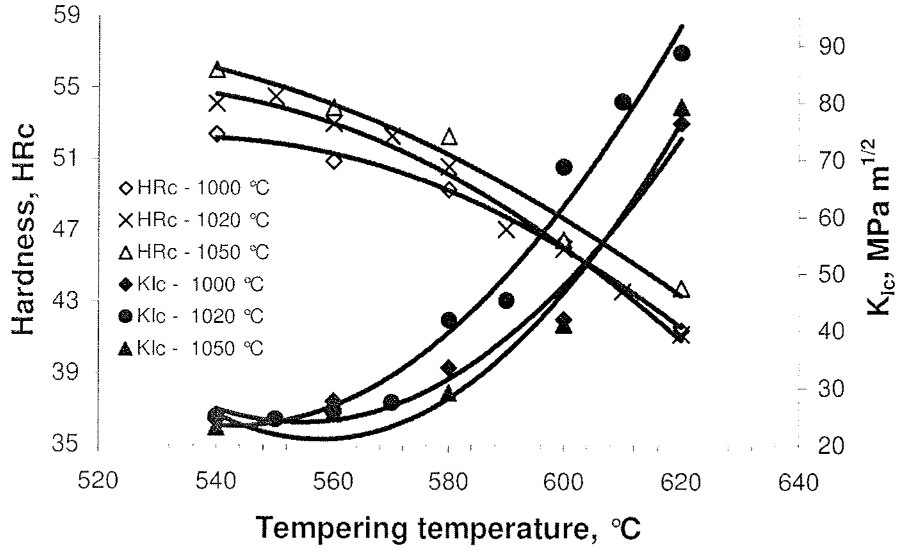
However, it has been shown that since the Palmqvist crack morphology is characterized by shallow cracks emanating from the corners of the Vickers indentation, then it is more appropriate to use the following relationship when characterizing the fracture toughness of nitride layers on tool steels [7];

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

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, 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 derived. Lightly polished treated surfaces were subjected 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 crack and indent half-diagonal lengths were used to calculate K_{Ic} according to Equation (1) above. A mean value of K_{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 normally used range of working hardness (40 to 55 HRc) in a so-called combined tempering diagram (Rockwell-C hardness, Fracture toughness K_{Ic}, Tempering temperature) in Fig. 3. It is clear that the highest fracture toughness K_{Ic} and 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_{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_{Ic} of the investigated tool steel is shown in Figure 4 for selected tempering temperatures.



K_{Ic} test specimens: circumferentially notched and fatigue-precracked tensile specimens ϕ 10 x 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 to 100°C
Cooling parameter $\lambda_{800-500}$: 1.04; 1.02; 1.11
First tempering: 1 x 2h at 540°C
Second tempering: 1 x 2h between 540°C and 620°C

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

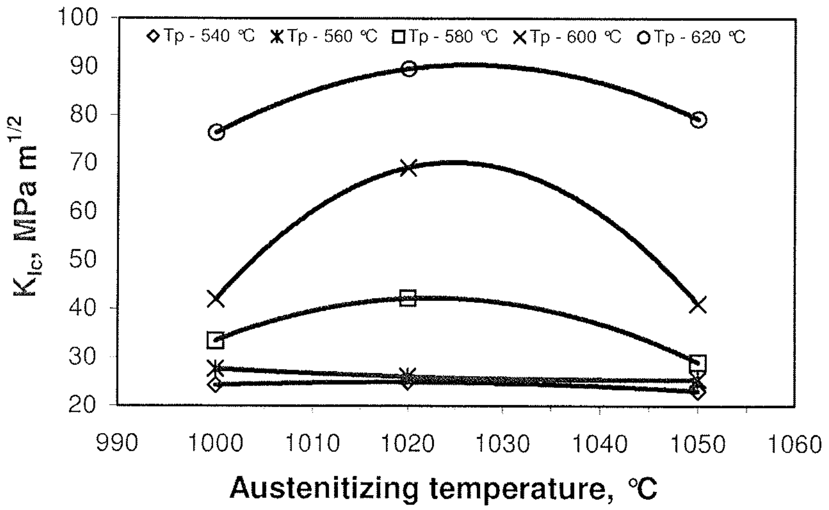


Figure 4. The influence of austenitization temperature on the fracture toughness K_{Ic} of the investigated tool steel for selected tempering temperatures. Vacuum-heat-treatment parameters are listed in Figure 3

As shown in Figure 4 the influence of austenitizing temperature on the fracture toughness K_{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 temperature, especially in the range from 580 °C to 600 °C that is generally applied for most hot-work applications, the influence of the austenitizing temperature on the fracture toughness K_{Ic}, is significant. As well as 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.

