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Arch. Metall. Mater. **63** (2018), 1, 217-225

DOI: 10.24425/118931

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ANALYSIS OF FACTORS INFLUENCING DRY SLIDING WEAR BEHAVIOUR OF LASER REMELTED PLASMA SPRAYED Mo COATING USING RESPONSE SURFACE METHODOLOGY

Plasma spraying is a process widely used to fabricate wear resistant coatings. However, various problems are associated with plasma spraying out of which poor bonding strength between the coating and the substrate and the high porosity in the as sprayed coatings are of major concern. In order to eliminate these problems and enhance wear performance, the laser remelting process has been used. The laser remelting of plasma sprayed Mo coatings alters the wear mechanism and improves the wear resistance. The wear mechanism and wear volume loss depend on the applied load, sliding speed and sliding distance. Hence, an effort has been made to investigate the effect of process parameters on volume loss using Response Surface Methodology (RSM) based mathematical models. The experiments were planned as per Central Composite Design (CCD). The investigations revealed that the applied load was the most dominant factor affecting the volume loss of the coating. The sliding speed, sliding distance and interaction effects were considered as the next important parameters influencing the volume loss. The investigation also reveals that, the wear volume loss depends on two wear mechanisms, one being the formations of grooves along surface tribo films and other being fracture of splats with delamination of the coating.

Keywords: Laser remelting, Plasma spray, Central composite design (CCD), Response surface methodology (RSM), Mo Coating

1. Introduction

The plasma spraying technique is commonly used to fabricate wear resistant coatings. However, there are problems associated with plasma spraying during substrate and coating interaction. The most important problem is the poor bonding strength between the substrate and the coating. The sprayed coating peels off under heavy load due to poor bonding strength. Another problem related to plasma sprayed coating isthe high porosity. The rate of high porosity in the sprayed coating can reduce the wear performance of the coating [1,2].

The problems associated with plasma sprayed coating can be reduced or eliminated by using post treatments like laser remelting and electro deposition technique. The use of laser remelting may lead to the elimination of porosity, improvement of the bonding strength and chemical homogeneity. Laser remelting at optimum process parameters can also offer better flexibility such as a short processing time, minimum thermal distortion and minimum micro structural alterations to the substrate [3-5].

Mateos et al. [6] studied the tribological behaviour of laser remelted plasma sprayed tungsten carbide (WC) coatings. The results reveal that laser remelting improves the microstructure, microhardness and coating-substrate adherence to the substrate material. The study also shows laser remelted coatings to have better wear resistant under dry contact condition. Fernhdez et al. [3] investigated the effects of laser treatment on the wear behaviour of plasma sprayed Al_2O_3 coatings and reported a better wear resistance after laser treatment. Fu et al. [2] have studied the effect of laser remelting on the wear behaviour of plasma sprayed ZrO_2 ceramic coatings. Investigations proved the reduction in porosity of the coating considerably after laser treatment with improvements in wear resistence. Liang et al. [7] studied wear resistance of plasma sprayed and laser remelted coatings on aluminium alloy. The results showed that the laser treated plasma sprayed coating exhibited excellent wear resistance properties.

The laser remelting changes the wear mechanism and wear volume loss. Also, the wear mechanism and wear volume loss of coating depends on the applied load, sliding speed and sliding distance [8]. The wear of plasma prayed molybdenum coating increases with increase in applied load and sliding distance [5]. Rao and Das [9] have investigated the effect of sliding distance on the wear behaviour of as cast and heat treated Al-SiCp composites. It was observed that, the wear rate of the composite is invariant to the sliding distance and increased with applied load. Anoop et al. [10] analysed the factors influencing dry sliding wear behaviour of Al/SiCp brake pad tribosystem. They found that, at lower loads the wear rate was very less. At room temperature, the effect of velocity was negligible, however, at higher temperatures the increase in wear rate was observed with increase in velocity.

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Hence, the study of the available literature corroborates that, the wear volume loss is mainly influenced by the process parameters such as applied load, sliding speed and sliding distance. To reduce the wear volume and optimize the process parameters, different modelling methods can be used to denote the relationship between the input parameters and output variables with least number of experiments. Response Surface Methodology (RSM) is one broadly used method to denote relationship between input variables and output variables [8]. It assists to develop a model to study the correlation between applied load, sliding speed and sliding distance to the wear volume loss. Hence, in the current investigation, RSM design is used to explore the interdependence of the process parameters and second order quadratic model for the prediction of wear data. The significance of process parameters was studied by using analysis of variance (ANOVA) and the mathematical model was validated by the normal probability curve along with R^2 and Adj R^2 values.

2. Experimental details

2.1. Materials

Materials used for coating are commercially available Molybdenum (Mo) powder with a particle size ranging from 15 µm-40 µm. The substrate material used for the coating was AISI 1020 steel an extensively used material in wide variety of general engineering and construction applications such as pins, chains, shafts, hard wearing surfaces, axles and automobile parts. The AISI 1020 steel sample pins were prepared as per ASTM G99 standard.

2.2. Plasma Spraying

Molybdenum coating with a thickness of 300 µm were deposited on AISI 1020 steel surface by Argon (Ar) + 20 to 25 vol. % Hydrogen (H_2) based atmosphere plasma spraying (ALT-F 3MB) process. The coating powder particles were injected vertically into the plasma stream by argon (Ar) carrier gas for primary flow and hydrogen (H_2) for secondary flow. The plasma spraying was performed with a parameter combination as shown in Table 1. Before spraying, the substrate surface was grit blasted to improve adherence.

