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Estimation of fracture toughness of nitride compound layers on tool steel by application of the Vickers indentation method

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Abstract

Nitriding and nitrocarburizing treatments are established methods of improving the wear performance of tool and die steels. However, our understanding of the relationship between nitriding process parameters, and microstructure and fracture behaviour of the surface layers is far from complete. This paper describes an investigation of the application of Vickers indentation fracture testing to nitrided and nitrocarburized AISI H11 hot work tool steel. Optical microscopy and microhardness testing were used to characterize structure and mechanical properties of the compound and diffusion layers. The results show 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. However, the validity of the method appears to be a function of the presence and thickness of the compound layer, and possibly the mechanical properties of the diffusion layer, and further work is proposed to establish the sensitivity of the method for fracture toughness calculations.

Keywords: Nitriding; Nitrocarburizing; Fracture toughness; Indentation fracture; Hardness testing; Palmqvist cracks

1. Introduction

Despite the fact that nitriding and nitrocarburizing treatments are well-established 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, characterization of the relationship between nitriding process parameters, and 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 number of 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 [1–6], 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 [7–11].

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

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term "halfpenny-shaped" cracking). The Palmqvist crack morphology is characterized by much shallower cracks emanating from the corners of the Vickers indentation. In brittle bulk materials, such as glasses, ceramics and ceramic composites, the possibility of a transition in cracking mode from Palmqvist to radial-median as a function of indentation load and c/a ratio (where c is the total crack length and a is the indentation diagonal half-length) has been previously discussed [4,12]. 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 and Boniardi [7] used the simplified relationship developed by Shetty et al. [3], which is valid for the Palmqvist crack mode:

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

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 and Charles [13] for materials exhibiting the radial-median crack mode. Similarly, 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, although this fact was not explicitly stated.

It has been suggested that, strictly speaking, the above equation 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 Eq. (1) above (for Palmqvist cracking mode) can be used to give a valid and reliable measure of fracture toughness for nitrided compound layers on nitrided H11 tool steel.

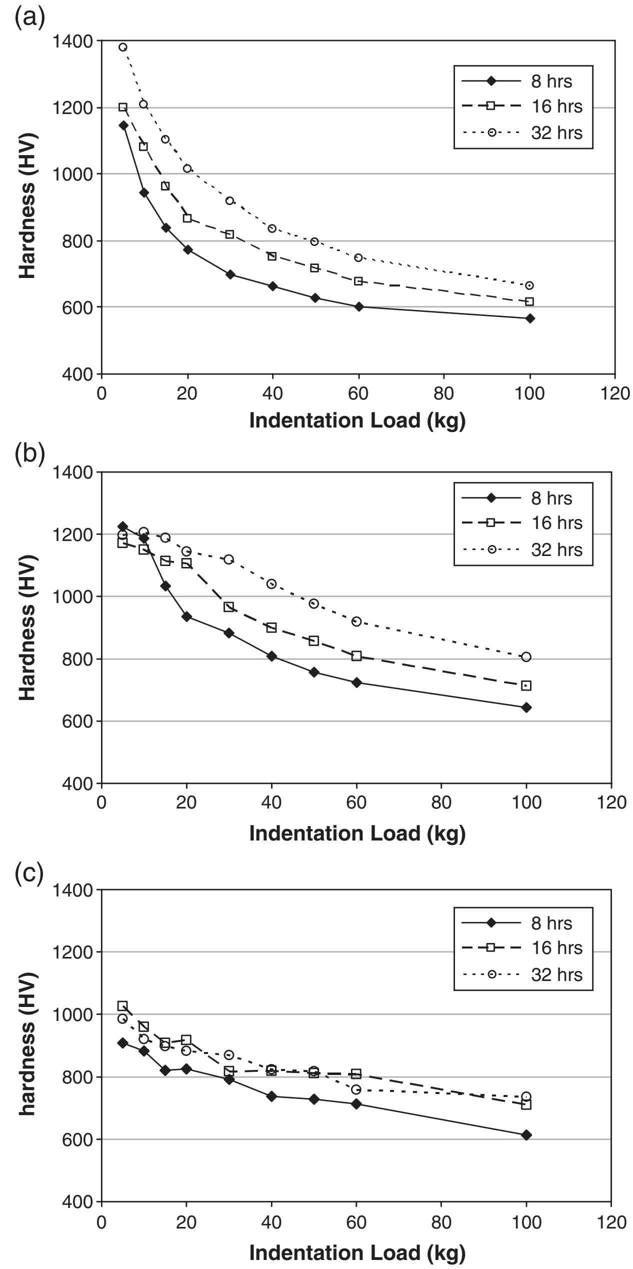

Fig. 1. Graphs showing surface hardness as a function of indentation load for processing temperatures of (a) 480 °C, (b) 540 °C and (c) 580 °C.

2. Experimental

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 bar. The specimens were heated at 10 °C/min to the austenitizing temperature of 1020 °C, soaked for 30 min, gas quenched to a temperature of 100 °C and then double tempered.