Linear reciprocating wear behaviour of plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coatings at different loading and sliding conditions

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Abstract. Linear reciprocating wear test is carried out on atmospheric plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coatings applied on steel substrates. Linear bi-directional sliding wear test of the coatings is performed at different loading and sliding conditions such as load (10, 20 and 30 N), reciprocating amplitude (1.5, 3 and 6 mm) and frequency (5, 10, 15 and 20 Hz) using ball on flat linear reciprocating tribometer. The patterns of tribological behaviour of the coatings, as manifested at the tribo-contact surface, are judged. Results have shown that the wear rate increases with increasing applied load and frequency and that decreases with increasing reciprocating amplitude. Plastic deformation, detachments of unmelted core, reattachments, delamination and adhesive wear dominate the main failure mechanism of coating.

Keywords. $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$; coating; load; wear; frequency; reciprocating amplitude.

1. Introduction

Coatings developed by thermal spray techniques enhance the service life of various functional components by improving resistance to wear, corrosion, oxidation at normal or elevated temperatures [1–3]. A number of research groups have performed tribological characterisation of plasma-sprayed Al_2O_3 and Cr_2O_3 coatings under a single set of wear test parameters. Tao *et al* [3] chose dry sliding conditions to characterise the coating. Bollelli *et al* [4] studied the wear resistance using pin on disk and dry sand steel abrasive wheel setups. Fernandez *et al* [5] and Pantelis *et al* [6] have performed similar tests on plasma-sprayed coatings. Erickson *et al* [7] and Vicent *et al* [8] reported the tribological properties of plasma-sprayed Al_2O_3 coating using the same set of parameters. Similar studies are performed by Manojkumar *et al* [9] on wear rate analysis of plasma-sprayed $\text{Al}_2\text{O}_3\text{--ZrO}_2$ composite coating. A number of research groups have explored the tribological properties of thermally sprayed coatings under varying test parameters, such as load and sliding velocity [10–26], sliding distance [22, 27], time of sliding [28], number of cycles [12, 29], frequency, reciprocating amplitude [26] and high temperature [20, 23, 29] conditions. However, few reports are available on the compound effects of the variations in loading and sliding conditions on the tribological performance of the coating. In the present study, the authors tried to map the tribological behaviour of plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coating under the compound effects of

frequency, applied load, and amplitude in a linear reciprocating tribometer.

2. Experimental procedure

2.1 Materials and methods

$\text{Al}_2\text{O}_3\text{--}4\text{wt}\%\text{Cr}_2\text{O}_3$ powder is deposited on low carbon steel (AISI1020; Dimensions: $75 \times 60 \times 8 \text{ mm}^3$) by atmospheric plasma-spraying technique [30]. A plasma spray gun mounted on a CNC X-Y manipulator (3MB II, Sulzer Metco, USA) is used for this purpose. The steel substrates are grit blasted ($R_a = 4 \pm 0.03 \mu\text{m}$) prior to the application of plasma-sprayed layer of Ni-5wt%Al (Metco 450NS) bond coating (thickness: 70–90 μm). The deposition is made using nitrogen as plasma-forming gas and hydrogen as secondary gas. The spraying parameters used in the deposition process are as follows: N_2 flow: 50 SCFH, H_2 flow: 5 SCFH, current: 450 A, voltage: 75 V, standoff distance: 125 cm, powder feed rate: 4.5–4.8 kg/h, scanning velocity of robotic arm: 200 mm/s, gun nozzle diameter: 7 mm and powder feed nozzle diameter: 2 mm. Agglomerated Al_2O_3 (Hindalco, India, Indal calcined HI grade, purity: 99.6%) and Cr_2O_3 (Saint Gobin, India, #2020, purity: 99.05%) powders are used as feedstock material. The size ranges of Al_2O_3 and Cr_2O_3 feedstock powders selected are of $-100 + 30 \mu\text{m}$ and $-20 + 5 \mu\text{m}$, accordingly. The same set up is used for the application of top coating of $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ on the previously applied bond coat. The average thickness of the $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coating is 350 μm .