TABLE 1

Selected process parameters for plasma spraying

Parameters		**Range**
Plasma system Gun		ALT-F 3MB
Plasma gases	Pressure (N/mm^2) – Argon & Hydrogn	0.689-0.827
	Flow rate (m^3/min) – Argon	2.26-2.54
	Hydrogn	20-25
Power	Current (A)	490
	Voltage (V)	70
Powder feed rate (gms/min)		40-50
Spraying conditions	Nozzle diameter (mm)	8
	Spraying Distance (mm)	75-125

2.3. Laser remelting process

The plasma sprayed Mo coating is a blend of lamellar and half melted or unmelted particles, with certain content of oxide exhibiting porosity. Based on the literature/experimental findings the post treatment of Mo coating was found to be beneficial in improvising the adhesion behaviour of coating on the substrate. Hence, as shown in Fig. 1 the coating was subjected to laser remelting using Nd: YAG Laser. Having flexible laser beam micro processing unit with an inertial Table and positioning system for laser head, a programmable pulsed Nd:YAG laser (Laser Cheval: LEM2 Emeraude) with a maximum average power output of 75W. For all the experiments, the surface protection was assured by argon gas flow, using a laser protection nozzle, fitted off axis to laser beam. The flow rate of argon gas was set to 12 l/min. The laser process parameters used in remelting are shown in Table 2. The surface of coating was protected by using argon gas during Laser remelting. The microstructure of plasma sprayed molybdenum coating before laser remelting and after laser treatment is shown in Fig. 2.

Fig. 1. Laser remelting set up

Fig. 2 (a) shows the exterior surface of the plasma sprayed Mo coating. The plasma sprayed coating surface is illustrated by undulations consisting of unmelted, partially melted and fully melted splats. The surface also comprises a large number of micro pores originating from the spray process. Fig. 2 (b) shows the morphology of laser remelted plasma sprayed Mo coating. Laser remelting of coating helps to reduce the porosity. However, the network of micro cracks was founded on the laser remelted surface of the plasma sprayed coating. The network of micro cracks was generated due to shrinkage and thermal stresses. The shrinkage and thermal stresses crop up during the rapid cooling after laser remelting.

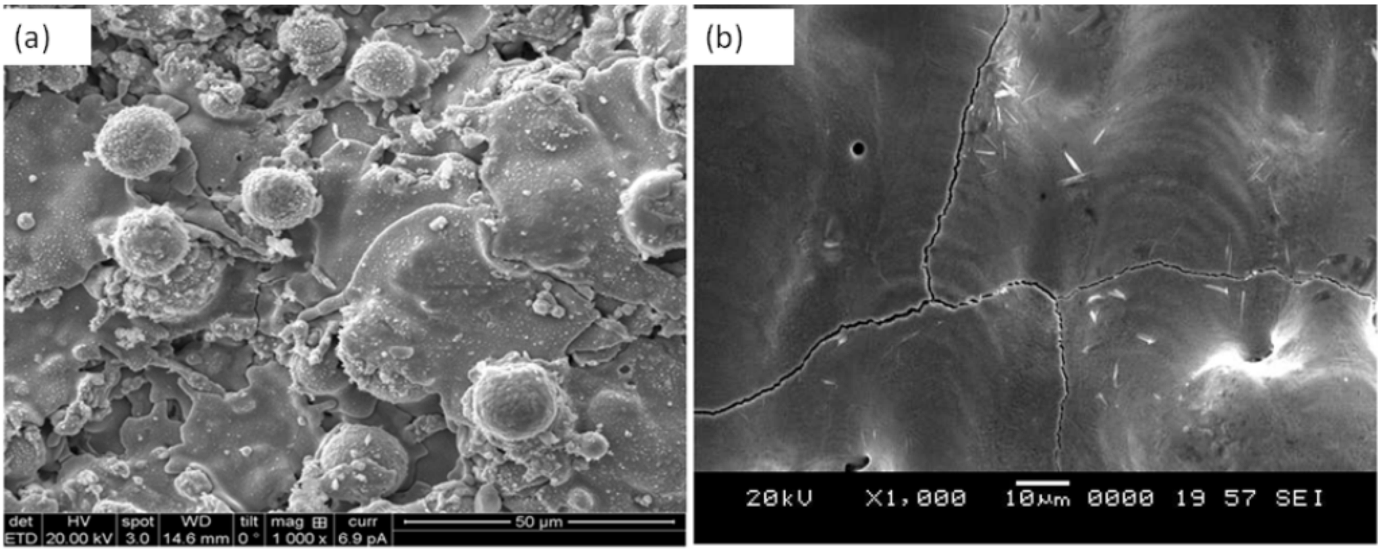

Fig. 2. (a) plasma sprayed molybdenum coating before laser remelting (b) after laser remelting

TABLE 2

Laser remelting process parameters applied for plasma treated Mo coatings

Parameter	Range
Power (W)	75
Laser Scanning Speed (mm/s)	128
Pulse Frequency (khz)	35
Pulse width (µs)	25
Argon gas flow (l/min)	12
Travel speed (mm/sec)	4
Focal setup	Focused laser beam
Wave length (nm)	1060-1070

TABLE 3

List of wear parameters and corresponding levels selected for experimentation

Factors	Notations	Units	Levels				
			Lowest (–1.68)	Low (–1)	Middle (0)	High (+1)	Highest (+1.68)
Load	L	(N)	10	15.60	30	57.63	90
Sliding speed	S	(rpm)	180	250	402	650	900
Sliding distance	D	(m)	2000	2500	3463	4801	6000

2.4. Design of experiments (DOE) and Experimentation

Aspire of present investigation is to analyze the influence of parameters affecting the volume loss of laser remelted plasma sprayed Molybdenum coating. The mathematical modeling offer reliable equations accomplished through the data of suitably designed experiments. The Response Surface Methodology (RSM) based mathematical modeling using design of experiments (DOE) was found to be an effective modeling tool [8,11]. The response surface methodology decreases the cost and time and gives the necessary information about the main and interaction effects of process parameters. Hence, in the current study, the effect of process parameters on response is tested through a set of planned experiments based on Central Composite Design (CCD).

