

A Preliminary Study on Hybrid Use of Thermal Spray Coating and Ultrasonic Nanocrystalline Surface Modification Technique on the Tribological Properties of Yttria-Stabilized Zirconia Coating

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An ultrasonic nanocrystal surface modification (UNSM) technique was applied to a thermally sprayed yttria-stabilized zirconia (YSZ) ceramic coating deposited onto a hot tool steel substrate to improve the mechanical and tribological properties. The friction test results showed that the UNSM-treated coating had a smoother surface, a lower friction, and a higher resistance to wear compared to that of the as-sprayed coating. It was also demonstrated that the UNSM technique improved the adhesion behavior of the coating by about 24%. Hence, it was found that a hybrid use of thermal spray coating (TSC) and UNSM technique is a meaningful way to bring together synergy effect of two emerging surface technologies in terms of tribology. [DOI: 10.1115/1.4032524]

Introduction

Thermally sprayed YSZ coatings conventionally using 10–100 μm sized powders are metal compound sprayed onto substrates made of various metallic materials and alloys to increase wear-resistance, erosion, cavitation, fretting and to provide protection from corrosion at high temperatures [1,2]. Over the past years, YSZ ceramic coatings are widely used in various industries such as aerospace, automotive, biomedical, etc., requiring low friction and high resistance to wear at high temperatures [3–6]. It is considered that the surface roughness of the YSZ ceramic coatings that are used with their surfaces in the as-sprayed condition for some applications is too rough including a huge number of pores and cracks. It has been reported earlier that thermally sprayed YSZ coatings have a structure with inherent porosity (>15 vol. %) which can deteriorate the mechanical properties and wear performance by the presence of cracks which can be easily initiated during sliding [7,8]. The durability and wear behavior of YSZ coatings depend on the size and shape of porosity and crack. It has been reported earlier that surface defects such as porosity and cracks of ceramic coatings can be eliminated using additional treatments such as hot isostatic pressing and peening densification [9]. Moreover, Li et al. have reported the study on the improvement in wear-resistance of plasma sprayed YSZ ceramic coating using a nanostructured powder [10]. The coatings were prepared using conventional and nanostructured powders with the optimized process to achieve the highest surface hardness and the smoothest surface roughness along with the smallest number of porosities. It was found from the results of ball-on-disk wear tests that the YSZ coating sprayed using the nanostructured powder exhibited higher resistance to wear compared to that of the coating sprayed using the conventional powder. They conclude that the enhancement in resistance to wear of the YSZ coating sprayed

using the nanostructured powder may be attributed to the decrease in number of pores and cracks on the surface of the YSZ coating. Ramachandran et al. have investigated the possibility of controlling the friction and wear behavior of the YSZ ceramic coatings [11]. The effect of porosity on the friction and wear behavior of the YSZ coating against a sintered tungsten carbide (WC) has been studied. It was found that the friction and wear of the coating can be controlled by porosity, where the resistance to wear increased with decreasing the number of pores on the surface of the coating.

Hence, it is obvious that in order to improve the sliding friction and wear behavior of the YSZ ceramic coatings, it is essential to modify the surface of coatings to obtain outstanding mechanical properties and a small number of pores on the surface. The UNSM technique in this respect is one of the widely used surface modification technique which is able to increase the mechanical properties and to improve the sliding friction and wear behavior of thermally sprayed YSZ coating. To our best knowledge, there are no investigations pertaining to hybrid use system (UNSM + TSC) in the literature which need to be investigated in-depth to understand the microstructure of the YSZ coating. The main objective of this study is to evaluate the effectiveness of the UNSM technique on the mechanical properties and sliding friction and wear behavior of YSZ coating. In the present study, the thermally sprayed YSZ ceramic coating deposited onto a hot tool steel substrate was treated by the UNSM technique. Subsequently, the mechanical, tribological, and adhesion properties of as-sprayed and UNSM-treated YSZ coatings were investigated. It is expected from this study that understanding such properties of thermally sprayed YSZ ceramic coatings would be important consideration to develop a hybrid use of two emerging surface technologies with high durability and reliability.

Experimental Procedure

Specimen Preparation. Coatings were prepared using an air plasma spraying coating gun mounted on an FANUC[®] robot