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The substrates are preheated up to 200°C using plasma torch prior to the deposition of coating material.

2.2 Characterisation of coating

Cross sections of coating are polished using (*Struers LaboPol 25*) auto polisher unit for metallographic study. Polishing is carried out by using wet abrasive paper (SiC) and diamond paste on selvyt cloth to achieve mirror finished cross-sectional surface. The microstructure of the polished samples is studied by using field emission scanning electron microscope (*FESEM, Zeiss Supra-55*). The phases present in the as-sprayed coatings are identified by X-ray diffraction (XRD) using a *Rigaku Smart Lab 3 KW* diffractometer with nickel-filtered Cu K α radiation ($\lambda = 0.15406$ nm). The scanning angle ranges from 20° to 90° with a step size of 0.01° and a scanning speed of 2°/min. In order to assess the tribological properties of the coatings, sliding wear tests are performed with *Ducom CM-9104* fretting wear tribometer against WC-6wt%Co balls (5 mm diameter) used as counterbody. Prior to wear testing, the top surface of the coating is ground and polished. Roughness of the polished top surface is kept about 1 ± 0.12 μm . Thickness of the coated layer is maintained at 320 μm after polishing. Test is carried out under different conditions such as load, sliding amplitude, and frequency for 20 min on the top surface of the coating as

shown in table 1. The result of the wear test shown is based on five repetitive wear tests performed on the same condition. Tested specimens are cleaned with acetone in the ultrasonic bath (*Bio-Technics*) and dried with a hair dryer to remove the loose debris from the contact area between the coatings and counterbody. These loose wear debris particles influence the wear rate calculation because it adheres to the contact area. Weight loss is measured by weighing balance (*Citizen CX265N*, accuracy 0.0001 g) and volume loss is calculated by measured track depth and width by *Altisurf 500* optical profiler (figure 1a) and *Taylor-Hobson, Surtronic 25* contact-type stylus profilometer (figure 1b) for an accurate wear rate evolution. Figure 1c shows three-dimensional view of wear track obtained at 3-mm reciprocating amplitude. Wear mechanism of the coating is judged by using SEM (*FESEM, Zeiss Supra-55*).

3. Results and discussion

The SEM micrograph of agglomerated Al₂O₃ and crushed Cr₂O₃ powders used for coating are shown in figure 2a and b. The cross section of plasma-sprayed Al₂O₃-Cr₂O₃ coating is depicted in figure 2c. SEM of coating displays a typical layered structure, consisting of a bond coat next to the substrate and a top coat. Pores, partially melted regions and craters appear as darker regions at the SEM micrograph of coating.

X-ray diffraction analyses of a blend of Al₂O₃ and Cr₂O₃ powders to be used as feedstock and the coating are shown in figure 3. From X-ray diffraction pattern of as-sprayed Al₂O₃-Cr₂O₃ coating, it is found to be consisting of α and γ -Al₂O₃ phases along with Cr₂O₃, whereas the powder mixture to be used as feedstock contains α -Al₂O₃ and Cr₂O₃. During atmospheric plasma spray-coating process, a very high rate of cooling is achieved ($\sim 10^6$ K/s) as most of the α -Al₂O₃ phase converts to γ -Al₂O₃ during plasma-spraying process due to lower nucleation energy of γ -phase as compared to other phases [30, 31].

Table 1. Test conditions during sliding wear test.

Parameters	Value
Counterbody	WC-6wt%Co (5 mm diameter ball)
Load (N)	10, 20, 30
Frequency (Hz)	5, 10, 15, 20
Sliding amplitude (mm)	1.5, 3, 6
Time (min)	20
Temperature (°C)	25
Humidity (%)	40 $\pm$ 2

Figure 1. Optical image of worn surface of wear track obtained 5 Hz frequency, 10 N load and 3 mm reciprocating amplitude.

Figure 2. SEM micrograph of (a) Al_2O_3 powder, (b) Cr_2O_3 powder and (c) $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coating.