The experiments were performed using pin on disc wear test rig. The range of the process parameters were choosen based on authors' preliminary experiments with five levels for each of the three parameters identified. The wear test parameters and their levels are tabulated in Table 3. The twenty trials based CCD were planned; the layout plan for the current experimental investigations is presented in Table 4. The wear test was conducted as

TABLE 4

Experimental design matrix for wear test

Std	Run	L	S	D	Wear volume loss (mm^3)
9	1	–1.68179	0	0	0.3272
13	2	0	0	–1.68179	0.2156
12	3	0	1.68179	0	0.39
1	4	–1	–1	–1	0.053
7	5	–1	1	1	0.312
15	6	0	0	0	0.3749
8	7	1	1	1	0.75
16	8	0	0	0	0.4241
14	9	0	0	1.68179	0.38
6	10	1	–1	1	0.5078
4	11	1	1	–1	0.73
5	12	–1	–1	1	0.2
20	13	0	0	0	0.39
19	14	0	0	0	0.44673
11	15	0	–1.68179	0	0.0873
17	16	0	0	0	0.419
18	17	0	0	0	0.37101
3	18	–1	1	–1	0.1794
10	19	1.68179	0	0	1.01307
2	20	1	–1	–1	0.4198

per the ASTM G 99 standard. Wear tests were carried out by sliding the coated pin against the EN32 steel disc of hardness 62 HRC, under dry conditions. The experiments were conducted in a random order to prevent systematic errors. To estimate the volume loss of laser remelted coatings, the pin mass was measured before and after the test by using a precision weighing machine with 0.0001g of resolution. The mass loss is converted into wear volume loss. The computed values of volume loss are presented in Table 4.

3. Response Surface Methodology (RSM)

In this study, a response surface methodology was utilised to predict the volume loss of laser remelted plasma sprayed Mo coating.

Generally, it is convenient to represent the independent parameters in quantitative form and the performance criterion in terms of input parameters can be expressed as [11]

$$Y = \phi(x_1, x_2, x_3, ..., x_k) \tag{1}$$

Where Y is the performance criterion, $(x_1, x_2, x_3, ..., x_k)$, are the input factors and ϕ is the response function.

In the current investigation, second order RSM based mathematical models for volume loss have been developed with applied load (L), Sliding speed (S) and Sliding distance (D) as the process parameters.

$$Y = b_0 + b_1 * L + b_2 * S + b_3 * D + b_{11} * L^2 + b_{22} * S^2 + \\ + b_{33} * D^2 + b_{12} * L * S + b_{13} * L * D + b_{23} * S * D \tag{2}$$

Where, Y is response, i.e., volume loss; b_0... b_{23}: regression coefficients of the models are to be determined for the performance criteria.

The values of regression coefficients of the quadratic model are determined by [11]

$$B = (X^T X)^{-1} X^T Y \tag{3}$$

Where, B is the matrix of parameter estimates; X is calculation matrix including linear, quadratic and interaction terms, X^T is transpose of X and Y is the matrix of desired performance criterion.

The developed mathematical model to predict volume loss of laser remelted plasma sprayed molybdenum coating is given by:

$$\text{Volume loss (mm}^3) = 0.40 + 0.21 * \text{L} + 0.095 * S + \\ + 0.049 * D + 0.039 * LS - 0.021 * LD + \\ + 0.092 * L^2 - 0.060 * S^2 - 0.039 * D^2$$

Where, applied load (L) in N; sliding speed (S) in rpm; sliding distance (D) in m; Volume loss in mm^3.

3.1. Examination of data and Adequacy of model

3.1.1. Normality of the data

The Normality of the data is inspected by using a normal probability plot [8]. The normal probability plot of the residuals for volume loss is shown in Fig. 3. The normal probability plot reveals that the residuals tend to fall on the straight line. It indicates that the errors are distributed normally.

3.1.2. Independency of the data

Independency of the data was assessed by plotting a graph between the residuals and the run order [12]. The residual plot for volume loss is as shown in Fig. 4 demonstrating no predictable pattern.