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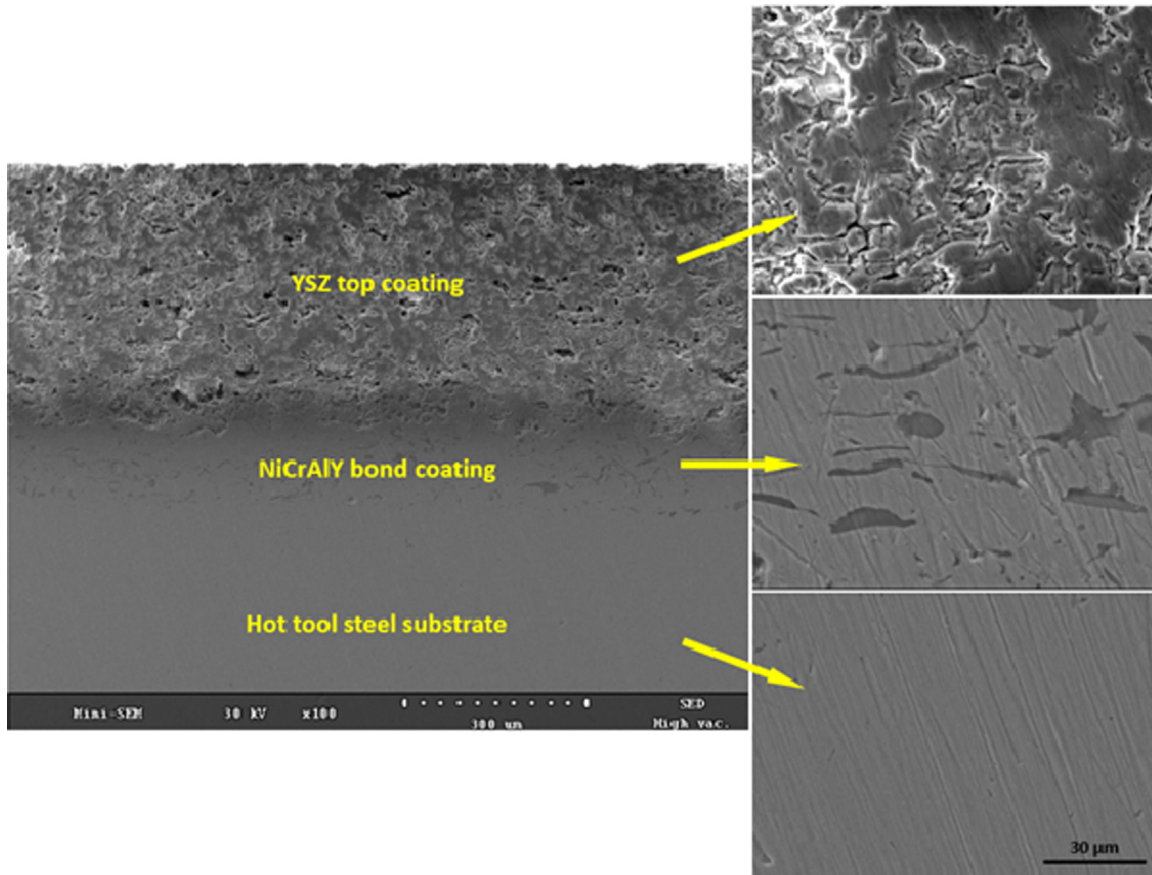


Fig. 1 Cross-sectional scanning electron microscope (SEM) image of the specimen: YSZ top coating, NiCrAlY bond coating and hot tool steel substrate

Table 1 UNSM treatment parameters

Frequency (kHz)	20
Amplitude (μm)	30
Impact load (N)	35
Speed (rpm)	30
Ball size in diameter (mm)	7.62
Ball material	Si_3N_4

(M-710, Germany). Primary nitrogen (N_2) and secondary hydrogen (H_2) gases were used to deposit both bond and top coatings. Nozzle spray distance for the YSZ ceramic coating and the NiCrAlY layer was 100 mm. The specimens were sprayed onto hot steel substrate disks with a diameter of 25 mm and a thickness of 7.9 mm. Prior to spraying of the YSZ ceramic coating with a chemical composition of ZrO_2 92% and Y_2O_3 8% in wt.%, a NiCrAlY layer with a chemical composition of Cr 22%, Al 10%, Y 1% and Ni bal. in wt.%, was applied as bond coating to enhance the adhesion between the substrate and the top coating. Figure 1 shows the cross section morphology of the specimen where the

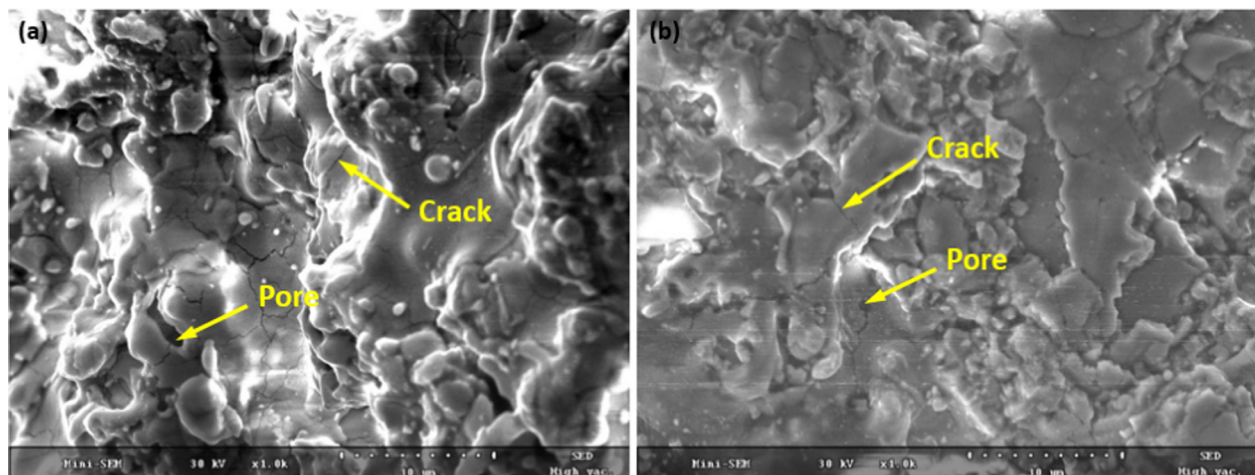


Fig. 2 SEM images of the thermally sprayed YSZ ceramic coatings before and after UNSM treatment

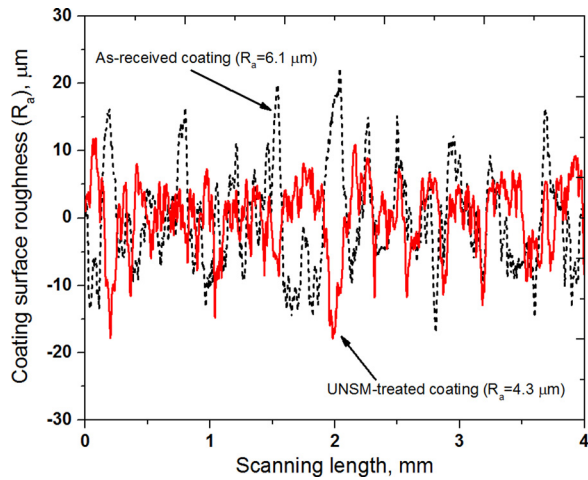


Fig. 3 Surface roughness profiles of the thermally sprayed YSZ ceramic coatings before and after UNSM treatment

