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Tribologically enhanced self-healing of niobium oxide surfaces

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ABSTRACT

Activating a self-healing process is a viable approach for preventing the failure of ceramics experiencing mechanically-induced crack propagation. Previously, it was demonstrated that niobium oxide (Nb_2O_5) exhibits self-healing properties activated by the formation of Nb-Ag-O ternary oxide when heated above 945 °C in presence of silver. In this study, we explore the mechanism of lowering the high-temperature healing requirement by assisting the process of crack repair with a normal load and shear stresses. Specifically, we propose to use tribologically-induced local heating as a mechanism to enhance the self-healing ability of Nb_2O_5. During a pin-on-disk test, whereby a niobium oxide flat was sliding against a silver-coated ball, a sudden lowering of the coefficient of friction was observed at elevated temperatures (~600 °C). The better performance of the coating was associated with a surface reconstruction process initiated inside the wear track. Extensive characterization analysis of the wear track using energy-dispersive x-ray spectroscopy, Raman spectroscopy, and x-ray diffraction confirmed the presence of an Nb-Ag-O ternary oxide phase inside the wear track formed at elevated temperature. The formation of an Nb-Ag-O ternary oxide at a much lower than thermodynamically-required temperature suggests that the self-healing process can be initiated directly during mechanically induced stresses. Such a process is a new recipe for improving wear and crack resistance characteristics of ceramic components and may be tuned to provide the desired frictional response.

1. Introduction

The quickly emerging need to finish surfaces of additive manufactured parts in addition to parts produced using more traditional techniques requires the use of materials with adaptive surfaces that automatically adjust their properties in response to external stimuli via interaction with both the ambient environment and contact with the parts [1]. The new design requires a surface that satisfies a specific protective functionality that will enable the main part to last longer. Most protective surface finishes require the use of ceramic coatings in pure or composite form that may be produced using a variety of techniques that include but are not limited to physical vapor deposition, chemical vapor deposition, laser cladding, plasma spray, and cold spray [2]. Functional ceramics are prone to cracking as a result of external mechanical stimuli, which are further exacerbated by thermal stimuli [3]. Once cracks have formed within a ceramic, the integrity of the protective surface is significantly compromised. A potential solution would be to resort to the self-healing/surface reconstruction concept in the design of next-generation protective surfaces, which would significantly increase the lifetime and reliability of materials and would drastically reduce replacement costs [4].

Surface reconstruction (self-organization) during sliding contact has received less attention so far but has the potential to create self-healing and self-lubricating materials that are crucial for environmentally-friendly tribological applications [5,6]. Friction and wear are usually viewed as irreversible processes that lead to energy dissipation (friction) and material deterioration (wear) [7,8]. These adverse effects can be mitigated using solid lubricants that are able to self-organize on sliding surfaces to minimize friction and/or wear [6,9]. The formation of an optimum solid lubricant at the interface between sliding surfaces is crucial to the efficiency and lifetime of applications, especially when operating in harsh environments [10,11]. For example, ternary oxides that contain a noble metal were recently shown to shear easily at elevated temperatures and to exhibit extremely low friction coefficients (< 0.2) when tested at temperatures that exceed $T > 500$ °C [12–14]. These oxides were also successfully incorporated into the design of an adaptive coating, whereby multiple lubricious phases and a hard phase are combined to form a composite material that reduces both friction and wear over a broad temperature range [15–18]. The most promising high-temperature solid lubricant that has been reported in the literature is silver tantalate ($AgTaO_3$) [19,20]. In these studies, the measured coefficient of friction (COF) was found to vary with load and values as

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low as 0.04 were reported when tested at 750 °C using a 1 N load.

