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INDENTATION FRACTURE TESTING OF NITRIDED LAYERS ON H11 TOOL STEEL

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ABSTRACT

Nitriding and nitrocarburising treatments are well accepted methods of improving the wear performance of tool and die steels. However, our understanding of the relationship between nitriding process parameters, 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. This paper describes an investigation of the application of indentation fracture testing to nitrided and nitrocarburized H11 hot work tool steel. 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.

Keywords: Nitriding, Nitrocarburizing, Indentation fracture, Hardness testing, Palmqvist cracks

1. INTRODUCTION

Despite the fact that nitriding and nitrocarburising treatments are well accepted methods of improving the wear performance of tool and die steels, our understanding of the fracture properties of these hard surfaces 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. Unfortunately, the very nature of these relatively thin surface layers makes fracture testing by conventional means unviable.

One method that has the potential to fulfil this requirement involves the use of indentation hardness testing as a pseudo non-destructive test for fracture toughness. The use of indentation fracture testing has a numberof advantages, since it relies on a 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 [1,2,3,4,5,6], 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 [7,8,9,10,11].

There are two basic cracking modes possible from Vickers indentations on brittle materials, the radial-median and Palmqvist cracking modes. The radial-median mode derives from sub-surface median cracks that initiate along the edges of the pyramidal indentation and extend deep into the material in a semi-circular manner perpendicular to the surface (hence the term "halfpenny-shaped" cracking). The Palmqvist crack morphology is characterized by much shallower cracks emanating from the corners of the Vickers indentation. It would seem reasonable to assume that for relatively thin brittle films on relatively tough substrates, such is the case for a nitrided tool steel, then it would be more appropriate to use the relationships based on the Palmqvist crack morphology, as this model is based on cracking initiating at the surface where the material is more brittle, rather than at depth, possibly beyond the extent of the compound layer in a nitrided sample. Indeed, the work on characterization of fracture toughness of Ni-P films deposited on tool steel by Bozzini et al [7] used the simplified relationship developed by Shetty et al [3] which is valid for the Palmqvist crack mode;

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

where P is the indentation load, a is the mean diagonal half length and l is the mean crack length. Note that K_{Ic} is assumed to be equivalent to K_c, the critical stress intensity for cracking in the Vickers indentation tests. Later work by Boniardi et al [8] claims to have successfully applied the indentation fracture method to determine crack-arrest fracture toughness of nitrided surface layers on case hardened Cr-Mo steels. However, it would appear that the calculations for K_{Ic} contained therein used an equation that was developed by Evans et al [12] for materials exhibiting the radial–median crack mode. Further, the relationships used by other workers [10,11] to define fracture toughness of hard brittle films on metallic substrates are based on equations that are valid for radial-median cracking modes. It has been suggested that, strictly speaking, the above equations will give an estimate of the crack arrest fracture toughness, K_{Ia}, rather than K_{Ic} [7]. However, the bulk of the literature does not discriminate in this way, and so K_{Ic} will be used in the present work. 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.

The fracture indentation method offers numerous potential benefits in terms of characterization of the mechanical properties of hard, thin coatings in industrial environments.

However, there are inconsistencies in the way that this method has been applied in previous work. The current work was undertaken to investigate the application of the indentation fracture test method to nitrided and nitrocarburized surface layers on H11 tool steel. The objective is to determine whether Equation 1 above (for Palmqvist cracking mode) can be used to give a valid and reliable measure of fracture toughness of nitrided compound layers on nitrided H11 tool steel.