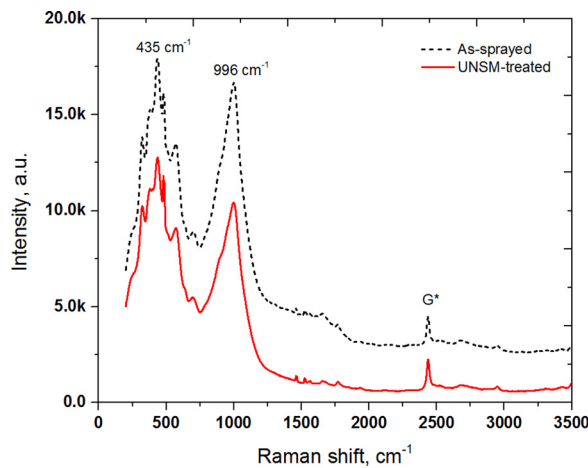


Fig. 4 Raman spectra taken from the as-sprayed and UNSM-treated thermally sprayed YSZ ceramic coatings

thickness of the bond and top coatings was found to be about 40 and 300 μm , respectively. The morphology of the layers is shown in high-magnified SEM images as well. The thermally sprayed YSZ coatings were subjected to the UNSM technique under the

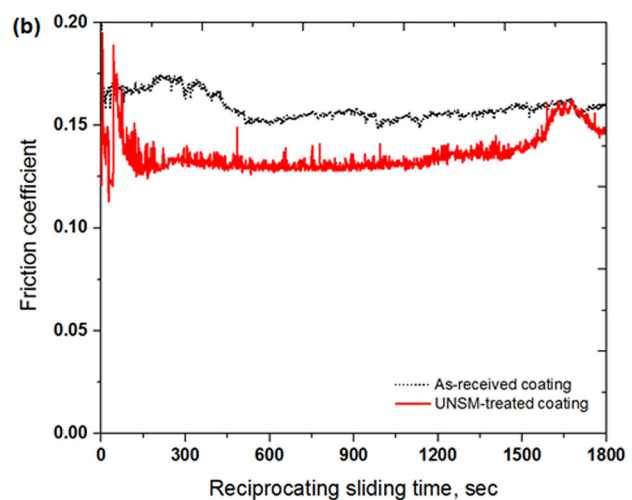
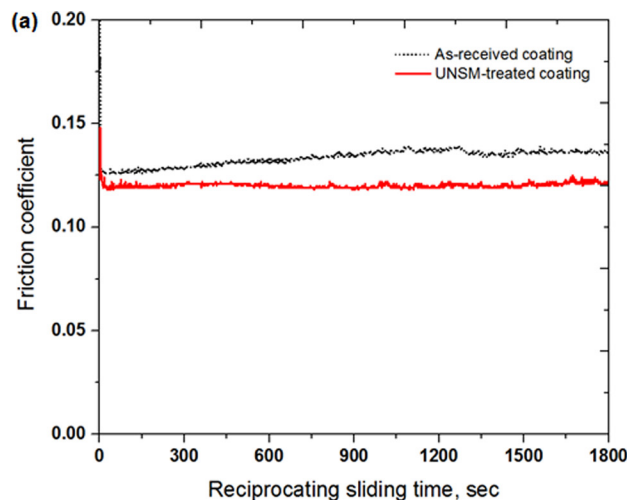


Fig. 5 Friction coefficient of the as-sprayed and UNSM-treated thermally sprayed YSZ ceramic coatings at temperatures of 25 °C (a) and 200 °C (b)

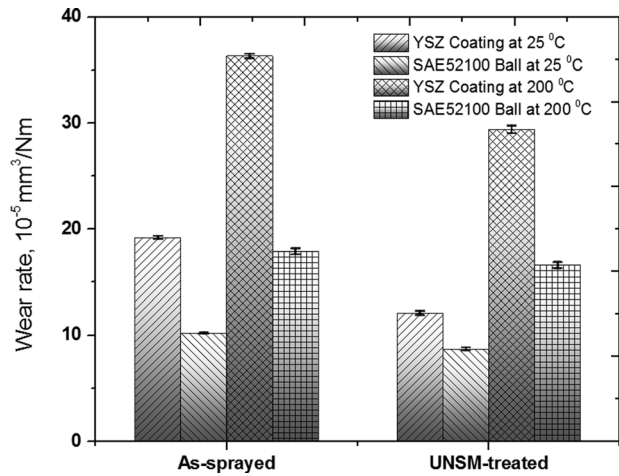


Fig. 6 Comparison of the wear rate for the as-sprayed and UNSM-treated coatings and counter surface balls at temperatures of 25 and 200 °C

parameters as shown in Table 1 (more details of the UNSM technique can be found in our previous studies [12–14]). Prior to the UNSM treatment, the coatings were cleaned in ethanol and de-ionized water for 10 mins each using an ultrasonic bath.

Friction and Wear Test. Tribological tests on the as-sprayed and UNSM-treated specimens against bearing steel ball (10 mm in diameter) were performed at a normal load of 50 N, a frequency of 30 Hz with a stroke of 1 mm for 30 mins at temperatures of 25 °C and 200 °C. The configuration of a tribometer can be found in our previous study [12]. Wear rate of the specimens was calculated based on the wear track profiles obtained by using a profilometer (Mitutoyo SJ-210, Japan).

Microscratch Test. Scratch tests on the as-sprayed and UNSM-treated coatings were carried out using a microscratch tester (CSEM, Neuchâtel, Switzerland) equipped with a Rockwell C indenter with a radius of 200 μm at a progressive load from 15 to 55 N and a scratch speed of 20 mm/min over a scratch distance of 4 mm. During the scratching, the applied progressive load, friction coefficient, and friction force were recorded.