Figure 3. X-ray diffraction pattern of as-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coatings.

Hardness testing of as-sprayed coatings and relating those results to the microstructure are important, as the coatings are to be subjected to wear where higher hardness is required [7]. Presence of several phases of Al_2O_3 in the coating, as observed in figure 3, significantly affects the global mechanical properties of the coating. Besides, the nature of local bonding between splats, extent of melting of Al_2O_3 feedstock particles retaining unmelted and partially melted zones, pores, voids, discontinuities and various other microstructural features govern the local hardness and the mechanical properties. Results of microhardness test

Figure 4. Variations in the microhardness against distance from top surface of the coating.

performed across the cross section of the plasma-sprayed coating are depicted in figure 4. Values of microhardness measured at the cross section of the coating varied from 12 GPa to 16 GPa with the higher values obtained mostly near the top surface of the coatings. Interpretation and explanation of the variation in the plasma-sprayed Al_2O_3 -based coating are difficult because a much wider range of heterogeneity cause variation in deformation and failure mechanisms on mechanical loading/indentations.

$\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coatings are subjected to sliding wear test (in a ball on flat tribometer) against WC (6%Co) counter-body reciprocating at different values of frequency and

Figure 5. Variations in the wear rate with respect to amplitude at different frequency.

amplitude. Figure 5a to c shows the variation in the specific wear rate with respect to the amplitude of reciprocation at varying load and frequency. Clearly, the trend appears to be similar at each of 10, 20 and 30 N. Specific wear rate reduces steadily as the frequency in the linear reciprocating wear test increases. Figure 5a to c displays the trend of changes in specific wear rate at different frequencies. Reduction in the actual area of interaction between the counterbodies with increasing frequency may have governed the specific wear rate. Separation between rubbing surfaces increases with frequency, resulting in reduction in the contact area between the sliding surfaces. Sliding wear test (ball on flat) performed on the samples is characterised as a relative motion between the coating and a hard spherical (WC+6%Co) counterbody in contact under load. The wear mechanisms accompanying these tests are predominantly adhesion, surface fatigue and abrasion. The extent of the involvement of these mechanisms in the tribological interaction between the counterbodies is governed by the mechanical properties and elasto-plastic behaviour of the interacting surfaces and test parameters (e.g. load, sliding speed and sliding distance). The loading conditions influence the stress patterns on the test sample surface. This directly corresponds to the hardness. In the

ball-on-flat test performed over coated samples, in sliding mode, the frictional force is ideally dependent on the shear strength and the contact area. A harder coating results in a smaller contact area affecting the friction and wear. Higher the frequency, lower the momentary load, which causes reduction in effective normal force at contact surface might be due to superposition of static and dynamic forces at higher frequency. It is also found that wear rate slightly decreases with increase in reciprocating amplitude; this phenomenon may be due to interaction time between ball and surface, which decreases with increase in reciprocating amplitude at fixed load and frequency.

The cumulative response of the coating towards variation of load at different frequencies of sliding and reciprocating amplitudes show a similar trend (figure 6a to c). Within the working range, as load increases, first the specific wear rate decreases slightly and then increases. For the working amplitudes (1.5, 3 and 6 mm), the lowest specific wear rate is displayed at 20 N. Similar observations are made by a number of research groups, presenting such marginal reduction of wear volume for a certain interim load value [32].

In the initial stage of sliding, the contact occurs between the asperities of the counterbodies. At this initial break-in

Figure 6. Variations in the wear rate with respect to applied load at different frequency.

period, the asperities deform, break or get ruptured. This increases the contact area and establishes the conformity in contact between the surfaces. This happens to various extents at a number of test-load values and, subsequently, various forms of wear cause the volumetric loss of material over the span of the test. Friction force is proportional to the applied load as the force is proportional to the real contact area between the counterbodies. The relationship between load and contact area is almost linear.