The focus of the current research is a subset of mating binary oxide surfaces that have the potential to self-heal and self-lubricate through the controlled precipitation of a ternary oxide phase at elevated temperatures. Studying fundamental self-healing and self-lubricating processes, in addition to the deformation mechanisms of oxide-based metastable mating materials, is lacking in the literature [3]. Lessons learned from prior work on adaptation and reconstruction mechanisms point to the importance of using an embedded phase that is able to flow to the damaged area or to the surface in order to facilitate the healing or self-lubricating process, respectively. In addition, it is very crucial to control the rate of material migration as well as the rate of new phase formation. Stone et al. [21] investigated the reconstruction mechanisms in Ta_2O_5/Ag nanocomposite coatings with a silver content of 14 at.% and found that this material adapts as a result of the combination of elevated temperatures and sliding. The authors showed that the surface of the coating changed over time as a result of the migration of silver to the surface. Combining temperature and applied shear stress resulted in the creation of a soft and lubricious $AgTaO_3$ phase on the surface while the supporting underlayer consisted primarily of a hard Ta_2O_5 phase. The relative slow migration of silver to the surface to form a thin lubricious layer on a harder surface suggested that it is possible to tune the friction and wear performance of a nanocomposite film by controlling the amount of embedded silver for a given set of operating conditions. These results suggested that the redistribution of silver plays an important role in the friction characteristics of this material [12,21]. Two other strategies were reported in the literature to be effective at controlling the migration of a noble metal in a ceramic matrix: (i) the use of a transition metal nitride diffusion barrier [22] or (ii) control the ceramic matrix grain size both of which help reduce the rate of lubricant depletion in the adaptive coating layer and prolong the life of the coating during sliding wear at elevated temperatures [23].

The goal of this study is to understand the surface reconstruction mechanisms that result from thermal and mechanical stimuli during sliding at elevated temperatures. To achieve this goal, the first focus will be to create a binary oxide counterface (Ag_2O) that slides over another binary oxide surface (Nb_2O_5) that will to adapt by forming a ternary oxide healing material on the surface (e.g., $Nb_2O_5 + Ag \rightarrow AgNbO_3$). The surface adaptation is expected to be facilitated by the thermos-mechanical stimuli that will be controlled during the sliding process. The second focus will be to use magnetron sputtering to produce (Nb_2O_5/Ag_2O) multilayer structures on silicon substrates. Subsequent heating activates metal diffusion out of highly strained sites in the oxide, where metal grain nucleation and growth is thermodynamically driven by the reduction of system potential and surface energy. Hence, the noble metal rapidly distributes itself over the entire surface, as the coating bulk becomes depleted in lubricious metal phases. The noble metal then reacts with the oxide phase to create a lubricious ternary oxide phase. In both the bulk and thin film cases, the structural and chemical properties of these materials will be investigated before and after tribotesting (thermal and mechanical stimuli) and the results will be correlated to the tribological performance of these materials under different sliding conditions,

2. Experimental procedure

Bulk Nb_2O_5 cylinders were prepared by pressing Nb_2O_5 powder made of 5 μm average diameter particles (Sigma-Aldrich). The diameter of the cylinder before annealing was 12 mm. The Nb_2O_5 cylinders were sintered at 1300 °C for 3 h to minimize the open porosity and maximize the strength. The post-sintering diameter of the cylinder was 9.65 mm.

Three-layer coatings of $Nb_2O_5/Ag_2O/Nb_2O_5$ were deposited on Si wafers using an ATC 1500 reactive magnetron sputtering. Prior to insertion into the vacuum chamber, the substrates were cleaned ultrasonically in acetone followed by methanol for 15 min each, then rinsed by deionized water, and dried using compressed nitrogen. The system was evacuated to a base pressure of 10^{-4} Pa. Elemental targets of Nb (99.98% purity) and Ag (99.997% purity) were acquired from Plasma Materials. Coating deposition was carried out in a mixed environment of argon (99.999%) and oxygen (99.99%). The substrates, which had a floating bias, were heated to 300 °C while being rotated at 50 rpm to maximize coating uniformity. Sputtering was conducted at 350 W for 60 min for the Nb_2O_5 layer and at 25 W for 30 min for Ag_2O layer. The thickness of the oxide layers was about 500 nm for Nb_2O_5 and 200 nm for Ag_2O, as determined using a Micro Photonics Inc. optical profilometer.