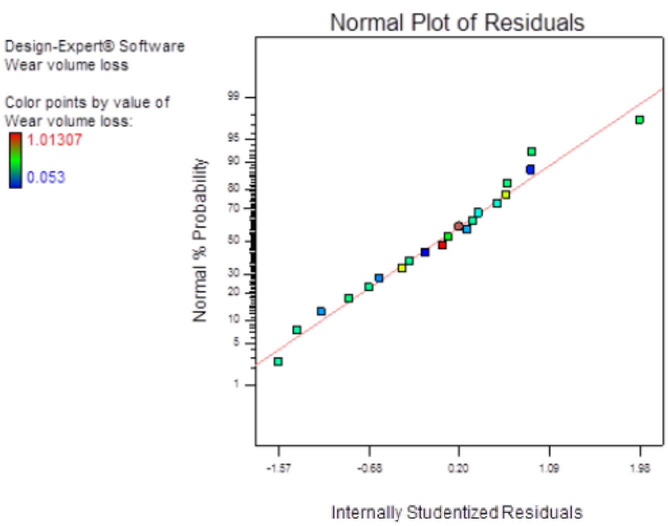

Fig. 3. Normal probability plot for volume loss

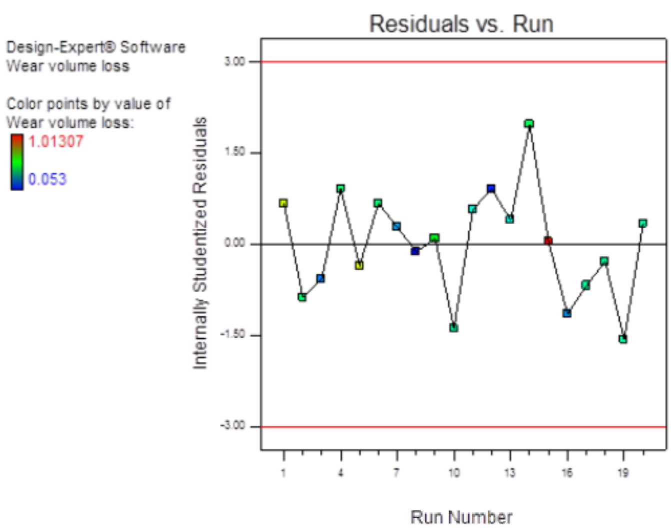

Fig. 4. Residual plots for volume loss

3.2. Analysis of Variance (ANOVA)

In the present study, Analysis of Variance (ANOVA) is used to check competence of the developed empirical relationships. ANOVA test results of the volume loss of laser remelted plasma sprayed Molybdenum coating are presented in Table 5. From the *F* value assessment, it was observed that the principal factors which have direct influence on the response as per hierarchy are applied load, sliding speed and sliding distance respectively. The interaction effect $L \times S$ and $L \times D$ also found to have influence on wear volume loss.

The determination coefficient (R^2) point out the goodness of fit for the model [13]. In this study, the value of the determination coefficient ($R^2 = 99.44\%$) indicates that 0.56% of the total variations are not explained by the empirical relationships. The value of the adjusted determination coefficient (98.94%) is also high; it indicates the high significance of the empirical relationships. The predicted R^2 value (98.58%) also show good agreement with the adjusted R^2 value. The values of probability (p) in Table 5 less than 0.05 indicates that the empirical relationships are significant. Lack of fit is not significant for developed empirical model as it is desired [10]. These results demonstrate that the exceptional potential of the regression model and developed model can be effectively used to predict the wear volume loss of coating.

TABLE 5

ANOVA for wear volume loss of laser remelted Mo coating

Source	Sum of Squares	*df*	Mean Square	*F* Value	*p*-value	Prob >*F*
Model	0.97	9	0.11	197.75	<0.0001	Significant
L	0.58	1	0.58	1062.67	<0.0001	
S	0.12	1	0.12	226.33	<0.0001	
D	0.032	1	0.032	59.07	<0.0001	
LS	0.012	1	0.012	22.55	0.0008	
LD	3.681E-003	1	3.681E-003	6.73	0.0267	
SD	8.487E-004	1	8.487E-004	1.55	0.2412	
L^2	0.12	1	0.12	224.56	<0.0001	
S^2	0.052	1	0.052	95.75	<0.0001	
D^2	0.022	1	0.022	40.83	<0.0001	
Residual	5.466E-003	10	5.466E-004			
Lack of Fit	8.810E-004	5	1.762E-004	0.19	0.9528	not significant
Pure Error	4.586E-003	5	9.171E-004			
Cor Total	0.98	19				

Std. Dev.	0.023	R-Squared	0.9944
Mean	0.40	Adj R-Squared	0.9894
C.V. %	5.85	Pred R-Squared	0.9858
PRESS	0.014	Adeq Precision	57.933

4. Results and discussion

4.1. Perturbation plot

The developed empirical model can be used to study the effect of load, sliding speed and sliding distance on the dry sliding wear behaviour. Based on this empirical model, the main and interaction effects of the process parameters on the volume loss was calculated and represented in the form of perturbation plots as shown in Fig. 5.