2. EXPERIMENTAL MATERIALS AND METHODS

The tool steel investigated was ESR AISI H11, with nominal composition 0.38 %C, 5 %Cr, 1.3%Mo, 0.4 %V. The steel was heat treated in a horizontal vacuum furnace with uniform high-pressure gas-quenching using nitrogen (N_2) at a pressure of 1.05 bars. The specimens were heated at 10°C/min to the austenitizing temperature of 1020 °C, soaked for 30 minutes, gas quenched to a temperature of 100 °C, and then double tempered. The first temper was performed at 540 °C for 4 hours and the second at 585 °C for 4 hours. The specimens were then plasma nitrided at 480 °C (sample A) or 540 °C (sample B), or nitrocarburized at 580 °C (sample C), using 300 hPa pressure and a total gas flow rate of 75 l/h. The gas atmosphere was 25 %N_2–75 %H_2 for nitriding, and 87 %N_2–2 %CO_2–11 %H_2. Heating to process temperature took approximately 3 hours and the treatment duration was 16 hours.

In order to facilitate accurate measurement of the dimensions of indentations and cracks, the surface of each sample was lightly polished using 1μm diamond paste in order to provide a mirror finish while ensuring minimal loss of compound layer thickness. The polished surfaces were then subjected to Vickers hardness testing at loads of 5, 10, 15, 20, 30, 40, 50, 60 and 100 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 value of K_{Ic0} is then derived by extrapolation to a zero load condition. Samples were cut and mounted in bakelite in cross-section, and prepared metallographically for analysis of microstructure and hardness depth profiles. Microhardness depth profiling was conducted using a Fischerscope H100C machine at a load of 0.1N (~10g) and hardness of near surface hardness in cross-section was measured at a load of 0.002N (~0.2g). Microstructures were revealed by etching with 2% nital solution and compound layers were etched with Marbles reagent.

3. RESULTS

A graph showing surface hardness as a function of indentation load is presented in Figure 1. The nitriding treatments produced higher hardness than the nitrocarburizing treatment at lower indentation loads. However, at higher loads the low temperature nitriding treatment showed hardness values ~100HV lower than for the higher temperature nitriding and nitrocarburizing treatments. In relation to the appearance of indentations and the mode of cracking observed, sample A produced only one case of Palmqvist cracking. The predominant mode of cracking in sample A was concentric rings, becoming apparent at 20 kg load and becoming more extensive with increasing load. An example of such concentric cracking is shown in Figure 2(a). Sample B showed some minor Palmqvist-type cracking at loads of 5, 10, 15, 20 and 60 kg loads. However, at 60 kg load, the concentric cracking predominant for sample A also became apparent in sample B, becoming more significant with increasing load. In contrast, sample C was characterized by very distinct Palmqvist cracking at all indentation loads (see Figure 2 (b)).

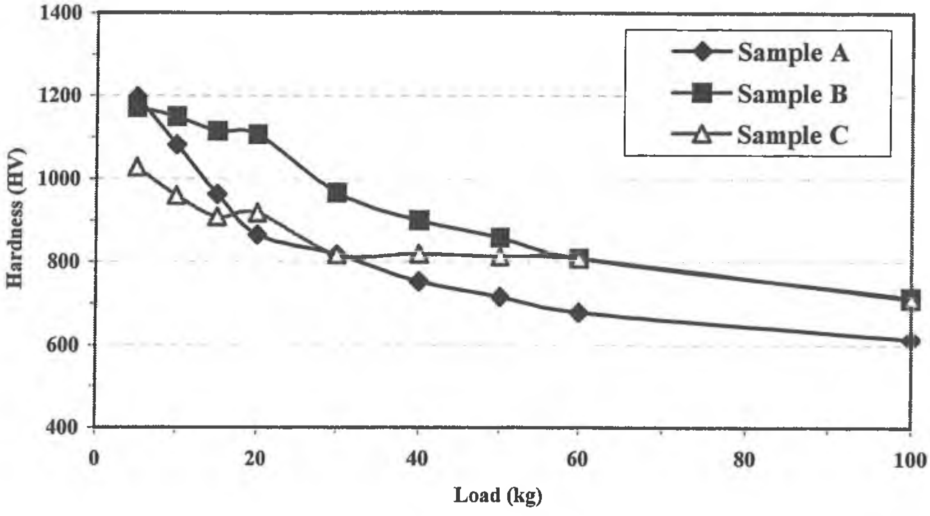


Figure 1: Graph showing surface Vickers hardness as a function of load.