Specimen Characterization. The microstructure, surface roughness, surface hardness, and Raman shift of the YSZ coatings

were characterized. The surface microstructure, wear track, and the chemistry of the coatings were investigated using a nano-eye mini SEM (SEM; SNE-3000 M SEC, South Korea) and an energy dispersive X-ray spectroscopy (EDS; EDAX, Metek, Mahwah, NJ). The surface roughness of the specimens was obtained using a two-dimensional (2D) surface profilometer (Mitutoyo SJ-210, Japan). The hardness of the coatings was measured using a portable microhardness (Vicker's) tester (MIC20, Krautkramer, Germany) at a load of 300 gram force for a dwell time of 15 s. For each coating, ten indentations and three surface roughness profiles were randomly obtained to measure the hardness and roughness, respectively. The phase transformation of the coatings and at worn surface was examined using a Raman spectroscopy (LabRam HR, Horiba, Japan).

Results and Discussion

Coating Characterizations. Figure 2 shows the microstructure of the as-sprayed and UNSM-treated coatings as observed by SEM. It is obvious that the UNSM technique improved the microstructure of the coating, where the number of cracks and pores were decreased. The pore size and pore distribution play a vital role in determining the tribological properties of the coatings. In particular, variations in density can be clearly observed as well. However, the as-sprayed coating possesses the most interlamellar and intralamellar cracks (see Fig. 2(a)) compared to that of the UNSM-treated coating. The typical thickness of the cracks was in the range of 0.04 and 0.8 μm with the distance between cracks varying between 2 and 5 μm .

Figure 3 shows the comparison of average surface roughness which was found to be 6.1 μm and 4.3 μm for the as-sprayed and UNSM-treated coatings, respectively. It has been reported earlier that the low surface roughness of YSZ coating has a significant influence on reduction in friction coefficient and wear [15]. The hardness measurement of the coatings revealed that the surface hardness of the as-sprayed and UNSM-treated coatings was about 495 and 539 Vicker's hardness, respectively. It is well known that the hardness plays an important role in determining wear-resistance of coatings. The increase in hardness by the UNSM technique can be attributed to the work-hardening, plastically deformed coating layer at the top surface and decrease in the porosity as shown in Fig. 2(b).

Figure 4 shows a Raman spectrum taken from the as-sprayed and UNSM-treated coatings. The Raman spectrum results revealed that no additional phase was detected after UNSM treatment, but the UNSM-treated coating has a lower intensity compared to that of the as-sprayed coating which may be attributed to the microstrain [16]. The highest intensity was found at 435 cm^{-1} for the as-sprayed and UNSM-treated coatings. The peaks below 200 cm^{-1} were not detected which may be attributed to the absence of O vacancies in the crystal lattice of ZrO_2 . The phase was found to be tetragonal for the as-sprayed and UNSM-treated coatings similar to that of the starting powder indicating the formation of nontransformable tetragonal zirconia phase.

Friction and Wear Characteristics. Figure 5 shows the friction coefficient of the as-sprayed and UNSM-treated coatings with