A Bruker Nanoindenter was used to test the self-healing ability of the $Nb_2O_5/Ag_2O/Nb_2O_5$ three-layer system. The nanoindentation was performed using a 50 mN load to produce cracks on the surface. After nanoindentation tests, samples were heated up to 950 °C to trigger the self-healing process. Select areas around the indentation were cross-sectioned by Focused Ion beam (FIB) to test the hypothesis that cracks healed as a result of the thermal stimulus. Energy dispersive x-ray spectroscopy (EDS) and SEM focused ion beam (FIB) cross-section analysis were performed by FEI Nova 200 NanoLab FIB/SEM. The Raman spectroscopy was done by Nicolet Almega XR Dispersive Raman. Phase detection of the ternary oxide was performed using a Rigaku Ultima III XRD with Cu Kα radiation scanned from 20 to 90° with a step size of 0.05° and a scan rate 3°/min. The surface roughness of the coating was characterized using a Bruker Multimode atomic force microscope (AFM) in tapping mode. The scan rate for the AFM measurements was 0.5 Hz for a scan area of $5 * 5\ \mu m^2$.

The tribological properties of the bulk ceramic sample and the multilayer ceramic coating were tested using the Nanovea high-temperature macroscale pin-on-disk tribometer. The tests were performed at room temperature (~25 °C) and at elevated temperatures (~600 °C). In these tests, a flat niobium oxide samples were sliding against a silver coated (2.5 μm thickness) silicon nitride ball. The ball diameter was 6 mm. The tabulated tribology tests parameters are shown in Table 1. The normal load during the tests was kept at 1 N and a wear track of 4 mm radius was used for each experiment. Sliding speed during the tests was 60 rpm (2.5 cm/s). The maximum Hertzian contact pressure experienced by the surfaces in contact was ~0.6 GPa and reduced when the contact area changed due to the wear of the materials.

3. Results and discussion

Bulk Nb_2O_5 pellet samples were produced in an effort to test the hypothesis that ceramics are able to withstand shearing stresses by in-situ healing of a damaged area. Fig. 1(a) and (b) shows SEM micrographs of a Nb_2O_5 sample before and after sintering, respectively. As shown in Fig. 1b, sintering of the samples resulted in the expected grain growth and a substantial decrease in porosity in the binary oxide sample, which results in an enhancement in mechanical strength. The sintered samples were tested at different temperature regimes with and without the presence of Ag (Fig. 1d–f). At room temperature (Fig. 1d),

Table 1
Tribology parameters during test.

Substrate	Load	Temperature	Counter body (6 mm diameter)	RPM	Wear track radius
Nb_2O_5 pellets	1 N	25, 400, 600 °C	Si_3N_4 coated with 2.4 μm Ag	60	4 mm
$Nb_2O_5/Ag_2O/Nb_2O_5$ multi-layer coating	1 N	600 °C	Si_3N_4 coated with 2.4 μm Ag	60	4 mm