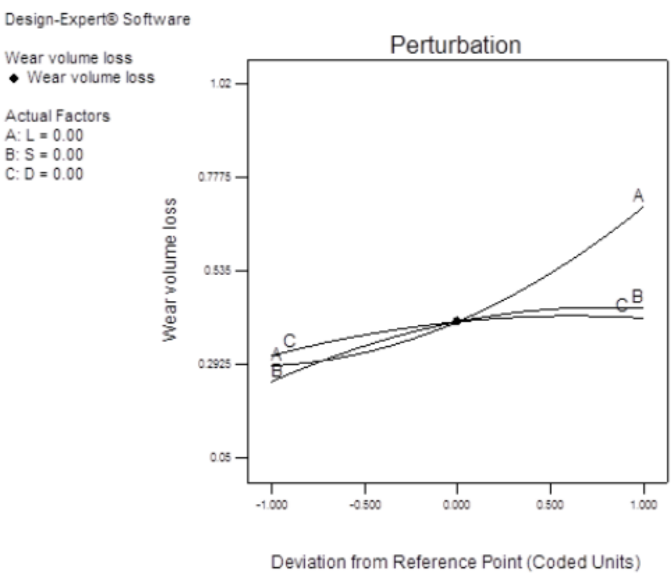

Fig. 5. Perturbation plot

The perturbation plot demonstrates how the response varies as each factor shifts from the selected reference point, while all other factors remain constant at the reference value. Generally, Design-Expert 9 software sets the reference point at the middle of the design space (coded zero level). A sharp slope or curve is observed for the load factor showing highly responsive character in perturbation plot. Compared to load factor, speed and distance factor has less steep, which are also the next important parameters influencing the volume loss. A comparatively flat line demonstrates insensitivity to change in that particular factor [8,10]. It is clear from Fig. 5 that the volume loss increases with increase in the levels of factors. The peak of the perturbation plot illustrates the maximum volume loss. This plot can be used in the prediction of the response for any region of the experimental domain.

The effect of load on the volume loss of the laser remelted plasma sprayed coating is shown in Fig. 5. The applied load is the main noticeable parameter that can affect wear. The volume loss values of the laser remelted plasma sprayed coating increased with increase in applied load. The magnitude of load is significant since it increases both the area of contact and shear stress as well as elastic or plastic deformation state [14,15].

The SEM images of the worn surface of coating under different loading conditions are shown in Fig. 6. In case of low load condition, formation of grooves and micro cracks were seen (Fig. 6 (a)). The area of contact at the asperity junctions is low and hence there is an elastic contact between the pin and the disc. Thus, the coating supports the applied load and causes for less material loss. As the applied load increases, the volume loss increased due to the formation of fatigue cracks on the coating surface resulting in the highest removal of the molybdenum lamellae. The lamellae, which cracked during the earlier wear stages, could be easily removed. In reality, the high loads may encourage de-cohesion of lamellar interlinked splats and detachment of splats from the coating is observed in Fig. 6(b). The splat detachment generally occurs in plasma sprayed coatings, where the inter-splat cohesion is very weak [7,16]. Furthermore, the abrasive action of the detached particles entrapped in the contact zone may considerably contribute to the material loss [15]. Further increase in material loss occurs at higher loads which may be due to drastic propagation of subsurface cracks by tensile and shear stresses in the contact area, consequential in the delamination of coating [12].

The effect of speed on the volume loss of the coatings is illustrated in Fig. 5. It could be confirmed from the Fig. 5 that with increase in sliding speed, the volume loss increases. The above statement could be defensible by tangential impact wear phenomenon, which states that when two sliding surfaces are in contact, interlocking in the sliding direction may occur between the asperities of the two sliding surfaces [12,17]. Actually, the sliding of the two surfaces is a discontinuous and instantaneous process. Two types of consequences exist, depending on sliding speed. In the case of low sliding speed, plastic flow may happen and no brittle fracture occurr to the asperity. When the sliding speed is high, the tangential impact effect will create an exceptionally high stress inside the asperity and the asperity will be fractured as wear debris. The impact stress in the asperity and fracture rate of asperity will be proportional to the sliding speed.

The worn surfaces of the coatings tested under different speeds are shown in Fig. 7. From Fig. 7(a) and (b), it can be seen that a mild wear volume loss with fine scratches and grooves can be found on the worn surface at lower sliding speed. The Fig. 7(a) shows the formation of micro cracks and the traces of tribofilm on the worn surface of coating. At higher sliding speeds a sharp thermal gradient develops due to high frictional heat and low thermal conductivity of coating. Thus, the thermal stresses

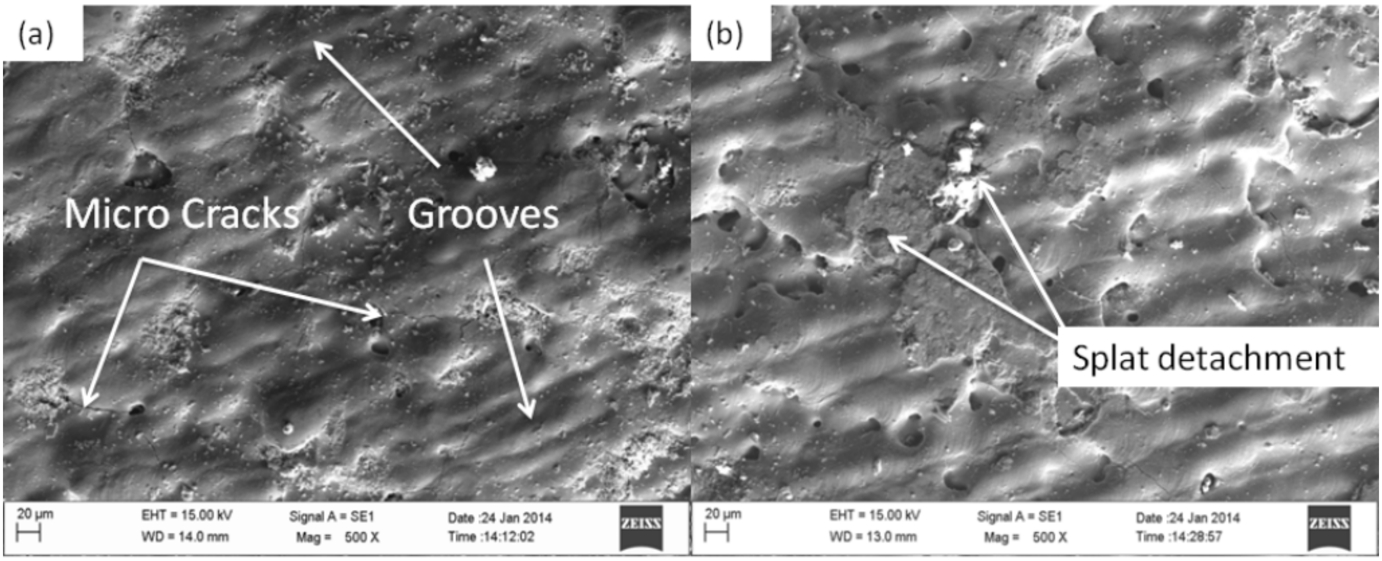

Fig. 6. Worn surfaces of laser remelted coatings subjected to different loads. (a) Lower load-10N, (b) Higher load – 90N