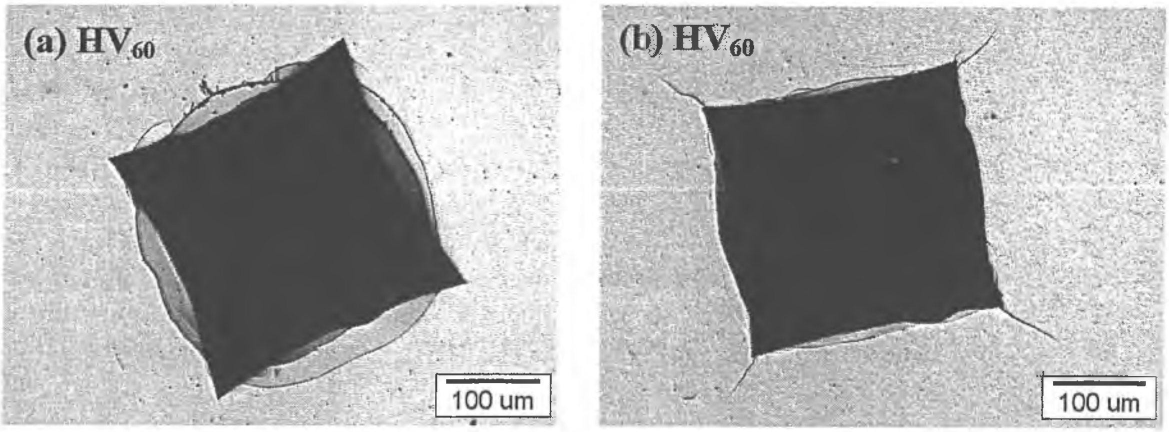


Figure 2: Images showing typical examples of (a) concentric ring cracks in sample A, and (b) Palmqvist cracks in sample C.

A graph showing the calculated K_{Ic} values for each condition is presented in Figure 3. 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 sample A is not possible, since there is only one case where Palmqvist cracking was evident. In the case of sample B, it may be possible to extrapolate a tentative value for K_{Ic0} in the order of 10-15 MPa.m$^{1/2}$, but the limited data and inconsistency of results suggests this derivation may not be reliable. However, in the case of sample C, there is a clear trend of fracture toughness data which enables extrapolation to a K_{Ic0} value of ~7 MPa.m$^{1/2}$. It should be noted that these values are of similar magnitude to those previously reported by Boniardi et al [8] for nitrided and nitrocarburized layers on a 0.3 %C-3.0 %Cr-0.35 %Mo steel.