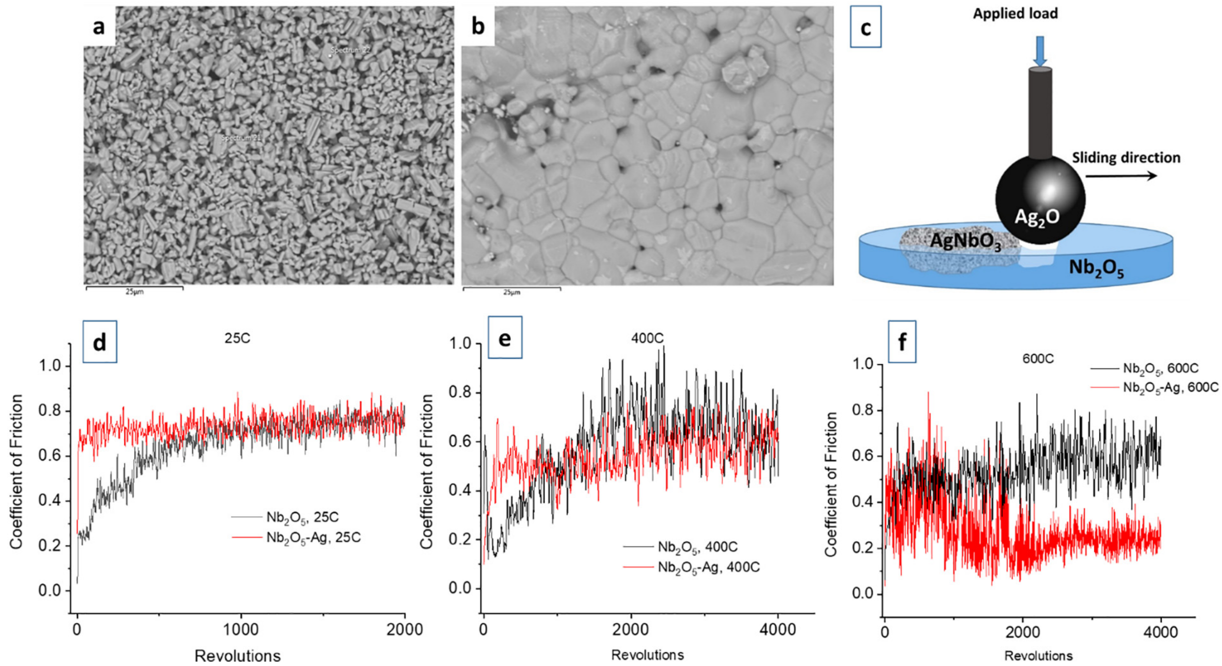


Fig. 1. Preparation of the bulk niobium oxide sample by (a) pressing and (b) sintering the pellet. The sample was further tested for the tribological performance (c). Tribology test of Nb_2O_5 with and without the presence of Ag at (d) 25C, (e) 400 °C and (f) 600 °C. Results indicate a reduction in the coefficient of friction in case of silver presence at 600 °C (f).

this test revealed a very high coefficient of friction (COF) of about 0.8 for both cases. Increasing the temperature of the tribological test up to 400 °C does not considerably change the frictional behavior (Fig. 1e). However, when the temperature of the tribotest was increased further to 600 °C, a sudden reduction in the COF value was observed (~0.24) for the Nb_2O_5 pellet tested in the presence of the Ag powder (Fig. 1f). The formation of Ag-Nb-O ternary oxide with perovskite crystal structure as a product of the reaction between Nb_2O_5 and Ag is responsible for the reduction in friction. Similar observations of the friction lowering due to formation of a ternary oxide and nitride phase were reported for the tribological contacts involving $AgTaO_3$ [24,25], TiN-Ag [26], and NbN-Ag [27,28] films. Therefore, the observed reduction in the COF is associated with the material reconstruction that takes place inside the wear track during the heat- and shear-induced self-healing process in tandem with the material softening that occurs at elevated temperatures [29].