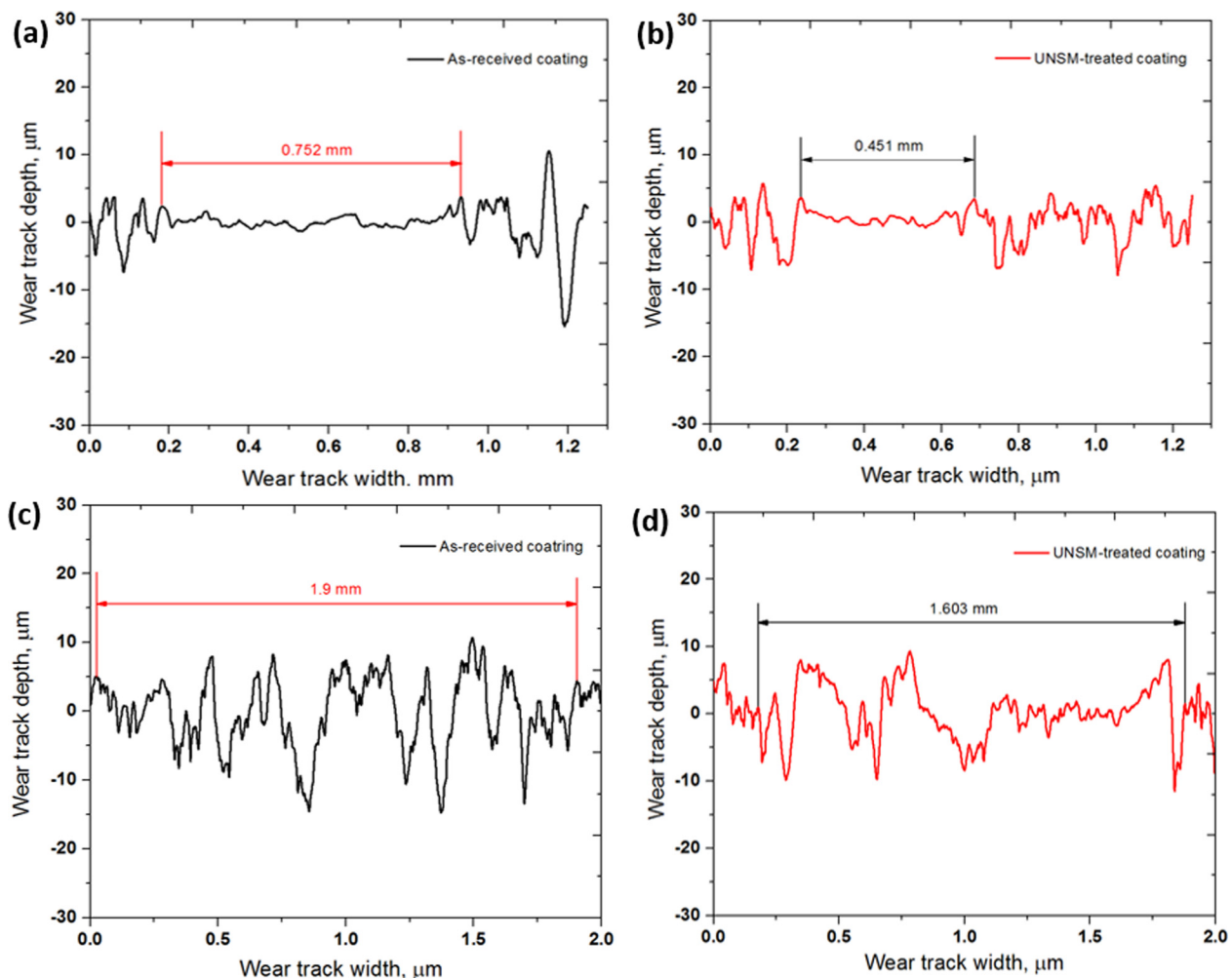


Fig. 7 Cross-sectional profile of wear tracks formed on the as-sprayed (a) and (c) and UNSM-treated (b) and (d) thermally sprayed YSZ ceramic coatings at temperature of 25 and 200 $^{\circ}\text{C}$, respectively

respect to sliding time at temperatures of 25 and 200 °C. It can be seen that the UNSM-treated had a lower friction coefficient compared to that of the as-sprayed coating at both temperatures. At a temperature of 25 °C, the friction coefficient of the as-sprayed coating increased slowly until 20 mins of sliding and then got stabilized, while the friction coefficient of the UNSM-treated was very stable with no fluctuation from the onset of the test. At a temperature of 200 °C, the friction coefficient of the as-sprayed coating was unstable from the beginning of the test with a value of about 0.161, while the friction coefficient of the UNSM-treated got stabilized after sliding time of about 5 mins. This behavior of the coatings may be attributed to the surface roughness where it plays a significant role in behaving running-in period and it can be explained that the interaction between the counter surface ball and the disk specimen occurs on the asperities where the asperities do not experience plastic deformation, while in the steady-state period the asperities experience plastic deformation and the wear debris come out within the wear track. It was found that the friction coefficient of the UNSM-treated specimen increased and again reduced gradually after sliding time of 25 mins. The increase in friction behavior of the UNSM-treated specimen may be attributed to the third-body abrasion and accumulation of wear debris/particles at the contact interface of the specimens, while the reduction in friction coefficient may be attributed to the absence of accumulated wear debris/particles which were pushed away from the contact interface by continuous sliding [17]. In addition, it needs to be mentioned here that the friction coefficient of the specimens at a temperature of 200 °C exhibited not only high friction coefficient compared to the specimens at a temperature of 25 °C, but also high fluctuations in the value of friction coefficient which is associated with the transfer of the coating onto the counter surface ball or vice versa and the formation of the oxide layers on the coating. Hence, the transfer of the coating and adhesion

under friction at a temperature of 200 °C are responsible for the high fluctuations in the value of friction coefficient. Rapoport et al. have reported that relatively high friction coefficient with high fluctuations of the coatings at a high temperature may be mainly attributed to the material transfer [18].

Figure 6 presents the wear rate of the as-sprayed and UNSM-treated coatings and the counter surface balls mated with those coatings at both temperatures. It is clear that the UNSM-treated coating led to a higher wear-resistance compared to that of the as-sprayed coating at both temperatures. Also, the wear of the counter surface ball mated with the UNSM-treated coating was smaller than that of the counter surface ball mated with the as-sprayed coating. It needs to be mentioned here that the distinction in friction coefficient was small, but in wear rate it was significant. As a result, the wear rate of the coatings and counter surface balls at both temperatures decreased by about 36 and 15%, and 20 and 8%, respectively. It was found that the wear rate of the coatings are in good agreement with the friction coefficient results.

Worn Surfaces. Figure 7 shows the profiles of wear tracks formed on the as-sprayed and UNSM-treated coatings at both temperatures. It was revealed that the UNSM-treated coating exhibited narrower and shallower wear track compared to that of the as-sprayed coating. It can be also seen from Fig. 7 that the wear track of the UNSM-treated coating had a relatively smoother surface than that of the as-sprayed coating at both temperatures, which is a smoothing phenomenon. It was also found that the surface roughness of the as-sprayed and UNSM-treated coatings after friction and wear tests increased with increasing the temperature. The high surface roughness of the specimen tested at a temperature of 200 °C compared to a temperature of 25 °C may be attributed to the experienced more plastic deformation, high-

