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Brittle to ductile transition of tribomaterial in relation to wear response at high temperatures

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A B S T R A C T

The third body formed in a contact between HS25 cobalt-based superalloy versus ceramic under fretting wear (small reciprocating displacements) was investigated. This tribomaterial, commonly called “glaze layer”, was created from nanosized, compacted and sintered wear debris and adheres on both rubbed parts. The glaze layer was investigated both from tribological and rheological points of view. In terms of tribology, the glaze layer was found stable above 450 °C, providing low friction and very low wear rate in the interface. To study the mechanical properties of the third body, micropillars have been FIB-machined within the glaze layer and compressed between 25 °C and 500 °C. Low temperature testing showed a brittle and hard behavior for the glaze layer which was confirmed by nanoindentation. By contrast, glaze layer at 500 °C evidenced a perfect ductile response with high strain rate during fretting and no brittleness. Made of 10–20 nm nanocrystals embedded in a ceramic-metallic amorphous matrix, the glaze layer is put forward in the brittle to ductile transition: the amorphous matrix may act as a metallic glass. This allows the correlation between glaze layer ability to plastically accommodate the strain without fracturing processes, thus without being damaged through debris generation. The ejected debris flow is stopped and the wear rate becomes negligible.

1. Introduction

The relative motion of two parts put into contact can lead to wear. When this surface damage affects assemblies at sufficiently high temperatures, the wear debris generated may form a tribomaterial called “glaze layer” in the interface. The “glaze layer” denomination has been introduced by Stott [1] to describe a tribofilm from compacted, sintered worn debris that adhere on both sliding parts [2]. Glaze layer formation has been studied for various metallic materials configurations like 0.45% C steel [3], high strength steel [4], stainless steels [5], nickel- [6] and cobalt-based alloys [7,8]. Although the debris generation is unintended, the resulting glaze layer provides both low friction and very low wear rate [8], giving this type of tribofilm favorable properties for tribological applications.

To reach higher temperature thus enhanced efficiency in hot mechanical systems, ceramic materials are being developed to replace metallic parts [9]. Hence, wear protection issues are being shifted to metallic-ceramic interfaces where studies evidenced good tribological properties and similar glaze layer morphology to metallic contacts [10,11]. The glaze layer was there described as nanocrystalline with amorphous matrix and composed of oxidized as well as non oxidized debris from both parts. While the glaze layer protection modalities are sometimes attributed to specific oxides formed [12], its friction properties are more likely to be mainly due to the mechanical behavior of the compacted debris [13,14].

However, the physical justification between mechanical and tribological properties of the glaze layer is missing. The present study is precisely aimed at characterizing the glaze layer mechanical behavior in its stability domain (above 500 °C). In order to do so, a glaze layer is created in its stability domain in a cobalt-based alloy versus silicate ceramic contact under fretting wear (small reciprocating displacements). Then, its mechanical properties are investigated through nanoindentation and micropillar testing. Indeed, introduced by Uchic [15], micropillar compression provides constitutive laws that are interpreted here in terms of elasto-plastic behavior of the glaze layer between 25 °C and 500 °C. Micropillar testing has already been used to characterize

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Nomenclature

a	contact diameter (mm)
A_c	projected contact area of nanoindentation imprint (mm^2)
d	grain size (nm)
δ	fretting displacement (µm)
δ^*	fretting displacement amplitude (µm)
δ_g^*	fretting sliding amplitude (µm)
ε	compression strain (-)
f	fretting frequency (Hz)
F	nanoindentation load (N)
F_{max}	nanoindentation maximal load (N)
H	hardness (GPa)
k	specific wear rate ($mm^3\ N^{-1}\ m^{-1}$)
μ	friction coefficient (-)
N_c	number of fretting cycles
P	fretting normal force (N)
Q	fretting tangential force (N)
Q^*	fretting tangential force amplitude (N)
S	total fretting sliding distance (m)
σ	compression stress (GPa)
σ_{max}	maximal compression stress (GPa)
σ_Y	yield strength (GPa)
$\Delta\sigma$	brittleness criterion (GPa)
$\Delta\sigma_{norm}$	normalized brittleness criterion (-)
t_j	amorphous matrix thickness (nm)
$T°$	temperature (°C)
$T°_{GL}$	temperature threshold of glaze layer stability (°C)
T_g	glass transition temperature (°C)
V_-	volume loss (mm^3)
$V_{-,flat}$	volume loss for the flat (mm^3)
$V_{-,punch}$	volume loss for the punch (mm^3)
V_+	transferred volume (mm^3)
$V_{+,flat}$	transferred volume on the flat (mm^3)
$V_{+,punch}$	transferred volume on the punch (mm^3)
$V_{m,tot}$	total missing volume (mm^3)