Several physical and microstructural features present in the plasma-sprayed Al_2O_3 coatings, such as weak inter-splat bonding, porosity, and micro-cracks, play important role in its wear mechanism [7]. Such in-homogeneities within the coating provide easier pathways for stress-induced crack propagation at repeated cyclic loading and subsequent deformation during linear reciprocation of the counterbody [33]. This influences the formation of debris during a wear test. The debris particles may remain within the sliding contact and accumulate to form lump-like structures. The pressure exerted by the counterbody work-hardens this accumulated structures. The compressive stresses at a certain level of test load may become sufficient to embed them in the wear track surface. The accumulation on the wear track, depending on the load, may move slowly

between the counterbodies in the form of one distinctly non-uniform layer controlling the extent of interaction between the counterbodies [33]. This causes reduction in wear and volume loss from the wear track. At certain higher load and higher speed, the formation of the separating layer reduces and the volume loss increases due to de-lamination and pull out of the coating matrix at the wear track.

Sliding motions in ball-on-flat test performed in linear reciprocating tribometer on the Al_2O_3 coatings result in significant tangential forces at the interface of counterbodies leading to mainly fracture and a small amount of plastic flow. As sliding starts, the ball pressing against the plane surface gives rise to tensile stress in the plane surface outside the contact area. Tensile stress reaches a maximum value just at the edge of the circle of contact. In conformal contact, mean contact pressure reduces and the scale of fracture becomes smaller. With sliding, as the tangential force is applied to the conformal contact, normal load necessary to initiate fracture is reduced [33]. Besides, the tribochemical effects may become significant in various situations of sliding. Cracks nucleated at the surface or subsurface region of the coating during sliding grow and propagate parallel to the surface. A number of such propagating cracks, on joining together, cause a chunk of

Figure 7. SEM micrograph of worn surface of wear track obtained 5 Hz frequency, 30 N load and 3 mm reciprocating amplitude.

Figure 8. SEM micrograph of worn surface of wear track obtained 10 Hz frequency, 30 N load and 3 mm reciprocating amplitude.

coating to pull out. Increasing the contact pressure causes particle de-cohesion that increases the wear volume [34].

Scanning electron micrographs of the wear tracks helped the study of the tribological behaviour of the coating at different conditions of sliding. Figure 7a to c shows the surface features of the test performed at 5 Hz frequency, 30 N load and 3 mm reciprocating amplitude. A part of the tribo-contact region on the coating is visible in figure 7a. It shows sporadic dislodgement due to pitting and spallation caused by repeated occurrence of loading and unloading during linear reciprocation of the counterbody. It also shows a relatively smoother surface on the wear track with the presence of a moderate amount of reattachment and tensile cracks signifying adhesive wear as the predominant mode. The magnified images presented in figure 7b and c clearly depict the micro-sized pitted and spalled regions. Figure 7c helps to understand the topography of a spalled region. The region shows the shape and size distribution of

the particles generated out of dislodgement. The Al_2O_3 particles flaked off from the coating surface and remained in the contact zone between tribo-parts to get crushed under the applied load. The accumulation of the wear debris within the sliding contact to form lump-like structures and subsequent brittle fracture gives rise to the morphology as seen in figure 7c.

The SEM micrographs shown in figure 8a to c represent a test at 10 Hz sliding frequency, 30 N load and 3 mm reciprocating amplitude. The portion of the tribo-contact region visible in figure 8a depicts a relatively smoother surface with randomly dispersed zones of scuffing and spallation. Although these zones are considerably larger than those observed in figure 7, the extent of dislodgement, as understood from the magnified views (figure 8b and c), seems to be much lesser than what has occurred in case of a lower frequency of sliding (figure 7). Higher the sliding speed, the more rapid is the reduction in the friction

Figure 9. SEM micrograph of worn surface of wear track obtained 10 Hz frequency, 30 N load and 1.5 mm reciprocating amplitude.

coefficient. This situation is reflected well in the overall tribological characterisation of the coating. Lower value of friction has caused low-scale delamination and scuffing of the coating surface. The morphology of the debris particles, crushed and accumulated in the spalled regions, is considerably different in the view of the brittle fracture [35].