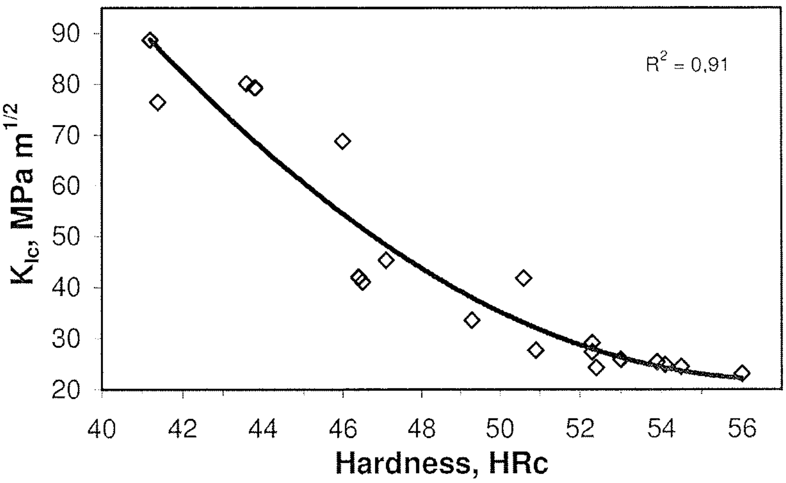


Figure 5. Relationship between the hardness and the fracture toughness of the investigated tool steel.

The correlation between the hardness and the fracture toughness is fairly good for all three austenitizing temperatures. At a particular hardness, i.e., the normally used working hardness between 45 HRc and 48 HRc, 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 optimising the ratio between the fracture toughness and the hardness for a given hot-work application.

However, 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. The properties of the investigated steel obtained with different parameters of vacuum heat treatment can thus be represented as the ratio of the fracture toughness to the hardness (K_{Ic}/HRc) versus tempered hardness, Figure 6.

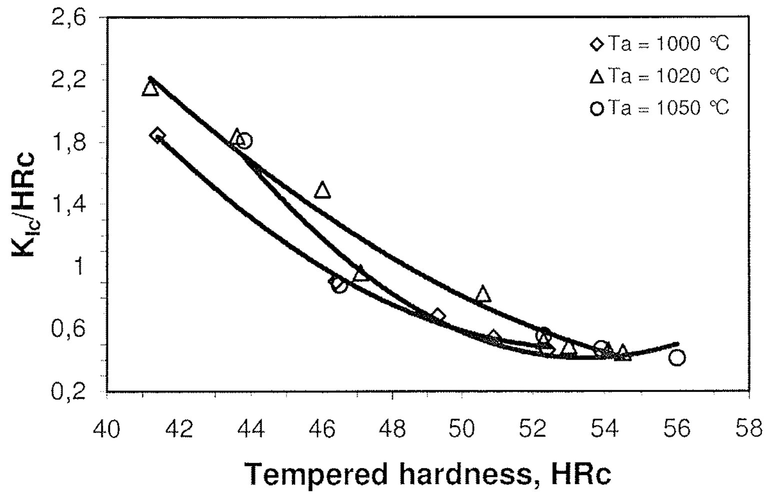
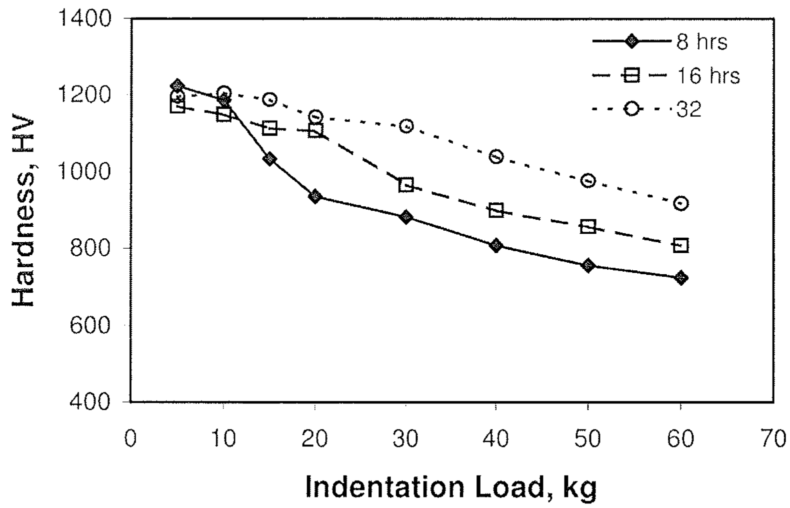


Figure 6. Ratio K_{Ic}/HRc of the investigated hot-work tool steels as a function of Rockwell-C hardness. Vacuum-heat-treatment parameters are listed in Figure 2.