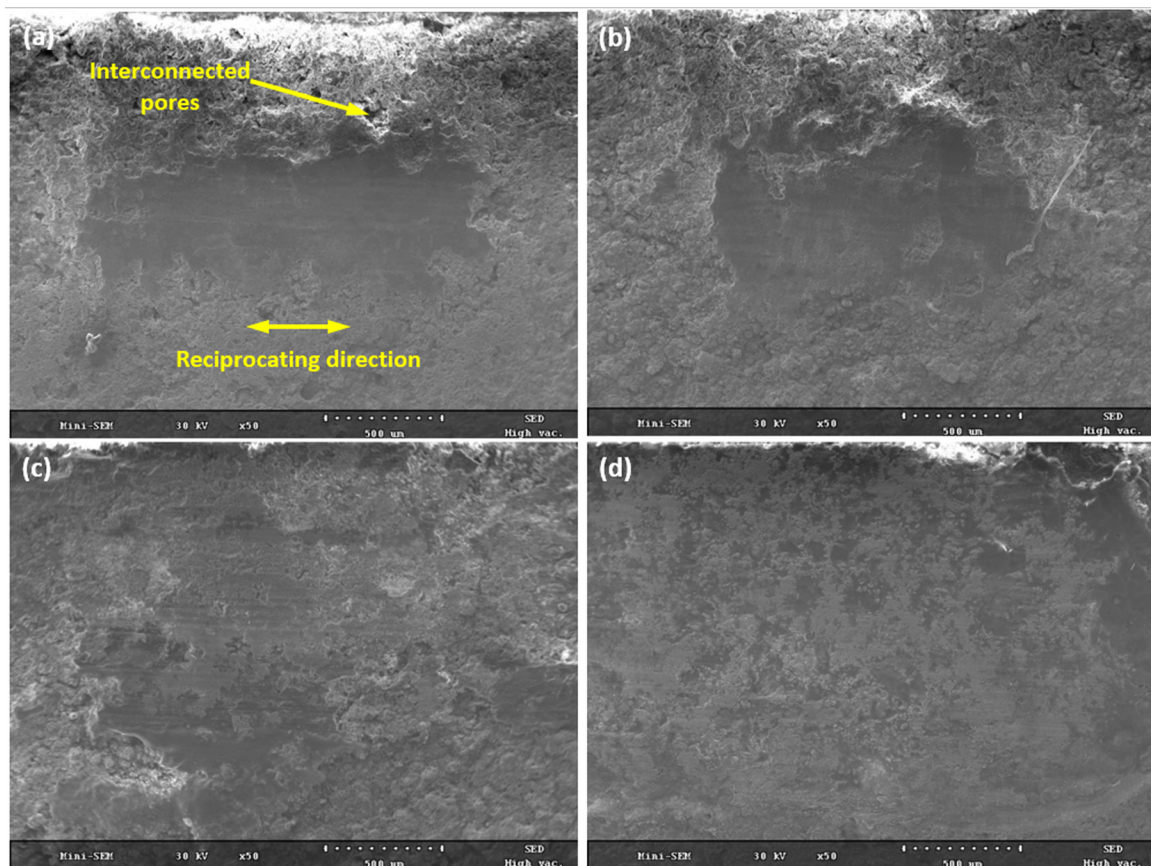


Fig. 8 SEM images of partially wear track generated on the as-sprayed (a) and (c) and UNSM-treated (b) and (d) thermally sprayed YSZ ceramic coatings at temperatures of 25 and 200 °C, respectively

temperature oxidation and material transfer from the counter surface. It has been reported earlier that the wear loss of YSZ coatings increased with increasing the temperature which can be reached to the maximum wear loss at a temperature of about 400 °C. The increase in the wear loss of YSZ coatings with temperature can also be explained by phase transformation from tetragonal to monoclinic [19].

The pores and cracks on the surface of the coatings may act as the crack initiation cite during sliding. Figure 8 shows the SEM images of the whole and partially worn surfaces on the as-sprayed and UNSM-treated coatings at temperatures of 25 °C and 200 °C, respectively. Some intralamellar interfaces and fracture on the worn surfaces at a temperature of 25 °C were observed. Interconnected large pores (see Fig. 8(a)) are also observed on the worn surface of the as-sprayed coating, while no such interconnected large pores on the worn surface of the UNSM-treated coating. It can be seen from Figs. 8(c) and 8(d) that the worn surface of the UNSM-treated coating was smoother having less fracture fragments compared to that of the worn of the as-sprayed coating, where the wear mechanisms are found to be adhesive and smearing. Randomly distributed areas of worn surface patches of the as-sprayed coating were relatively bigger than that of the worn surface on the UNSM-treated coating at both temperatures. The increase in wear-resistance of the UNSM-treated coating compared to the as-sprayed coating can be explained in terms of increase in surface hardness which is a primary material property that determines wear resistance.

Energy dispersive X-ray spectroscopy (EDS) is a valuable X-ray technique used to identify the qualitative and quantitative elemental composition of materials. Each element has the characteristic spectra showing peaks corresponding to the elements making up the true composition. Figure 9 shows the EDX spectrum taken from wear tracks formed on the as-sprayed and UNSM-

treated coatings at both temperatures. Results revealed that some elements such as C, Fe, Mo, and Ti were identified on the UNSM-treated coating, while C, Fe, Co, and Mo elements were detected on the as-sprayed coating at both temperatures. It is believed that those detected elements on the coatings surface were transferred from the counter surface ball during the test under dry conditions. It is worth mentioning here that the detected elements were more prevalent on the as-sprayed coating than that of the UNSM-treated coating at both temperatures. Thus, it can be considered that the counter surface ball was more penetrated onto the as-sprayed coating than that of the UNSM-treated coating due to low contact stress, low hardness, and less number of asperity contacts. In addition, O element was detected on the as-sprayed and UNSM-treated coating at both temperatures. However, the degree of oxidation on the UNSM-treated was less 4.30 wt.% than on the as-sprayed coating 6.19 wt.% at a temperature of 25 °C, while it was 4.52 wt.% and 6.91 wt.% at a temperature of 200 °C as shown in the inset of Fig. 9.

Material transfer from one to another between the mating surfaces usually plays an important role in the performance of many tribological contacts. After investigating the worn surface features by SEM-EDX, it can be concluded that the wear mechanisms of the coatings that were in contact with a bearing steel were adhesion and oxidation at both temperatures. Moreover, the wear of the counter surface balls came in contact with the coatings showed plastic deformation and ploughing. A slight plastic deformation occurred on the counter surface ball mated with the UNSM-treated coating, which may be mainly attributed to the increase in hardness.

