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Wear 258 (2005) 32-39

WEAR

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# Wear testing of high Fe–N–C steels

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Received 22 December 2003; received in revised form 25 February 2004; accepted 21 April 2004 Available online 14 October 2004

#### Abstract

A series of iron alloys consisting of iron, nitrogen and carbon has been produced, characterized, and evaluated for mechanical and wear properties. The Fe–C–N alloys have no carbide or nitride precipitates and have a duplex phase fcc–bct microstructure. The high 0.9 wt.% carbon + (0.4–0.9 wt.%) nitrogen interstitial concentrations produced significantly higher strength, higher hardness and much improved abrasive wear (scratch, pin-on-drum, pin-on-disk) resistance over that of the starting bcc phase iron alloy with 0.9 wt.% carbon and carbide precipitates. Results from compression, indent hardness, and scratch wear tests were analyzed using similar data reduction. This new data analysis technique resulted in an improved and coherent understanding of abrasive scratch wear process, which was directly related to the alloy's mechanical properties.

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Keywords: Nitrogen; Carbon; Steel; Abrasion; Pin-on-drum; Pin-on-disk

# 1. Introduction

Wear analysis is presented for a new series of iron-based alloys in which the additions of nitrogen to a Fe–C alloy: (i) changed the matrix phase composition and (ii) resulted in the loss of the carbide precipitates in favor of interstitials, both effects greatly enhance the material wear resistance. In addition to describing the microstructure and properties of these Fe–C–N alloys, a new approach to abrasive wear analysis is presented in which a close relationship between a material's mechanical strength, hardness, and wear is established.

A new series of high nitrogen and carbon steels have recently been developed [1,2]. These materials have interstitial concentrations that can exceed 1.8 wt.%, approximately 7 at.%. Optical, scanning electron, and transmission electron microscopy studies showed the Fe–C–N alloys are a duplex fcc–bct phase alloys without either nitride or carbide precipitates [1–3]. Mossbauer analysis has shown: (i) these Fe–C–N alloys to be precipitate free, (ii) the nitrogen and carbon atoms are highly dispersed interstitials, (iii) the interstitials are limited to a single interstitial nearest neighbor site, i.e., interstitial atoms have no next nearest neighbor interstitials and do not form multi-atom interstitial complexes [4,5]. (Interstitial atoms are atoms situated in the open spaces between atoms in a lattice structure.) Interstitial atoms distort the local matrix structure making dislocation movement through the lattice difficult, thereby strengthening the alloy. Theoretical understanding for the high nitrogen and carbon solubility has recently been developed from determination of the electronic structure of interstitials in iron [5]. High interstitial concentration: (i) strains the lattice [6,7], (ii) increases mechanical strengths [8], and (iii) increases hardness while retaining ductility [1-3]. Wear, especially abrasive wear resistance, has been found to be highly correlated with a material's higher hardness [9]. Thus, these Fe-C-N alloys would be expected to also display excellent wear resistant characteristics.

This investigation describes the manufacturing experimental technique used to produce Fe–0.9C–(0.0–0.9)N alloys, their resulting microstructure, mechanical properties, hardness, and the results from several abrasive wear tests. The Fe–C–N alloys are duplex fcc–bct phase alloys and have high strength ( $\sigma_{\text{YIELD}} > 1400$  MPa), high hardness (HRC > 50),

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and excellent ductility ( $\epsilon_{FAILURE} > 20\%$ ). All the mechanical properties of these new alloys predict these alloys should provide excellent wear characteristics. The starting or base material was an O-1 tool steel, Fe–0.9C, an alloy often chosen for its excellent wear resistance. The addition of nitrogen to these alloys increased the alloy's strength and wear resistance by a minimum factor of two. Wear data were highly correlated to compression and indent data. Using a modified Meyer's hardness equation, a direct correlation between the material's mechanical properties (strength and hardness) and its abrasive scratch wear resistance is established.

## 2. Experimentation

Nitrogen addition was accomplished by high-isostaticpressure (HIP) melting a high carbon commercial steel (heat-treated and oil quenched O-1 tool steel: 0.9 wt.% carbon) cylinder approximately, 8 cm diameter by 12-15 cm in length. Each cylinder, weighting approximately 5 kg, was placed in a crucible with a hole in the bottom. This crucible was placed over a second crucible and both crucibles were placed in a HIP furnace. The samples were melted in a nitrogen atmosphere under 50, 100, or 200 MPa. As the O-1 steel melted and dripped into the bottom crucible, nitrogen diffused into the molten iron resulting in a uniform nitrogen concentrations of 0.4, 0.6, or 0.9 wt.%. To assure the nitrogen remains in solution during solidification, just prior to reducing temperature, the nitrogen pressure was increased by 10 MPa. After cooling and extraction, the alloys were evaluated in the as-cast condition. Phase composition and concentration analysis was determined by (i) X-ray diffraction: line structure and intensity comparisons, (ii) chromatic etch: color and area ratios, (iii) SEM and TEM, for microstructure determination, and (iv) Mossbauer: line structure and area ratios.