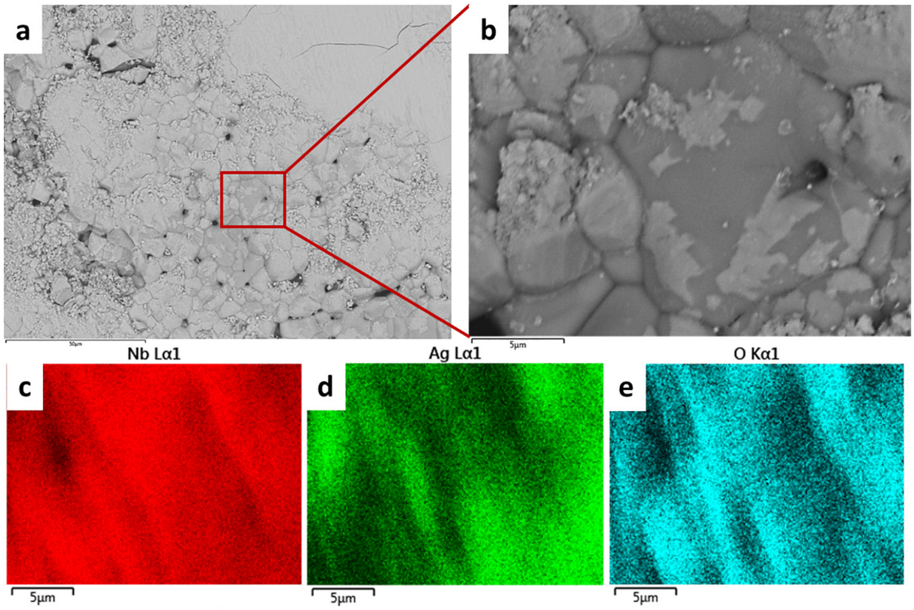
To study the origin of the tribological changes, we characterized the samples with EDS (Fig. 2). The distribution of Nb, Ag, and O inside the wear track shows the signs of reaction between bulk Nb_2O_5 and Ag powder which was placed on top of the sample prior to the tribology test. The formation of an Nb-Ag-O ternary oxide was previously shown to require temperatures in excess of 945 °C to proceed [4]. However, with the application of a mechanical stimulus, friction-induced heating can potentially assist the process of phase transformation at lower temperatures. As a result, silver can react with the Nb_2O_5 substrate at much lower temperatures (~600 °C as in the case of the current study). EDS analysis confirms that there is a significant degree of overlapping between Ag, Nb, and O maps, which were mostly along the grain boundaries of the samples (Fig. 2c–e).

Since the contrast in the EDS mapping of the wear-track is largely affected by the material underneath the deformation zone, clear identification of the self-healing reaction is challenging. Therefore, we further explored the self-healing process initiation by testing thin coatings. For this purpose, a layered structure of $Nb_2O_5/Ag_2O/Nb_2O_5$ was sputtered on a silicon substrate (Fig. 3a). Surface analysis of the sputtered samples indicated variations in the silver concentration, associated with the tendency of the material to agglomerate (Fig. 3b). AFM indicated that the surface topography of the layered coating consisted of a texture with a scale of about 10 nm (Fig. 3d). The elemental EDS analysis (Fig. 3c) shows 6.15 wt% of Ag which comes from deposited Ag_2O sublayer. Silver migrated to the surface as a result of the heating process to react with the Nb_2O_5 phase at the surface, creating a ternary oxide phase (self-healing/self-construction process).

To further understand the mechanisms involved in the observed self-healing process in the coatings (Fig. 4), Vicker's microhardness was used to create localized mechanical stress. As demonstrated in Fig. 4a, the indentation resulted in cracks being initiated at the corners of the sample. The sample was subsequently annealed at 950 °C for 12 h. The surface of the resulting sample shows no signs of cracks (Fig. 4b). We further analyze the FIB cross-sections of the sample at the locations of the cracks by SEM (Fig. 4c, d). Our results indicate an almost complete repair of the cracks during treatment. Coincidentally, the crack in the Si substrate was healed as well (Fig. 4c). EDS analysis demonstrates the dominant effect of Ag to heal the Si substrate, suggesting silver's ability to flow along the cracks.

The thermo-mechanical activation of the self-healing mechanism in the coating was explored when tested against a silver coated Si_3N_4 ball. Fig. 5a depicts the changes in the frictional behavior of the coating during the tribology tests at 600 °C. Similarly, to the bulk sample, at the beginning of the test the COF was high (~0.8). However, after 300 cycles, the COF values reduced by 30% down to 0.45. We further analyzed the morphology of the sample after tribological testing (Fig. 5b, c). Due to the high applied load, the coating is worn down completely to uncover the silicon substrate at different locations. EDS mapping analysis in Fig. 5c–e indicated that, in the wear track, the signals that correspond to Nb, Ag, and O overlapped. However, the measured COF values in the case of the bulk (~0.24) and thin film (~0.45) samples after sliding tests at 600 °C were different. The difference may be attributed to the larger amount of silver on the surface of the bulk sample that was able to create a larger area of the lubricious ternary oxide phase.