Figure 9a to d shows the morphology of the tribo-contact surface after performing the test at 1.5 mm amplitude, 10 Hz sliding frequency and 30 N load. The wear track on the coating shows considerably higher order of adhesive wear and reattachment of debris particle over each other. SEM micrograph of worn surface is shown in figure 9a. The worn surface is seen to have experienced dislodgement and brittle fracture in certain areas as shown in figure 9b. The dislodged chunks of material have mostly stayed within the zone and have crushed under the counterbody. Smaller crushed particles staying within the wear track cause further abrasive wear of the surface and increase the wear rate. Brittle fracture and reattachment of debris are observed on the track. Part of the wear track showing scuffing and adhesive nature of failure is shown in figure 9c. The debris particles remaining in the worn surface are clearly visible to have accumulated in the pits form at the surface. These loose particles, under the action of moving counterbody, cause widening of the tensile cracks formed on the surface [33, 35]. This augments further dislodgement of bigger chunks of material from the contact surface. Figure 9d nicely depicts one such area where loose accumulated particles have started the act of broadening a crack where dislodgement of chunks of material is about to commence. Besides, reattachment and some amount of plastic deformation at the coating surface are also visible.

The wear debris collected from all the coatings indicates considerably higher order of abrasive failure with fracture and dislodgement at the coating surface governing the sizes of the debris particles (figures 10–12). In most of the cases, under microscope, the debris particles are seen to have conglomerated to different sizes depending on the number and distribution of the sizes of the individual particles contributing to it. After the particles are pulled out from the coating surfaces, during the period of retention of the debris in the contact surface, the as-formed particles are comminuted and few larger particles are mixed into finer particle debris. These larger particles are either broken pieces generated from the original surface fractures or residues of larger fragments, those that have not been completely comminuted in the wear track [35]. Figure 10a to c shows the morphology of wear debris observed under SEM after performing the wear test at 3 mm reciprocating amplitude, 5 Hz sliding frequency and 30 N load. Figure 11a to c shows the wear debris formed in wear test carried out at 10 Hz frequency, 30 N load and 3 mm reciprocating amplitude. Figure 12a and b represents wear debris from test performed at 10 Hz frequency, 30 N load and 1.5 mm reciprocating amplitude. The higher value of specific wear rate observed in the test related to figure 10a to c corroborates to the higher rate of material removal in the form of larger chunks of material dislodged and spalled off from the coating surfaces. The specific wear rate related to figure 11a to c is lower with almost similar situations of debris formation being reflected on the surface. However, in figure 12a and b, the debris particles show evidences of the adhesive nature of interaction, leading to subsequent fracture and dislodgement of material (inset of figure 12a).

Figure 10. SEM micrographs of wear debris of wear track obtained by test at 5 Hz frequency, 30 N load and 3 mm reciprocating amplitude.

Figure 11. SEM micrographs of wear debris of wear track obtained by test at 10 Hz frequency, 30 N load and 3 mm reciprocating amplitude.

Figure 12. SEM micrographs of wear debris of wear track obtained by test at 10 Hz frequency, 30 N load and 1.5 mm reciprocating amplitude.

4. Conclusions

The effects of linear reciprocating wear test parameters on atmospheric plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coating are investigated. The present study performed with linear bi-directional sliding reveals that the tribological properties of the atmospheric plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coatings depend heavily on test conditions, such as load, frequency and reciprocating amplitude. Sliding frequency indeed affects the wear rate considerably. The wear rate decreases with the increase in the frequency of sliding. Wear rate of plasma-sprayed coatings decreases slightly as the reciprocating amplitude increases. There the wear rate first slightly decreases and then increases with rising levels of normal load. Plastic deformation, detachments of unmelted core, reattachments, delamination, brittle fracture and adhesive wear dominate the main failure mechanism of plasma-sprayed $\text{Al}_2\text{O}_3\text{--Cr}_2\text{O}_3$ coating. The study performed may help in finding the suitability of the coating for various loading and sliding conditions required for any application.

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