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The effect of in-service work hardening and crystallographic orientation on the micro-scratch wear of Hadfield steel

P.C. Machado [a,b,*], J.I. Pereira [a], J.J. Penagos [a,c], T. Yonamine [d], A. Sinatora [a,c]

[a] *Surface Phenomena Laboratory, Department of Mechanical Engineering, Polytechnic School of the University of São Paulo, Brazil*
[b] *Department of Mechanical Engineering, Federal University of Pará, Brazil*
[c] *Instituto Tecnológico Vale, Av. Juscelino Kubitschek 31, Bauxita, 35400-000 Ouro Preto, MG, Brazil*
[d] *Institute for Technological Research of the State of São Paulo, Av. Prof. Almeida Prado 532, 05508-901 São Paulo, SP, Brazil*

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ABSTRACT

This work presents the micro-scratch wear analysis of a sample of Hadfield steel previously used in a jaw crusher machine. The in-service work hardening material derived from dynamic high loads during the abrasive contact on the steel surface. The scratch resistance of hardened samples was evaluated by micro-scratch tests on a cross-section surface. Scratches were made on a deformed layer and on an undeformed material. In the undeformed region, the two crystallographic planes 001 and 111 were analyzed. The scratch tests were performed under various normal loads (from 20 mN to 250 mN) with a diamond cone stylus of 60° and a 5 µm radius. The electron microscopy techniques (FEG, FIB, and EBSD) were applied to characterize the microstructure, the wear micromechanisms, and the change of the submicrostructure due to scratches and crystallography. Additionally, an optical interferometry analysis was done for the topography characterization of the scratches. The microhardness profile analysis showed a variation of the hardness values from 300 to 700 $HV_{0.3}$, which resulted from the twinned grains found near the worn surface. Low loads (20 mN and 50 mN) corresponded to mild wear regime, with a wear regime transition at 100 mN. A severe wear regime corresponded to higher loads (150 mN, 200 mN, and 250 mN). A significant variation of the friction coefficient along with the severely hardened layer for low normal loads was not observed. On the other hand, high loads resulted in significant variations of the friction coefficient, without correlation with microhardness profile. Additionally a strong relationship was observed between the scratch wear and the individual grain crystallographic orientation. For scratches in the (001) plane microploughing was established as the predominant wear micromechanism, whereas for (111) plane microcutting was identified as the micromechanism responsible for wear, presenting a higher friction coefficient than for the (001) plane.

1. Introduction

Hadfield steel is commonly used in gouging abrasion and impact-abrasion conditions, because of its high toughness, work-hardening capacity, and strong wear resistance. Critical machinery parts for ore crushing in primary and secondary stages of the mining process are among the most common applications of Hadfield steel, depending on the level of hardening, combination of chemical element contents and heat treatment process [1]. Usual chemical compositions are defined in ASTM Standard A128 [2] with nine different grades depending on their carbon, manganese, chromium and molybdenum content.

The interdependence of hardening and wear resistance of some steels has been discussed by different authors [1–3] although not reaching a unified conclusion. Moore [2] analyzed the abrasion resistance of steels with different chemical compositions, cold work and heat treatments. However, the increase in hardness after wear (with high load and large size of abrasive particles) was not considered for the examined austenitic steels in this study. Avery [1] showed that the work-hardening capacity of Hadfield steel can reach a hardness up to 700 HV in strain-hardened conditions. Lindroos et al. [3] explained the difficulty in understanding the relationship between the hardening of the outer layer and the wear resistance, is caused by the fact that only the initial hardness is commonly reported.

The hardening mechanism was analyzed by Lindroos et al. [4], using a scratch schematic model of the abrasive process. The authors [4] determined the compression stress as the main stress while abrasive particles pass on to the surface. The mechanical

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[*] Corresponding author at: Department of Mechanical Engineering, Federal University of Pará, Brazil.
E-mail address: pcmachado@usp.br (P.C. Machado).

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behavior in compression tests is different for each load direction of Hadfield single crystal, as reported by Karaman et al. [5]. The authors [5] showed that the [111] direction in compression tests presents a higher yield stress and a lower toughness than the [001] direction.

The single abrasive process can be simulated by a scratch test that aims to clarify the mechanisms of deformation and material removal, as well as to evaluate or to rank the abrasion resistance of the studied materials [6]. These scratch test results are also dependent on the tip geometry [6]. Hokkirigawa et al. [7,8] found a correlation between the critical angle of attack (α_c) and the degree of penetration (D_p) for a Rockwell tip; both parameters affected the change in the wear micromechanism. Additionally, the same authors also observed a dependency of these parameters (α_c and D_p) on the hardness (in the case of a diamond indenter), or the shear resistance of the interface (in the case of a steel indenter). Challen et al. [9] showed that α_c decreased with the increase of the hardness of metallic materials.

Moore [10] evaluated the effect of frictional heating during abrasive wear and concluded that frictional heating promoted the transformation of physical, chemical and mechanical properties. Although the overall effects on the abrasive wear process could not have been observed. Focused ion beam microscopy allowed the observation of a thin layer formed by the abrasion process. This layer was composed by ultrafine-grains near the body and counter-body interface [11,12].