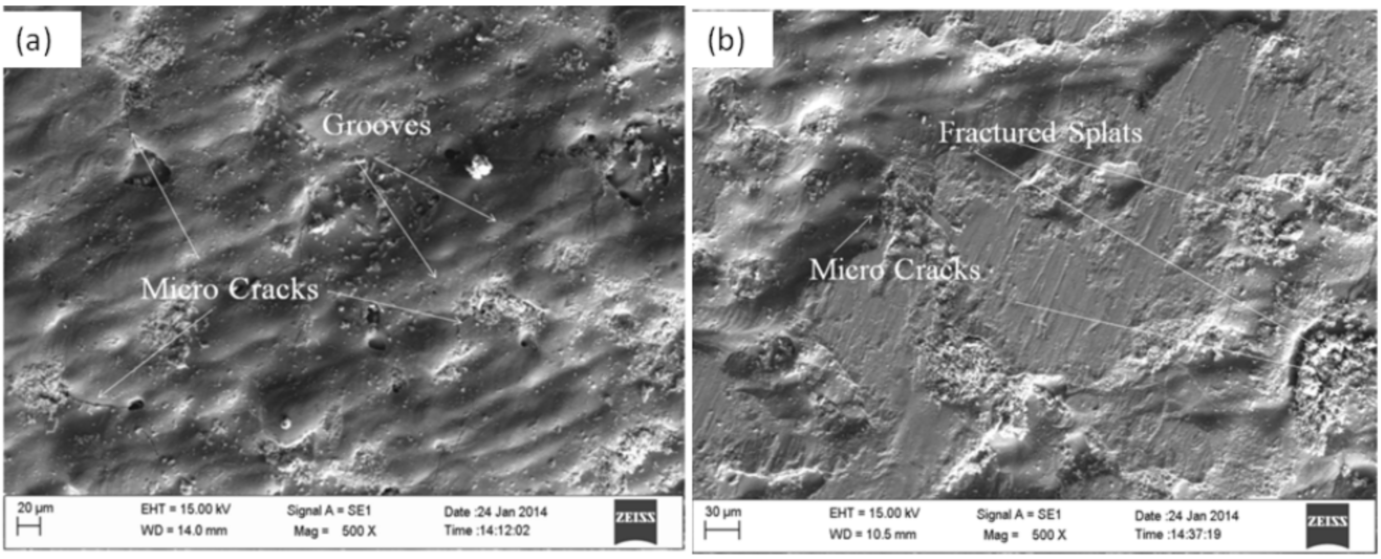

Fig. 7. Worn surfaces of coatings under different sliding speeds

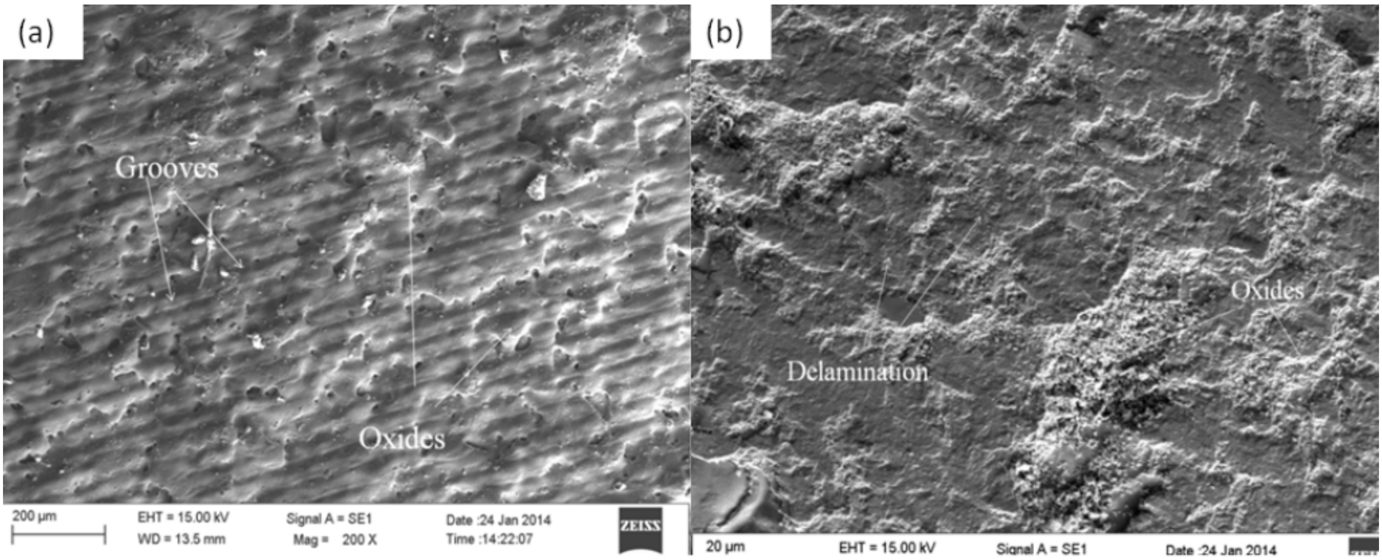

Fig. 8. Worn surface of coatings under different sliding distances

in the surface layer of coating increases and leads to formation of micro cracks [18]. The micro cracking increases with increase in sliding speed. The combination of cracks, started at different depths ultimately reach the surface to cause de-lamination of the coating [5,19]. It is essential to note that, propagation of the micro cracks is accountable for the hasty removal of the coating material. Fig. 7(b) shows, at higher sliding speed, the wear volume is more intense. Pulling-out and fragmentation of the molybdenum splats were also observed, which leads to the rigorous wear.

The effect of sliding distance on volume is shown in Fig. 5. It is concluded from the Fig. 5 that the volume loss of laser treated plasma sprayed coating increases with increase in sliding distance. Usmani and Sampath, [19] and Khedkar et al., [5] also observed that material loss of the sprayed coating increases with increase in sliding distance. Fig. 8 shows the worn surface of laser remelted plasma sprayed coating for different sliding distances. There is a formation of groove running parallel to the sliding direction. This feature illustrates a distinctive adhesive wear. In this study, the hard particles entrapped between the coated pin and disc, plough the coated pin, causing wear volume loss by the elimination of the small chunks of material causes abrasion wear. These findings concurs with the previous studies [3,6,19]. The worn surface discloses that the surfaces are covered extensively by oxide. The oxidation of the coating surface occurs due to frictional heating. The volume loss of coating is mild at lower sliding distance due to oxide layer acting as a protective layer [12]. As the sliding distance increases there is a gradual changeover in the wear mechanism occurred from the oxidation to delamination wear as shown in Fig. 8 (b). Due to this, an increment in the volume loss was observed at higher sliding distance.