Samples were sectioned for chemical analysis, polished for microstructure characterization, and machined for compression, hardness, and wear evaluation. Mechanical properties were determined from compression testing cylindrical samples 10 mm diameter by 20 mm in length, ASTM E9. True stress–true strain calculations were determined from the applied load, load head travel, and sample diameter expansion.

Wear tests were conducted to ASTM standards and included: (i) abrasive single and cyclic scratch: ASTM G171, (ii) pin-on-disk: ASTM G99-95a, (iii) pin-on-drum: ASTM G132-95, and (iv) jet erosion tests were conducted to ASTM G-76.

Abrasive single scratch tests were conducted by tracing a diamond stylus with 20, 50, 100, or 200  $\mu$ m radii spherical tip across the horizontal sample surface, with a 0.26, 0.52, 1.1, 2.1, 4.3, or 8.5 N vertical or normal gravity loads, at a travel velocity of 0.3 mm/s, over a travel distance of 10–12 mm. Optical microscopy was used to measure scratch width. Stylus profilometry was used to evaluate scratch wear for material

loss, i.e., the amount of material deformed by the indenter minus the amount of material pushed up to form the scratch shoulders. Wear was characterized by measuring the scratch width.

Abrasive cyclic scratch tests were conducted on all alloys using a single indenter radius and load combination, 50  $\mu$ m and 4.2 N. The same scratch track was reciprocally cycled over 1, 2, 3, 5, 8, 12, 17, and 23 times. Four additional cyclic tests series were conducted on the O-1 tool steel: (i) 20  $\mu$ m–1.1 N, (ii) 50  $\mu$ m–2.1 N, (iii) 100  $\mu$ m–4.27 N, and (iv) 200  $\mu$ m–8.53 N. The same scratch track was again cycled 1, 2, 3, 5, 8, 12, 17, and 23 times. Wear was characterized by measuring the scratch width.

Pin-on-disk tests were conducted using 6.35 mm diameter metal sample pins and (i) three different abrasive grinding wheels as disks (100 grit SiC, 240 grit Al<sub>2</sub>O<sub>3</sub>, and 400 grit Al<sub>2</sub>O<sub>3</sub>) and (ii) D-2 tool steel disk (hardness: HRC 60). Loads were 0.7, 1.3, 3.5, and 7.0 N. Disk speed was approximately 0.3 m/s (100 rpm). Track radius was 30 mm. Total travel distance was 1500 m. Wear was characterized by pin weight loss per meter traveled:  $\mu$ g/m.

Pin-on-drum tests were conducted using 6.35 mm diameter metal sample pins and 150 grit SiC abrasive paper on the drum. Travel distance was 8 m. Applied load was 66.7 N. Speed was 0.045 m/s. Tests were run with and without pin rotation (10 rpm). Wear was characterized by the volume of material loss per load-meter traveled: mm<sup>3</sup>/Nm.

Jet erosion tests were conducted using 50  $\mu$ m diameter Al<sub>2</sub>O<sub>3</sub> powder at a particle delivery rate of 2.0 g/min. Particles velocity was 40 m/s. Erosion particles were entrained in an argon gas stream. Two different impact angles, 90° and 30°, were used. Total test time was 20 min. Wear was characterized as weight loss per minute tests:  $\mu$ g/min.

## 3. Scratch analysis

Strength and hardness of steel alloys are highly correlated with interstitial concentration. Hardness measurements are most commonly determined by the compression load indent tests, i.e., Rockwell, Vickers, etc. Tabor developed a relationship [10] between (i) Hollomon's tensile/compression hardening exponent determined from true stress–true strain tests ([11]; Eq. (1a)):

$$\sigma_{\text{TRUE}} = k \epsilon_{\text{TRUE}}^n, \tag{1}$$

where  $\sigma_{\text{TRUE}}$  is the true stress,  $\epsilon_{\text{TRUE}}$  the true strain, *n* the compression strain hardening exponent, and *k* the material constant, and (ii) Meyer's indent hardness exponent determined from hardness test using different indenter loads and radii ([12]; Eq. (2a)):

$$\left(\frac{L}{D^2}\right) = A\left(\frac{w}{D}\right)^N,\tag{2a}$$

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