Analysis of the ball side of the tribopair was performed to further understand the nature of the formed wear debris. The surface of the Si_3N_4 ball was examined after the test vs. the Nb_2O_5 pellet at 600 °C.

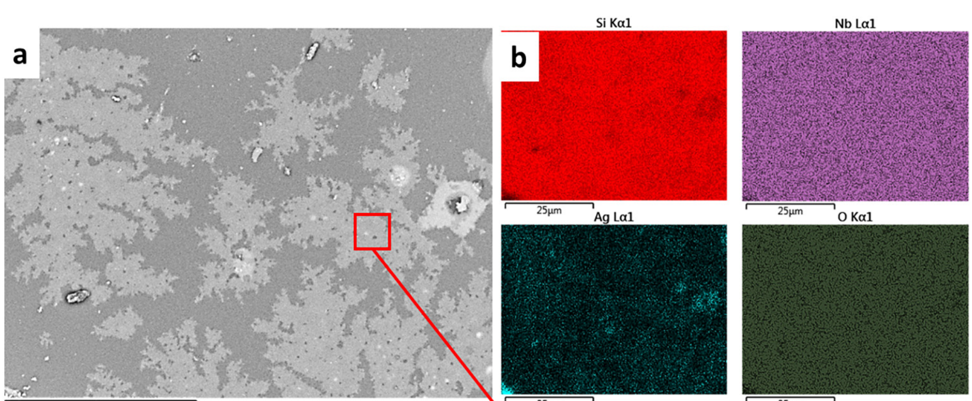


f

Element	Line Type	Weight %	Atomic %
O	K series	26.01	68.12
Nb	L series	50.31	22.68
Ag	L series	23.68	9.20
Total		100.00	100.00

Fig. 2. (a) Micrograph of wear track shows the entire wear track. (b) Higher Magnification of the wear track indicates preferred reaction point at grain boundaries. The EDS maps of (c) Nb, (d) Ag and (e) O illustrate the uniform distribution of contributed elements. (f) Corresponding concentration of each element.

The high wear resistance of Si_3N_4 resulted in minimal wear losses. However, the surface displayed the existence of a transfer film. EDS analysis of this transfer film, not shown here, indicated that the wear debris consisted of Nb, Ag and O, with the elemental maps for Nb and Ag being almost identically indicating the formation of a ternary Nb-Ag-O phase as a result of the tribotest. The formation of the ternary oxide phase is further explored with XRD and Raman spectroscopy analysis of the wear track formed after the pin-on-disk tests. Fig. 6a displays a comparative analysis of the changes in the XRD signature of the initial non-annealed niobium oxide coating and of the coating after the tribology test at 600 °C., XRD of the sample after the tribology test confirmed the presence of ternary oxides such as $AgNbO_3$ and



c

Element	Weight %	Atomic %
O	18.02	36.06
Si	45.27	51.59
Nb	30.56	10.53
Ag	6.15	1.82
Total	100.00	100.00

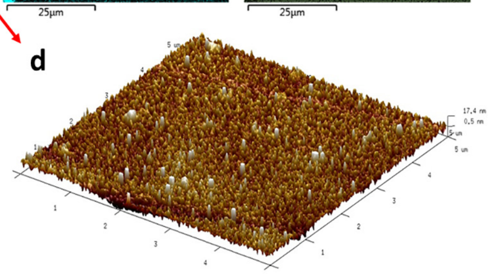


Fig. 3. (a) Surface morphology of the thin $Nb_2O_5/Ag_2O/Nb_2O_5$ layered sample with (b), (c) detailed EDS elemental analysis overview the surface composition of the sample. (d) AFM topography of as-deposited coating.