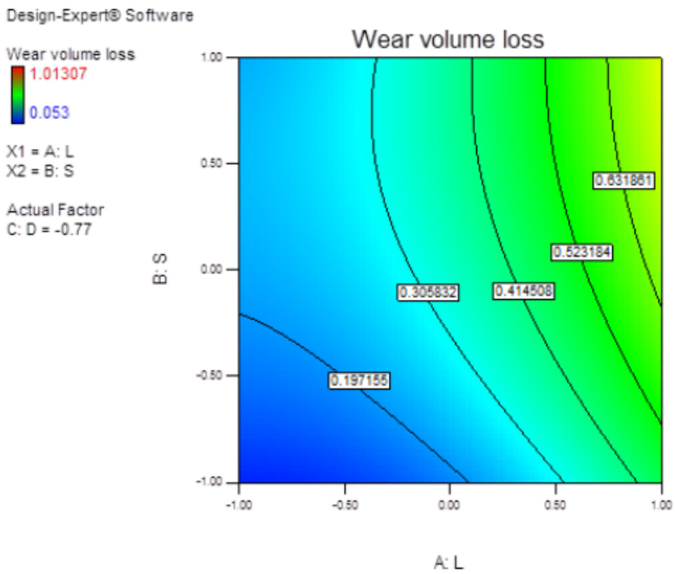

Fig. 9. 2D contour plot for Variation of volume loss with Load and sliding speed

that the volume loss of laser remelted plasma sprayed coating increases with simultaneous increase of applied load and sliding speed. As the load and speed increases, the surface contact area and interface temperature between pin and disc also increases. This is responsible for the formation of cracks, lamellae detachment and fracture of splats. For a constant load as the increase in speed the increase in volume loss is minimal. But for higher load (90 N) the effect of sliding speed is negligible and the volume loss is very high. The interaction exists only at lower values of load and sliding speed.

4.2. Interaction of load and speed

The load and sliding speed interaction also influences the wear volume loss as shown in Fig. 9. The Contour plot confirms

4.3. Interaction of Load and sliding distance

Fig. 10 shows the interaction effect of load and sliding distance on volume loss. 2D contour plot substantiates that the volume loss of laser remelted plasma sprayed coating increases with increase in applied load and sliding distance. It is also noticed from the plot that beyond a load of 56.71N and sliding distance of 4300 m, there is no interaction between the load and sliding distance. The increased volume loss occurs mainly due to increase in applied load irrespective of sliding distance. The increased volume loss was observed due to the reason that the surface contact area and the interface temperature between pin and disc also increases. This leads to the formation of cracks, fracture of oxide film, lamellae detachment and fracture of splats.

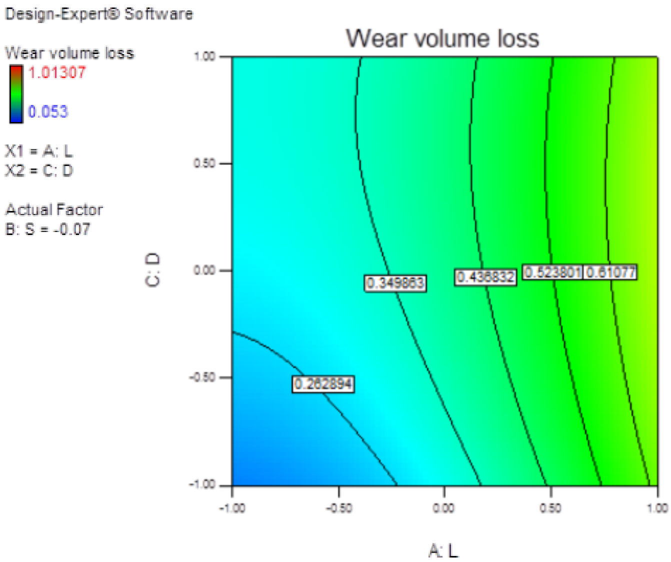

Fig. 10. 2D plot for Variation in volume loss with load and sliding distance

4.4. Validation

From Fig. 11 it is observed that, the predicted values are well fitted with in the actual value. It shows that the model can be used for the prediction of wear volume loss within the experimental domain.