The present study evaluates the micro-scratch wear of Hadfield steel, undeformed and in-service hardened. Single-scratch tests were performed at the micro-scale in order to evaluate the hardening gradient. Additionally the effect of the crystallographic orientation, (001) and (111) planes, on the friction coefficient, the wear micromechanism formation, and the scratch sub-microstructure were investigated.

2. Experimental procedure

2.1. Materials and test conditions

A ASTM A128 grade C Hadfield steel (12.4Mn–1.2C–1.5Cr (% wt), [13]) was used in this study. The in-service hardening sample was taken from the surface of a used jaw crusher plate (Fig. 1a) and the undeformed sample was obtained from the core of the same plate. The mean austenitic grain size of the steel was 135 ± 13 μm.

The micro-scratch tests (classification: type 1 - single pass [6]) used a Hysitron Nano-Indenter with a 60° diamond cone stylus and a 5 μm radius tip. The scratch parameters were length: 100 μm and velocity: 3.3 $\mu m.s^{-1}$. That the radius tip geometry did not change after the scratch tests, was verified, performing a SEM (Scanning Electron Microscopy) analysis. The scratch test was done on the cross-section of the deformed layer (Fig. 1b) in a direction parallel to the worn surface. The scratch positions (p) along the cross-section hardening surface started at 100 μm bellow the worn surface, with a gap between the scratches of 500 μm (Fig. 1c). The friction coefficient (COF) and the depth (h) of the scratches were evaluated. The in-service hardened samples were tested in each of the wear regimes (mild, transitional and severe) under two series of tests with different normal load (W) ranges:

i. 20 mN, 50 mN, 100 mN, 150 mN, 200 mN, and 250 mN;
ii. 50 mN, 100 mN, and 200 mN.

In addition, a series of scratch tests using a normal load of 200 mN were carried out along the (001) and (111) planes of the undeformed sample.

2.2. Sample characterization

The microscopy samples for SEM (Scanning Electron Microscopy), FEG (Field Emission Gun), FIB (Focused Ion Beam) and EBSD (Electron Backscatter Diffraction) and the samples for the scratching tests were polished for 30 minutes, at 40 RPM using 50 nm colloidal silica. The arithmetic mean height (S_a) of the surface roughness obtained from the Taylor Hobson CCI 3D optical interferometer after the manual polishing resulted to be 0.007 ± 0.001 μm. In order to perform the microstructural characterization, the specimens were etched with Picral 4%. The cross-section hardness profile was obtained using a Vickers microhardness tester with a load of 0.3 kgf.

The EBSD technique was used to identify the mean crystallographic planes of the microstructure of the Hadfield steel in order to perform the scratch analysis. A FEI-Inspect 50 FEG with a EDAX camera was employed and the analysis was carried out with a step size of 0.5 μm.

A FEI-Quanta 3D FEG/FIB was used to investigate the sub-microstructures of the scratches along the (001) and (111) planes. The FIB technique was used to mill the sample and to scratch the cross-section surface at 60 μm length. The milling process was operated at 30 kV and 30 nA to open a cavity of 30 μm x 20 μm. The finishing proceeded in two steps: 30 kV and 5 nA, and 30 kV and 3 nA.

2.3. Scratch characterization

The Hysitron Nano-Indenter was used to obtain the normal indenter displacement h (depth of scratch) and the friction coefficient. The mean depth and friction coefficient were extracted between 20 μm and 100 μm length.

The f_{ab} fraction[1] was acquired from the FIB scratch cross-section and measured by the Image J Software. This analysis was applied in the scratch analysis of the crystallography orientation.

Fig. 2 shows the cone indenter profile with the angle of attack model for the used indenter. The angle of attack of the indenter with a 5 μm spherical tip varied corresponding to the indenter penetration depth. For instance, for a 0.5 μm depth the angle of attack was 26°, whereas for a depth of 2 μm the angle was 54°. The radius designed the indenter relative to the surface a and the angle of attack α are obtained by the following regression equations: $a = 2.9505{*}h$^0.4405; and $\alpha = 37.392{*}h$^0.5208. The cone angle of the indenter was 60°, representing the system's maximum angle of attack. This geometrical configuration resulted in a geometric transition depth (Z_{gt})[2] of 2.5 μm, i.e. a depth at which the rounded tip merged into the conical portion of the indenter [14]. The degree of penetration (D_p)[3] was calculated following the research of Hokkirigawa and Kato [8] and using the results of the angle of attack model.

3. Results and discussion

3.1. Microstructure

The microstructure shown in Fig. 3a presents a continuous network of carbides re-precipitated at grain boundaries of

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[1] $f_{ab} = (1 - A_{hole}/A_{pile\text{-}up})$, where $A_{pile\text{-}up}$ is the sum of the areas above the baseline (pile-up) and A_{hole} is the area below the baseline, both observed on the scratch cross-section.

[2] $Z_{gt} = r.(1 - cos.\theta_{deg})$, where r is the tip radius, $\theta_{deg} = ½.(180 - \alpha_{deg})$, and α_{deg} is the tip apex angle (degrees).

[3] $D_p = h/a$, where h = depth of penetration and a = radius designed the indenter relative to the surface.