The original existing defects in the as-sprayed coating such as the pores, intralamellar cracks, and high surface roughness (see Figs. 2 and 3) may act as the spot where crack initiates and even propagates during friction and wear test under dry conditions,

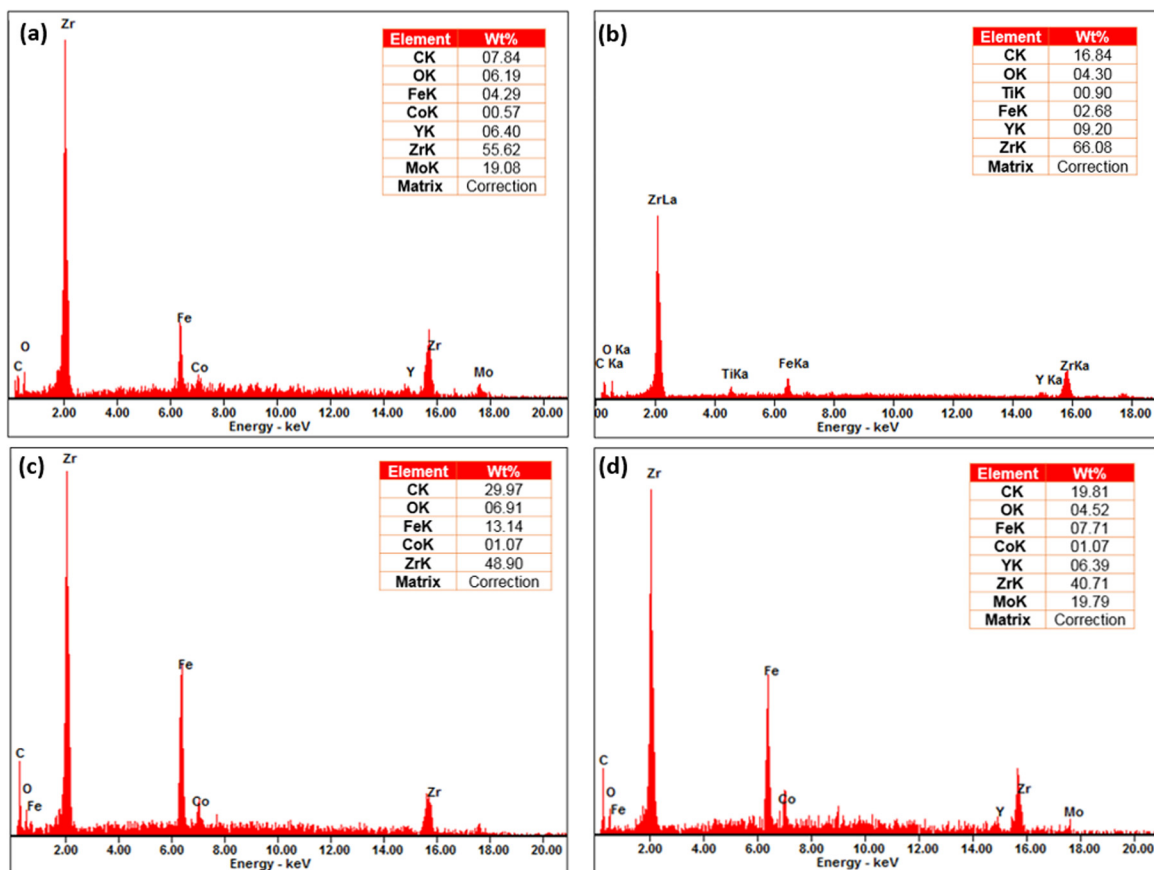


Fig. 9 EDX spectra taken from wear tracks formed on the as-sprayed (a) and (c) and UNSM-treated (b) and (d) YSZ ceramic coatings at temperature of 25 and 200 °C, respectively

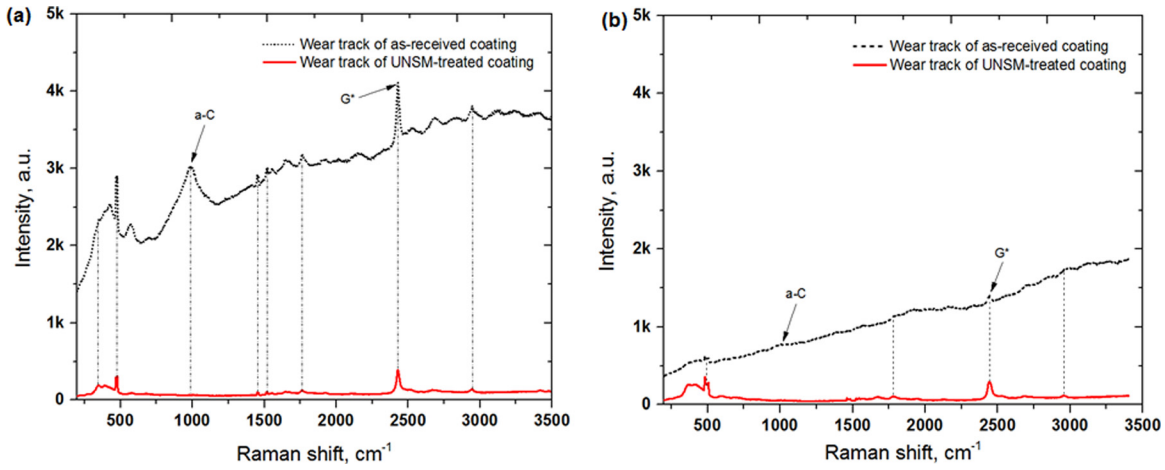


Fig. 10 Raman spectra taken from wear tracks formed on the as-sprayed and UNSM-treated coatings at temperatures of 25 °C (a) and 200 °C (b)