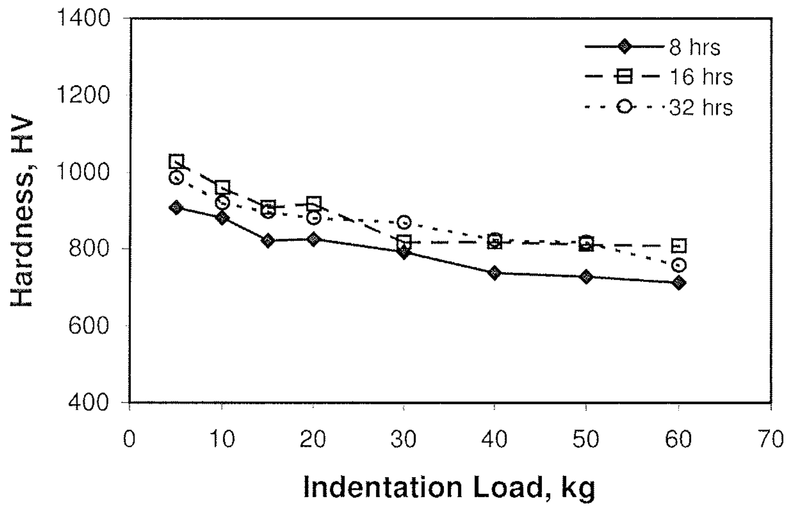
For the investigated steel hardened from three different austenitizing temperatures, 1000 °C, 1020 °C and 1050 °C, and double tempered to the same hardness of 45 HRc, these ratios are 1.19, 1.52 and 1.4 and 0.73, 1.08 and 0.83, after hardening from the same austenizing temperatures and double tempering to the same hardness of 48 HRc, 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 tool and die from H11 hot work tool steel. However, our understanding of the relationship between nitriding process parameters, and microstructure and fracture behaviour of the surface layers is far from complete. The nitride phases that arise from these surface treatments have 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 conditions. For this reason, characterisation of the relationship between nitriding process parameters, microstructure and fracture behaviour of the nitride layers is crucial to ensure these surface treatments can be adopted commercially with confidence.

Graphs showing 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 significantly higher hardness than the nitrocarburizing treatment at lower indentation loads for all processing times. For nitriding, increasing process time from 8 to 32 hours appeared to have a consistent increase in surface hardness for a given indentation load. In the case of the 580°C nitrocarburizing treatment, surface hardness at all loads increased from 8 hours to 16 hour treatment time, but a longer treatment time (32 hours) does not appear to result in further increase in surface hardness.



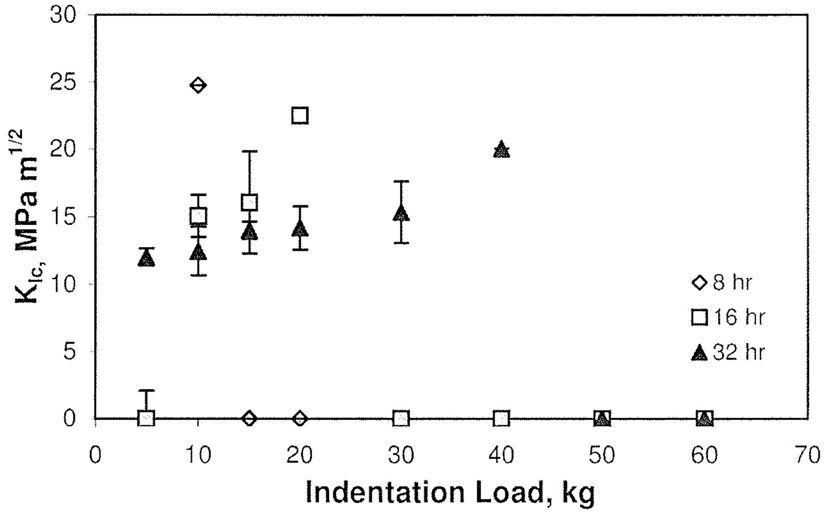
a)



