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# Increasing the coefficient of sliding friction of NiCr at low loads by interstitial surface hardening

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#### ARTICLE INFO

## ABSTRACT

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#### 1. Introduction

NiCr alloys are of technical interest for vibrational damping structures operating at high-temperature (  $> 1000 \circ C$ ) [1]. For this purpose, woven wire meshes have been developed that consist of NiCr alloy (Ni-20Cr) wires. The inter-wire forces in these structures are estimated to be about 0.05 N [2]. Increasing the coefficient of sliding friction (COF) between the wires at these loads would improve the damping behavior.

It has been reported that hardening metal alloys without changing the composition decreases COF. For example, implanting martensitic steel with carbon or nitrogen increases hardness by about two times and reduces the COF (in air) by a factor of 8 [3]. Work hardening of an aluminum alloy doubles the hardness and decreases the COF from 1.4 to 0.9 [4]. Annealing a CuZrAl bulk metallic glass raises the hardness from 480 HV to 530 HV and reduces the COF from 0.5 to 0.4 [5].

Interstitial hardening of stainless steel by low-temperature gas-phase carburizing, in contrast, has previously been found to increase the COF [6]. In this technique, carbide formation is inhibited kinetically, since the low processing temperatures effectively immobilize the substitutional elements, and a "colossal" supersaturation – over 10<sup>4</sup> times the equilibrium solubility at the treatment temperatures - of interstitially dissolved carbon is achieved [7]. In particular, low-temperature carburizing of 316L stainless steel, which increases the surface hardness from 2.0 GPa

http://dx.doi.org/10.1016/j.wear.2015.10.009 0043-1648/© 2015 Elsevier B.V. All rights reserved. to 7.6 GPa, increases the COF by 37% (from 0.51 to 0.70), as measured by a rotating ball-on-disk test at room temperature using 9.5 mm diameter balls and a 5 N load [6]. In addition, the wear rate decreases by over 98%.

Low-temperature interstitial hardening of NiCr alloys by gas-phase carburizing and nitriding increases

the coefficient of sliding friction (COF) at low loads by as much as 50%. While the substantial surface

hardening provided by the very high concentrations of interstitial carbon or nitrogen tends to decrease

the COF, the presence of these interstitial solutes also decreases the thickness of the air-formed surface

oxide layer, which tends to increase the COF. The overall result is an increase in COF.

A potential explanation for this unexpected result arises from the fact that on smooth samples, interstitial hardening can increase the surface roughness. The large stresses that develop due to the high concentration of interstitials cause severe near-surface plastic deformation that results in uneven uplift of the surface. The influence of surface roughness on friction has been the subject of extensive investigation [8-13], and its effect is complicated by many factors, including ductility, shear strength, and ambient conditions [14].

Further, low-temperature carburizing and nitriding can increase COF by reducing the thickness of the native, air-formed oxide layer on 316 L stainless steel [15,16]. Low-temperature carburizing, in particular, reduces the oxide layer thickness from  $\approx$  1.5 nm to  $\approx$  1.0 nm. It was postulated that the strong Cr–C bonds make the surface less reactive and less prone to form  $Cr_2O_3$  [15].

Increased surface oxide thickness decreases the COF of metals. For example, the COF at room temperature of a freshly cut Cu surface decreases substantially after forming a 50 nm thick surface oxide layer [17]. The COF of a Fe-19Cr alloy at room temperature decreases with increasing oxygen partial pressure in the testing ambient [18]. At elevated temperatures (800–1050 °C), the COF of 1018 mild steel decreases as the oxide thickness increases from 0.015 mm to 1.59 mm [19]. The COF of 304 L stainless steel at room temperature decreases after oxygen ion implantation into the near-surface [20]. Therefore, a treatment that leads to a reduced surface oxide thickness should increase the COF.





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In this work, we have investigated whether interstitial hardening, by low-temperature gas-phase carburizing or nitriding, alters the COF of NiCr at 0.05 N load, and whether the COF correlates with a change in the thickness of the air-formed surface oxide layer. Since this is a gas-phase process, the intricate geometry of a woven wire damping structure would not hinder complete conformal treatment of all surfaces. At very high temperatures, the interstitial atoms would react to from carbides and nitrides. Therefore, long-term operating temperatures of treated structures would be limited to less than  $\approx 500$  °C.

#### 2. Materials and methods

COF measurements were performed using ball-on-plate dry sliding tests in a Bruker/CETR UMT tribometer. The laboratory humidity was not monitored. The plates were NiCr (Ni–20Cr) and the 1.6 mm diameter balls were Hastelloy C276 (Ni–23Cr–14Mo–7Fe). The plates were purchased from Goodfellow, and polished with SiC grit paper through 1200 grit (P4000), which produces an arithmetic average roughness  $R_a$  of  $\approx 20$  nm [21]. The balls were purchased from Bal-tec, and were Grade 200, indicating a maximum  $R_a$  of 200 nm. Balls made of NiCr were desired, but could not be obtained in the necessary size, and therefore Hastelloy C276 was chosen as an alternate material due to its compositional similarity. Plates and balls were carburized and nitrided in a flowing-gas atmospheric pressure furnace built by CVD Equipment Corporation. Both treatment recipes included initial activation in HCl gas for 4 h at 325 °C [22]. The carburizing treatment consisted

of 20 h at 450 °C in 30%CO/45%H<sub>2</sub>/25%N<sub>2</sub>. The nitriding treatment consisted of 20 h at 440 °C in 10%NH<sub>3</sub>/10%CO/40%H<sub>2</sub>/40%N<sub>2</sub>. The nitriding recipe contains CO gas, which leads to carbon interstitials, but previous investigations with 316 L stainless steel [23,24] revealed that the carbon interstitials are located deeper into the sample, and only nitrogen interstitials are located close to the surface.

The furnace treatments could alter the surface roughness of the plates, either from plastic deformation caused by the carbon and nitrogen interstitials, or from etching by the HCl gas. Therefore, the plates were lightly re-polished with 1200 grit SiC paper after treatment.

COF measurements were performed at 0.02, 0.05, and 0.1 N loads. Assuming elastic Hertzian deformation, and using reported values of 210 GPa and 0.3 for Young's modulus and Poisson's ratio, respectively, for both ball and plate materials [25,26], the average contact pressures during testing were 180 MPa, 250 MPa, and 310 MPa for testing with 0.02 N, 0.05 N, and 0.1 N loads, respectively.

Microstructural and microchemical analyses were carried out in the Swagelok Center for Surface Analysis of Materials at Case Western Reserve University. The treated samples were investigated with X-ray diffractometry (XRD) using a Scintag X-1 diffractometer, and the concentration of interstitials and thicknesses of the surface oxide layers were measured with X-ray photoelectron spectroscopy (XPS, also called ESCA – electron spectroscopy for chemical analysis) using a Physical Electronics Versaprobe. Hardnesses were measured using a Buehler Micromet Vickers indenter, with a 0.25 N (25 gf) load.



Fig. 1. COF results for (a) unidirectional sliding of nontreated NiCr at varying velocities, and (b) reciprocating sliding of nontreated, carburized, and nitrided NiCr at varying loads. The error bars represent one standard deviation. (c) COF as a function of testing time, averaged over 5 s intervals for the reciprocating sliding tests performed at 0.1 N.

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