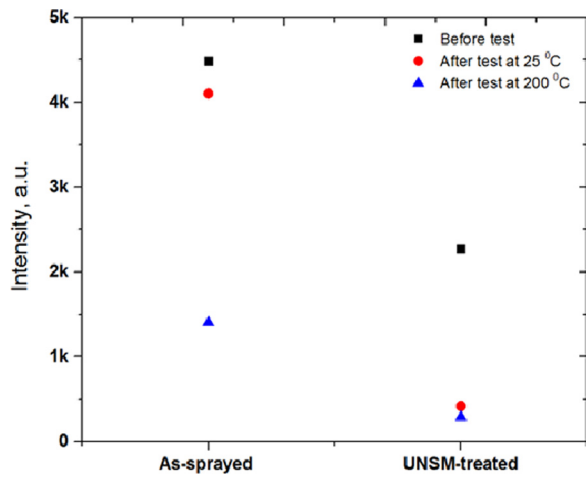


Fig. 11 Comparison of G-bond intensity peak taken from wear tracks formed on the as-sprayed and UNSM-treated coatings before, and after friction and wear tests at temperatures of 25 °C (a) and 200 °C (b), respectively

resulting in fracture of the coating. The improvement in friction and wear behavior of the UNSM-treated coating may also be related to the Raman phase transformation shown in Fig. 10, where the Raman spectrum taken from wear track formed on the as-sprayed and UNSM-treated coatings at both temperatures. It was found that no new phase was found on the specimens after friction and wear tests. However, no intensity peak at 989 cm^{-1} was detected for the wear track of the UNSM-treated coating at

both temperatures. It can be seen that the intensity peaks of the wear track of UNSM-treated coating reduced significantly compared to that of the wear track of as-sprayed coating which may be due to the surface being rougher of the as-sprayed coating compared to that of the UNSM-treated coating after friction and wear tests. Figure 11 shows the comparison of G-bond intensity peaks at 2436 cm^{-1} , 2426 cm^{-1} , and 2445 cm^{-1} for the as-sprayed and UNSM-treated coatings before and after tests at temperatures of 25 °C and 200 °C, respectively. It is obvious that the intensity of G-bond peak for the coatings before test was higher than that of the after test. However, no significant difference in intensity of G-bond peak for the UNSM-treated coatings was observed after tests at both temperatures. As a result, the UNSM technique capable of increasing YSZ coating hardness, reducing roughness, and partially eliminating pores and cracks. It has been reported earlier that fewer pores, cracks, finer grain size would also improve the hardness of the coatings [20], while wear-resistance of materials tends to improve with increasing hardness [21]. Moreover, Raman spectroscopy studies revealed that the UNSM technique reduced the intensity peaks of YSZ coating which may be related to the small change in C concentration before and after the UNSM treatment.

Adhesion Behavior. The variation in the progressive load, friction force, and friction coefficient with respect to scratch distance for the as-sprayed and UNSM-treated coatings recorded during the microscratch test is shown in Fig. 12. It is obvious that the friction coefficient of the UNSM-treated coating was lower compared to that of the as-sprayed coating by about 24%. As can be seen from Fig. 12, at the onset of scratch test, the friction coefficient of the as-sprayed coating increased to a value of 0.80 and started fluctuating till the end of a scratching, which was

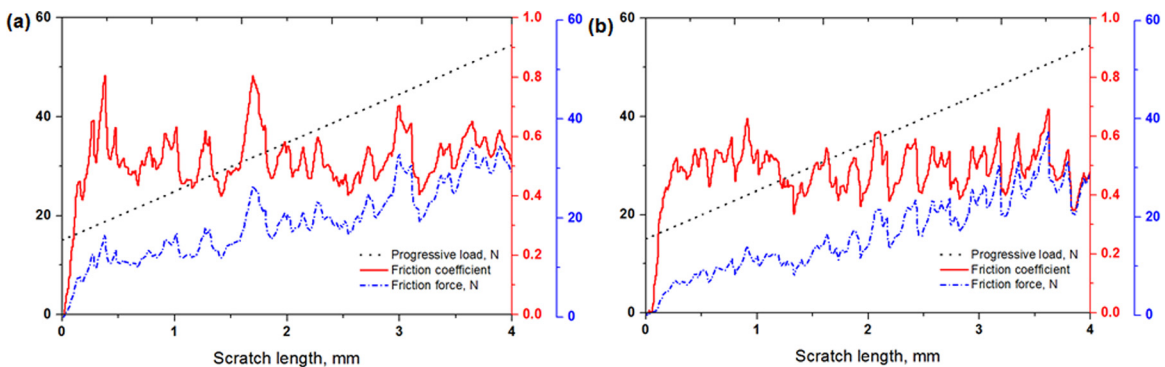


Fig. 12 Scratch behavior of the as-sprayed (a) and UNSM-treated (b) coatings

indicative of severe failure and deformation of the coating. However, the friction coefficient of the UNSM-treated coating increased up to a value of 0.55 with a relatively lower fluctuation which signifies the effectiveness of UNSM on adhesion property at the top surface of a coating. The reduction in friction and fluctuation of the UNSM-treated coating may be attributed to the reduced surface roughness, modified microstructure where the number of pores and cracks was decreased. It has been pointed out earlier that the acoustic emission signal is not a perfect method to define the adhesive failure of sprayed coatings [22]. However, it was possible to enhance the scratch resistance and to reduce the friction coefficient of a coating in comparison with the conventional thermally sprayed YSZ coating by the UNSM technique.

Conclusions

The mechanical and tribological properties of thermally sprayed YSZ coating deposited onto the hot tool steel substrate were investigated. Results revealed that the UNSM-treated coating has smoother surface, lower friction and higher resistance to wear compared to that of the as-sprayed coating. Wear mechanisms of the coatings were found to be adhesion, smearing and oxidation for the as-sprayed and UNSM-treated coatings at both temperatures. The enhancement in wear-resistance of the UNSM-treated coating may be mainly attributed to the increase in surface hardness, reduction in surface roughness and decrease in number of pores and cracks of the coating. As a result, it was found that hybrid use of TSC and UNSM technique is meaningful to bring together synergy effect of two emerging surface technologies. The effectiveness of UNSM technique on the mechanical properties and wear behavior at high temperatures ($>700^{\circ}\text{C}$) of YSZ top coating sprayed onto substrates made of aerospace materials will be investigated in the near future.

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