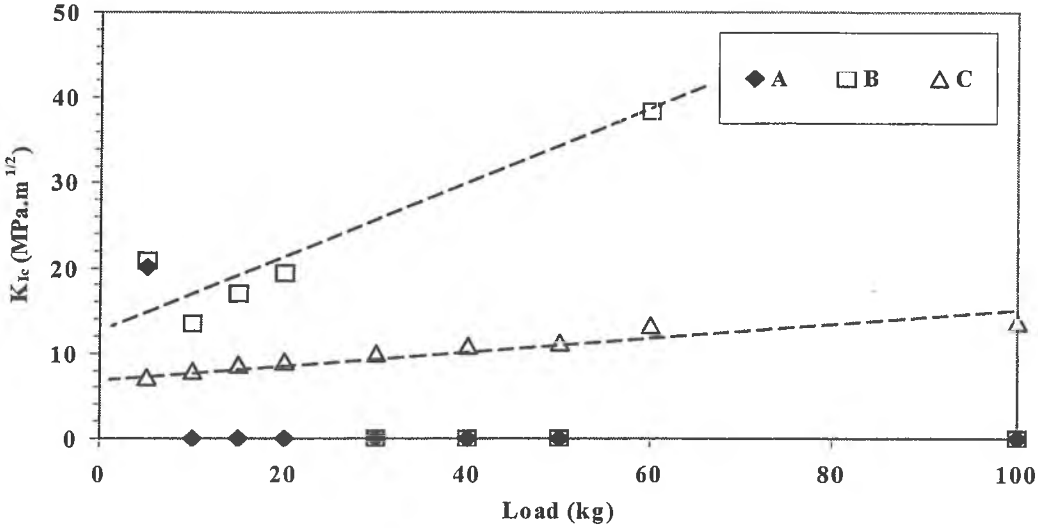
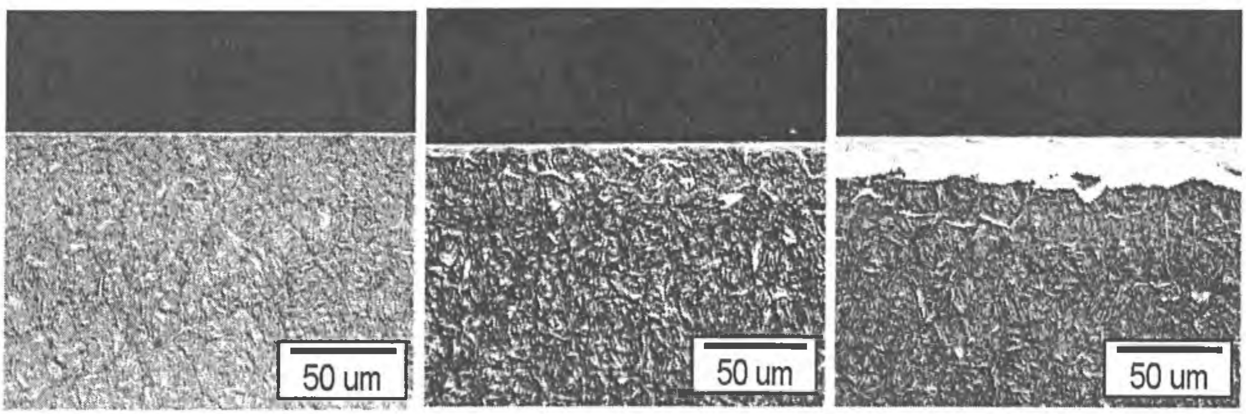


Figure 3: Graph showing calculated values for K_{Ic}.

Microstructures of the 3 materials are presented in Figure 4. It can be seen that sample A has no compound layer evident on the surface. Sample A was also characterized by a slow etching response, and a case depth of 80-100 µm. Sample B has a thin compound layer of <5 µm. It was characterized by a stronger response to nital etching and revealed a case depth of around 120µm. Sample C has a thick compound layer of ~20 µm and a deep case of 250-300 µm, revealed as a result of a very strong etching response. Etching with Marbles reagant showed the compound layer on sample B was composed only of γ'-Fe_4N phase, while the compound layer on sample C was composed of a mixture of γ' and ε ($Fe_{2\text{-}3}C_xN_y$) phases.



(a) (b) (c)

Figure 4: Micrographs showing microstructures of (a) sample A, (b) sample B and (c) sample C

Microhardness depth profiles for the three materials, obtained using an indentation load of 0.1N (10g), are presented in Figure 5. It can be seen that increasing the nitriding temperature from 480°C to 540 °C results in a similar hardness at the surface, but a deeper case-hardening effect. Nitrocarburizing at 580 °C results in a lower near-surface hardness, but a less severe decrease in hardness with depth. It should be noted that a load of 0.1N, it is only possible to measure hardness to within ~20 μm of the surface, due to the size of the indentation and interference effects from the free surface. Even using a load of 0.002 N (~0.2 g), it was not possible to accurately measure the hardness of the compound layer on the nitrided sample B, although indications are that the surface hardness at this load would be ~1250HV. Low load measurements of the surface hardness of the nitrocarburized layer on sample C show it to be ~1150HV. It might normally be expected that the ε-containing compound layer on sample C would be harder than the γ' layer on the nitrided sample(s). The lower hardness observed may be a result of very fine closed porosity apparent in the mixed phase compound layer.