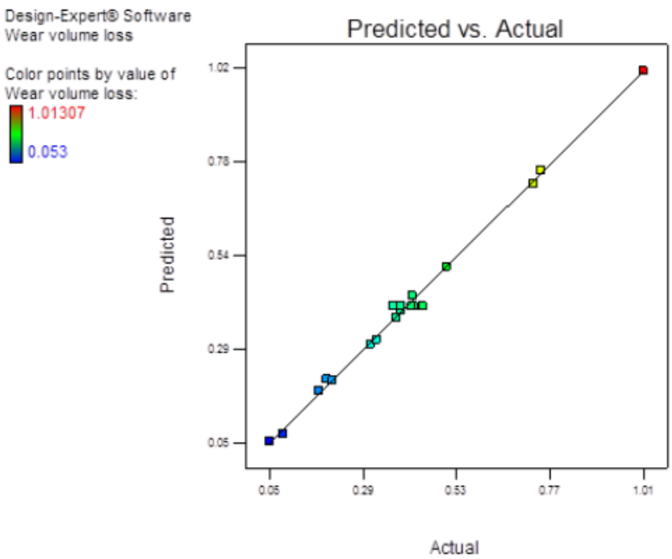

Fig. 11. Relationship between experimental and predicted values

4.5. Comparison between wear behaviour laser remelted and plasma treated

Fig. 12 illustrates the comparison of wear volume loss of plasma sprayed and laser remelted Mo coating. It shows that, the laser remelted Mo coated samples has greater wear resistance compared to plasma sprayed Mo coating. This is because, the laser remelting helps to fill the porosity present in the plasma coating and melts appropriately the deposited Mo material on substrate surface. The adhesion strength of coating is also enhanced by laser remelting which lead to lower wear volume loss. The laser remelting provides dense and pore free microstructure. The better tribological performance of laser remelted plasma spray coating was basically due to the presence of metallurgical bond between the splats. The plasma sprayed coatings usually fail along the interface of the splats. Certainly, the temperature gradient builts up between the substrate and the coating as the sliding speed increases causing stress development. The stress development in the coating weakens the interfacial bonding between splats and leads to failure of coating. This leads to increase in wear volume loss of plasma sprayed coating compared to laser remleting.

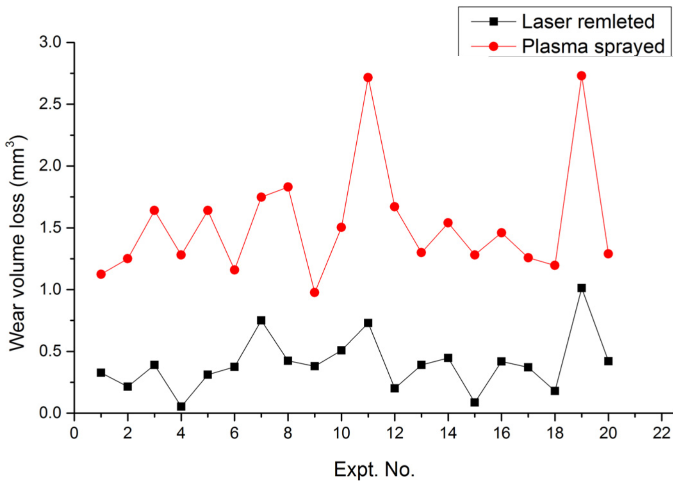

Fig. 12. Comparison of wear volume loss of laser remelted and plasma treated Mo coating

5. Conclusions

In the present experimental study, volume loss of laser remelted plasma sprayed molybdenum coating was analyzed through response surface modeling. The applied load, sliding speed and sliding distance have been identified as the process parameters and central composite design (CCD) was used to conduct the experiments. Based on the statistical analysis, the following conclusions are drawn within the range of the parameters selected:

- Empirical relationships were established using RSM to predict the volume loss of the laser treated pins.
- The applied load was the most major factor affecting the volume loss of the coatings. The sliding speed and sliding

distance are the next most important parameters influencing the volume loss.

- The volume loss of coating was also affected by the interaction of sliding speed and sliding distance. The interaction exists only at lower values of load and sliding distance.
- The interaction of load and speed affects the volume loss of laser remelted plasma sprayed coating. No interaction between load and sliding speed at higher values were observed. The load plays vital role on volume loss.
- The wear behaviour of the laser remelted plasma sprayed molybdenum coating was found to be dependent on two mechanisms, one being the formation of grooves, surface tribo films and the other being fracture of splats and delamination of the coating.
- It should be accentuated that, the range selected for parameters, the results and the conclusions refer specifically to the Pin on Disc test setup and the EN32 steel counter body material used in the present study. However, the demonstrated approach and the methodology of the response surfaces are common.
- The laser remleted has better wear resistance than the plasma sprayed Mo coating due to improved adhesion strength of the coating on the substrate and dense pore free microstructure.

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