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## Variations in the microstructure and mechanical properties of the oxide layer on high speed steel hot rolling work rolls

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#### ABSTRACT

This paper is part of a larger study to understand the wear of hot rolling rolls. A significant cost of the hot rolling process is associated with the consumption of rolls, which is why a comprehensive understanding of the wear of the roll material is important. Given that the surface of the rolls is covered by an oxide layer, it is important to know the tribological and mechanical properties of the oxides. Research in this area concentrates mainly on the morphologies and microstructures of the oxide layers. Previously published works give very little, if any, information of the mechanical properties of the layers on high speed steel. This paper presents a methodology to study the mechanical properties of the oxide layer formed on the surface of a high speed steel roll using combined nanoindentation tests and finite element simulations. Mechanical properties such as the elastic modulus (E), hardness (H), yield strength  $(\sigma_y)$ , and Poisson's ratio  $(\nu)$ , have been determined, and the work has revealed a variation of microstructure, porosity (f), and mechanical properties of the oxide layer across its thickness. The outer sub-layer has a higher E and H than the inner sub-layer. This variation of mechanical properties in the oxide layer was consistent with variations in the porosity and grain sizes in the two sub-layers.

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#### 1. Introduction

In the hot rolling of steel process, high speed steel (HSS) work rolls have been widely used because of their excellent mechanical properties and resistance to wear at elevated temperatures (>700 °C). Under these circumstances, oxides can rapidly form on the surface of work rolls by a mechanism that is controlled by the diffusion of ionic species across the crust as described by Garza-Montes-de-Oca et al. (2011). Garza-Montes-de-Oca and Rainforth (2009) elaborated that this oxide layer can be considered as a protective coating that controls the heat transfer, wear and friction conditions at the interface formed between the strip and roll. Therefore the life of work rolls is strongly related to the mechanical properties of this layer. As the wear of the roll substrate depends on the wear resistance of the oxide layer, it is important that the mechanical properties of this layer be known accurately in hot roll wear studies.

Molinari et al. (2000) showed that the layers of oxide on a HSS surface are multi-layered, with three distinct sub-layers. At  $700 \,^{\circ}$ C,

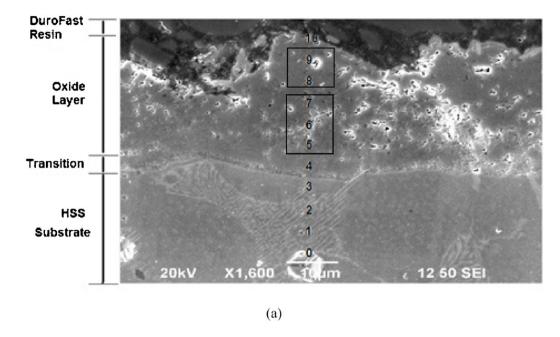
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the oxide can be divided into a thin  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> outer sub-layer, a thick and porous  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> intermediate sub-layer, and an M<sub>3</sub>O<sub>4</sub> spinel (M = Fe, Cr) inner sub-layer. Due to the micron size thickness of this oxide layer, and even thinner sub-layer, a cross sectional nanoindentation technique has been used by Hosemann et al. (2008) and Xia et al. (2006) to measure the hardness and elastic modulus of the oxide scale of ferritic/martensitic steels, and a  $\gamma$ -TiAl based alloy. Based on the measured load–displacement curves, the elastic modulus and hardness of the sample contact are determined using the Oliver and Pharr approach (1992).

Xia et al. (2006) investigated the mechanical properties of the oxide layer formed by thermal oxidation on a  $\gamma$ -TiAl based alloy using nanoindentation and nanoscratch tests. The nanoindentation tests were carried out in the top TiO<sub>2</sub> sub-layer, the inner  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> rich sub-layer, the diffusion zone, and the substrate, with the maximum load of 20 mN. It was found that the mechanical properties vary across the depth of the oxide layer, which may be attributed to different sub-layer microstructures of the oxide.

Hosemann et al. (2008) investigated the mechanical properties of the layers of oxide formed on ferritic/martensitic steels using nanoindentation tests. The elastic modulus and hardness of the layers of oxide on the surface of the sample were found to be lower than its values in a dense form. These lower values may be attributed to a higher porosity of the oxide layer than its dense form, although the actual porosity was not quantified. Based on an

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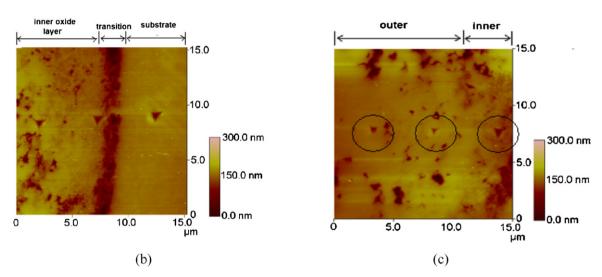


Fig. 1. (a) Distribution of the indentation locations in the oxide layers. An AFM analysis of the indents: (b) from the substrate to the inner sub-layer and (c) from the inner to the outer sub-layer.

SEM image of the cross sections of the layer, Hosemann et al. (2008) confirmed that the inner oxide sub-layer has a lower elastic modulus and hardness, which can be explained by the higher porosity of the inner sub-layer.

Although the oxide scale plays a significant role in the tribological performance of a work roll in hot rolling, to the best of our knowledge, only a few studies have been done on the mechanical properties of oxide layers formed on HSS surfaces. Nicholls et al. (1994) compared different techniques to measure the hardness and elastic modulus of the bulk oxides  $Al_2O_3$  and  $Cr_2O_3$  and then compared them with other types of oxides, including FeO,  $Fe_2O_3$  and  $Fe_3O_4$ . However they only measured the mechanical properties of bulk oxide systems. Therefore, the mechanical properties of the component layer of oxide formed on HSS surfaces remains unknown, and they need to be determined.

Nanoindentation tests can only measure some mechanical properties of the oxide layer. One of the parameters that cannot be obtained from the load-displacement analysis is the porosity of the oxide layer. In a porous media the load displacement relationship

depends on the porosity and grain sizes, which are largely heterogeneous. Moreover, from the measured load-displacement curve, and based on the Oliver and Pharr method (1992), only a reduced elastic modulus  $(E/1 - v^2)$  can be obtained directly, not the sample elastic modulus E due to the unknown Poisson ratio value, and which is dependent on porosity. In this work, the mechanical properties of the oxide layer, and the elastic modulus and hardness, were determined using combined nanoindentation tests (with 5 mN and 20 mN maximum loads) and finite element (FE) simulations. The mechanical properties of the oxide sub-layers that cannot be directly measured from the nanoindentation tests e.g. elastic modulus, E, yield strength,  $\sigma_v$ , Poisson's ratio,  $\upsilon$  and pore fraction, f were determined from the FE simulations input parameters. The parameters were optimum parameters that yielded a very close agreement between the simulated and measured nanoindentation load displacement curves. The finite element model used Gurson's model of plasticity for porous material. Information obtained from this paper will be used to further understand the mechanics of friction and the wear of high speed steel rolls in practice.

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