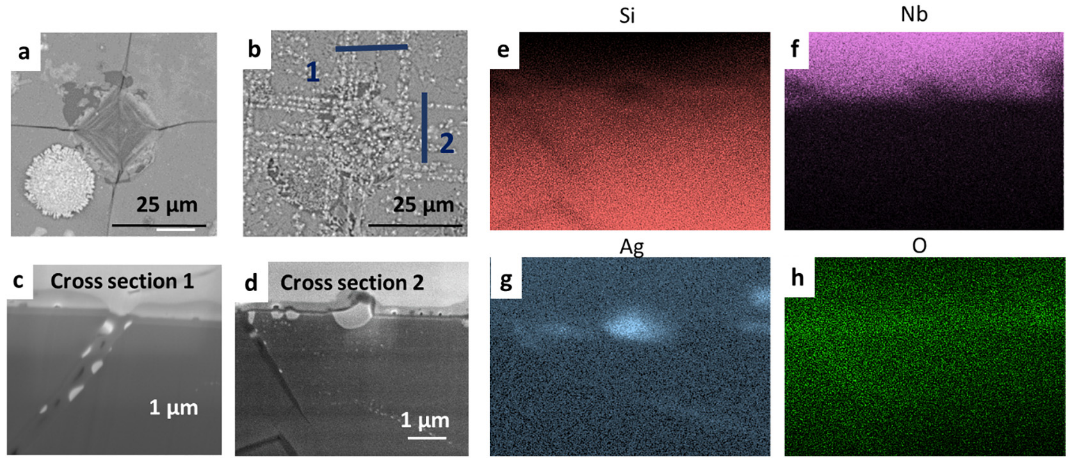
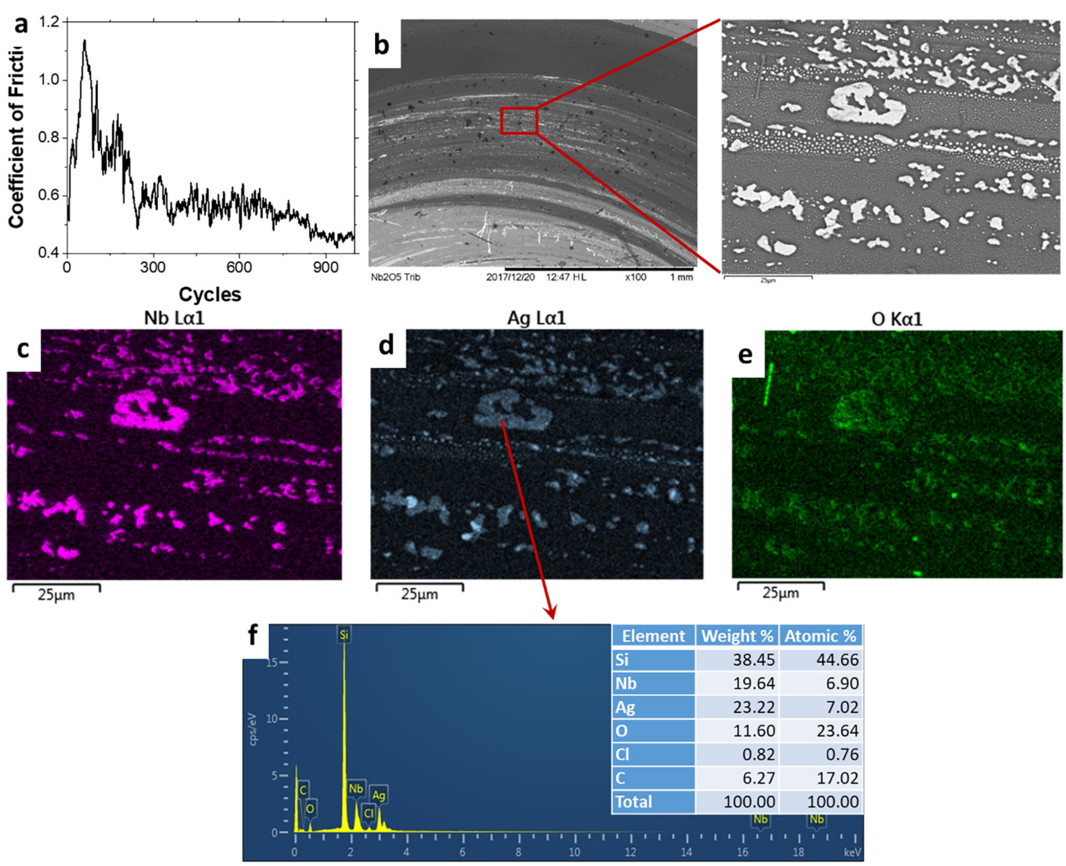