interfacial materials like Tribologically Transformed Structures (TTS) [16,17] but never glaze layers, which are compacted interfacial debris. In this paper, temperature-dependent mechanical properties of a glaze layer are investigated for the first time through the compression of micro-pillars that are milled by FIB (Focused Ion Beam) in a glaze layer pre-created at 700 °C. This micromechanical test [15] offers several advantages compared to nanoindentation experiments. First, the measurement of the local stress-strain curve at the microscale is straightforward [17] whereas classical nanoindentation experiments cannot provide more than two points, and require specific care to estimate the contact area [18]. Second, brittleness/ductility can be investigated up to large strain [19]. The main drawback lies in the additional effort for sample preparation.

The present study outlines first the glaze layer stability temperature range according to tribological criteria of friction and specific wear rate. The mechanical properties of the glaze layer are investigated in terms of hardness and yield strength, and related to the micro and nanostructure. The possible deformation modes for nanomaterials are discussed by taking into account the probable leading role of the amorphous matrix accommodating plastic strain. Finally, a brittleness stress criterion is introduced as the crucial factor to correlate high temperature plastic strain accommodation and tribological anti-wear properties of the third body "glaze layer".

2. Experiments

2.1. Tribological test

The glaze layer was created in a $a = 4$ mm diameter plane to plane contact between a metallic punch and a ceramic flat (Fig. 1a). The punch was processed in commercial Haynes n°25 (HS25®) alloy, known for good strength and hot corrosion resistance at high temperatures. The composition of HS25 is summarized in Table 1 [20]. The flat is a stoichiometric compound of silica (SiO_2) and metallic oxide, in the category of ionic ceramics.

The flat was clamped whereas the punch was connected to an electrical shaker providing horizontal vibrating motions. The fretting test allows the application of small amplitude alternate slides of amplitude $\delta_g^* = 50$ µm, frequency $f = 50$ Hz, normal force $P = 800$ N, number of fretting cycles $N_C = 1$ million and temperatures T° tuned and kept constant between 25 °C and 700 °C. The resulting measures of tangential force Q and total displacement δ generated the fretting cycle (Fig. 1b), thus the friction coefficient µ defined as:

$$\mu = \frac{Q^*}{P} \tag{1}$$

where Q* is the tangential force amplitude (Fig. 1b).

After the tribological test, the samples were ultrasonically cleaned in ethanol to be scanned using a laser interferometer for the determination of 3D surface profiles and the computation of wear volumes. V_- and V_+ are respectively defined as the material volume below and the

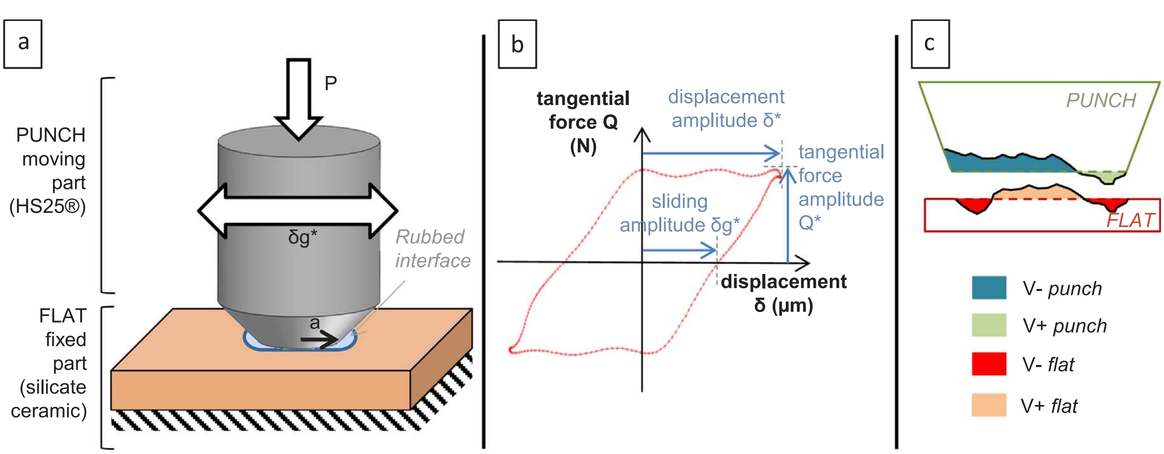

Fig. 1. (a) Fretting contact materials and geometry; (b) corresponding fretting cycle; (c) wear missing volume concept.