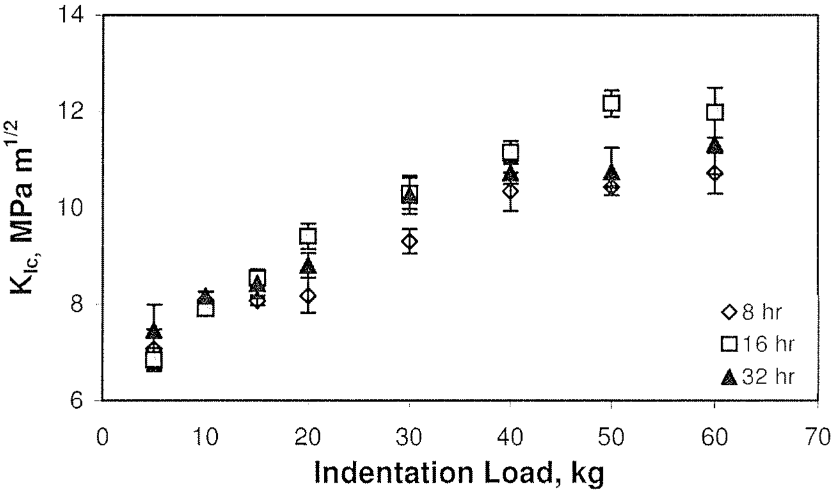
b)

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

Graphs showing the calculated K_{Ic} values for each condition are presented in Figure 8. Where a condition showed no Palmqvist cracking, a value of zero is presented for K_{Ic}. It can be seen that a valid estimate of K_{Ic0} for 8 and 16 hour nitriding treatments (Series 1) is problematic, since there are so few cases where Palmqvist cracking is evident. At the longer processing time of 32 hrs, the nitrided sample shows consistent Palmqvist cracking behaviour at loads of 40kg and less. In this case, it may be possible to extrapolate a value for K_{Ic0} in the order of 10-12 MPa.m$^{1/2}$.



a)



b)

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.

In the case of Series 2 materials, there are clear trends in the fracture toughness data, which enable extrapolation to a K_{Ic0} value of between 6-7 MPa.m$^{1/2}$ (see Figure 2(b)). Although there are some differences in fracture toughness values for each condition and load, there is no consistent trend apparent that can be correlated with processing conditions. These variations are more likely due to experimental errors associated with the measurement, 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 has the potential to be applied as a pseudo non-destructive method of 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, the fatigue crack can be created without having an effect on 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 we have confirmed that the microstructure can be substantially modified by vacuum heat treatment, with the aim to optimise the balance between the fracture toughness K_{Ic} and the hardness. 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 proposed combined tempering diagram – Rockwell-C hardness, Fracture toughness K_{Ic}, Tempering temperature – in Fig. 2 can be used for the selection of the proper vacuum-heat-treatment parameters to obtain optimised 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 to the hardness (K_{Ic}/HRc) versus tempered hardness of the investigated hot-work tool steels determined under specific vacuum-heat-treatment conditions is a reliable quantitative assessment of the used vacuum heat-treatment processing route.

The current work shows that the indentation fracture test method can be used to estimate K_{Ic} fracture toughness in nitrided and nitrocarburized compound layers on tool steels, provided that the compound layer is at least 10um in thickness in order to facilitate the reliable generation of Palmqvist-type crack morphology at the Vickers indentations.

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References

[1] R. Ebner, H. Leitner, F. Jeglitsch and D. Caliskanoglu: Methods of Property Oriented Tool Steel Design, Proceeding of 5th International Conference on Tooling, Leoben,Sept.29th to October 1st 1999, University of Leoben, Austria, 3-24

[2] H. Leitner: Einfluss der Austenitkorngrosse auf die Zähigkeit des Schnellarbeitsstahles S 6-5-2-5, Diplome Thesis, University of Leoben (1996)

[3] M. Janssen, J. Zuidema, R.J.H. Wanhill: Fracture Mechanics, 2-nd Edition, Delft University Press, Delft, Netherlands, 2002

[4] D. Nolan, V. Leskovsek, M. Jenko, Indentation fracture testing of nitrided layers on H11 tool steel, Proceeding of 1-st international conference on heat treatment and surface engineering of tools and dies, 8-th to 11-th June 2005, Pula, Croatia, 137-144

[5] B. Ule, V. Leskovsek, B. Tuma: Estimation of plain strain fracture toughness of AISI M2 steel from precracked round-bar specimens, Engineering Fracture Mech 2000; 65, 559-572

[6] Wang Chang: An improvement of applying similarity methods to fracture mechanics-measurement of fracture toughness K_{Ic}, K_Q values by small-scale single specimen, Engng Fracture Mech 1990;36, 313-20

[7] D. Nolan, V. Leskovsek and M. Jenko, Estimation of fracture toughness of nitride compound layers on tool steel by application of the Vickers indentation method, *Surface and Coatings Technology*, In Press, Corrected Proof, *Available online* 13 December 2005