Fig. 4. (a) Cracks formed during the indentation test on the $Nb_2O_5/Ag_2O/Nb_2O_5$ sample; (b) after annealing the cracks became invisible; Cross-sectional micrograph of (c) region 1 and (d) region 2;EDS analysis of cross section 2 for (e) Si, (f) Nb, (g) Ag, and (h) O, indicating a larger content of silver and oxygen at the crack site.

$Ag_2Nb_4O_{11}$ [30]. Raman spectroscopy revealed that the Nb_2O_5 contributions at 667, 810 and 946 cm^{-1} disappeared while two new peaks at 236, 622 cm^{-1} that correspond to the ternary phase appeared [4,20].

4. Conclusions

In this work, we explored surface reconstruction mechanisms in $Nb_2O_5 + Ag_2O$ systems, which are thermo-mechanically activated during tribological tests at elevated temperatures. The formation of lubricious ternary oxide films under tribological sliding conditions was demonstrated in bulk and thin film samples at 600 °C, the temperature > 300 °C lower than the one required in purely thermodynamical processes. The surface reconstruction process enabled the activation of the self-healing mechanism, which helped to increase wear resistance. Extensive characterization analysis indicated the formation of $AgNbO_3$ and $Ag_2Nb_4O_{11}$ phases associated with the improvement of the material's frictional properties. The study provides new insights into the self-healing processes in ceramics and proposes an approach for in-situ repair of materials.



Element	Weight %	Atomic %
Si	38.45	44.66
Nb	19.64	6.90
Ag	23.22	7.02
O	11.60	23.64
Cl	0.82	0.76
C	6.27	17.02
Total	100.00	100.00

Fig. 5. (a) Tribologically behavior of $Nb_2O_5/Ag_2O/Nb_2O_5$ multi-layered coating. Surface morphology of the coating after tribology test (b). EDS map of Nb (c), Ag (d) and O (e) are almost replica each other's, indicates reaction between Nb_2O_5 and Ag_2O layers to forming a ternary oxide of Nb-Ag-O. (f) The EDS spectra and percentage of each element of the coating after tribology tests.

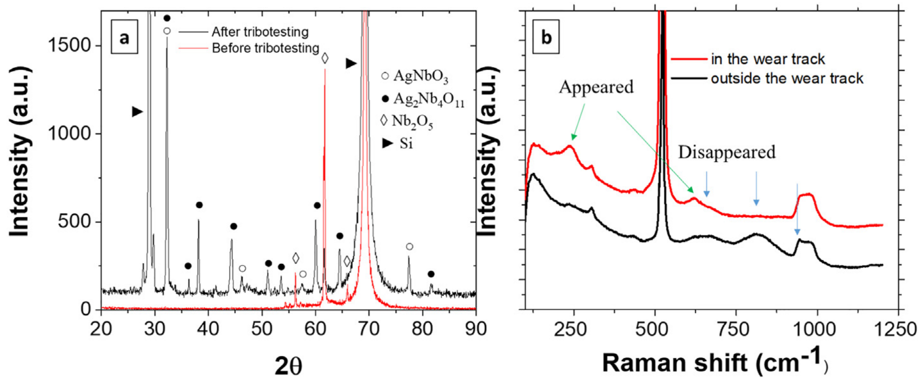


Fig. 6. (a) XRD and (b) Raman analysis of the healing-induced modifications in the composition of the wear track.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.surfcoat.2019.03.002.

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