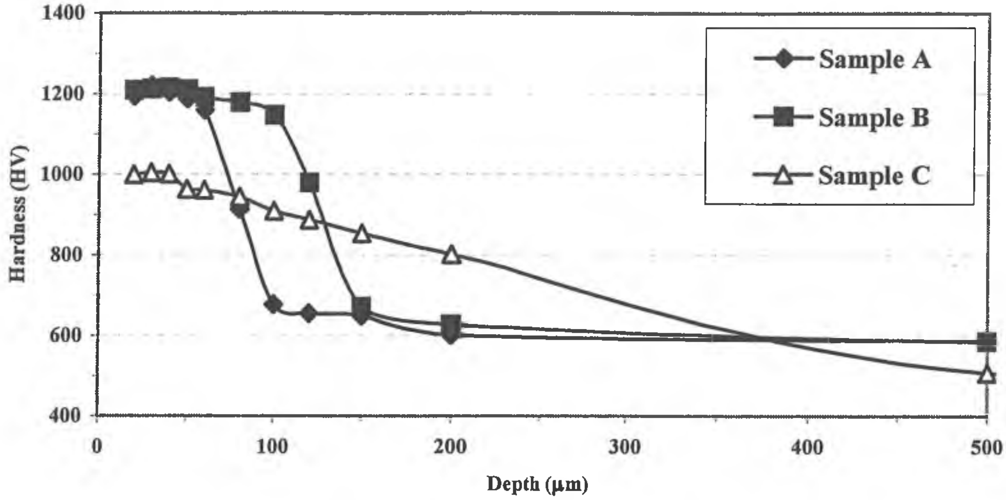


Figure 5: Graph showing microhardness depth profiles at a load of 0.1N.

4. DISCUSSION

The results suggest that the valid application of the indentation fracture test method to nitrided layers on tool steel is dependent on the presence of a relatively thick compound layer. Where no discrete compound layer is present, as was the case for sample A, the microstructure is not sufficiently brittle to enable initiation of fracture at the corners of the Vickers indentations. Despite having high hardness, the diffusion layer appear to have sufficient toughness for the fracture mode to be dominated by the compressive forces acting laterally as material is forced to deform plastically along the flat faces of the Vickers indentation. In the case of sample B, where a thin compound layer (<5 μm) is apparent, the surface layer exhibits some Palmqvist-type cracking. However, it would appear that some substantial compound layer thickness is required before this material response is reliable enough to validate the fracture toughness measurement. For example, where the compound layer is 20 μm thick (see sample C), the Palmqvist cracking becomes sufficiently reliable to enable valid estimation of K_{Ic0} fracture toughness, according to relationships previously derived. It may also be that, as the case-hardening effect is more progressively more substantial from samples A to B to C, the substrate material may be providing greater support for the developing compound layer, resulting in an enhancement of the brittle fracture response. However, there is insufficient evidence thus far to comment further on this matter. Further work is required to determine whether the fracture response of the surface layer(s) is dependent in some way on case depth.

In light of these findings, it is suggested that the indentation fracture methodology offers the possibility of determining estimates of fracture toughness of nitrided and nitrocarburized surface layers on tool and die applications. However, it is clear that more work is required to properly define the limitations of the method, in relation to compound layer thickness and statistical validity.

5. CONCLUSIONS

The present work has shown that the indentation fracture test method has the potential to be used to estimate fracture toughness of compound layers on nitrided tool steel parts, providing sufficient compound layer thickness. Use of such a simple test method for fracture toughness would give useful insight into the fracture properties of such 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. However, this work is by no means exhaustive, and a number of important issues remain to be investigated, particularly in relation to the effect of case depth and compound layer thickness on fracture toughness estimations.

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