



# Microstructure and tribological behavior of plasma sprayed NiCrAlY/WC-Co/cenosphere/solid lubricants composite coatings

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## ABSTRACT

Present investigation deal with NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaF<sub>2</sub>, NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaSO<sub>4</sub> and NiCrAlY/WC-Co/Cenosphere coatings deposited on MDN 321 steel using atmospheric plasma spraying. Tribological properties of MDN 321 steel and coatings are evaluated from room temperature (RT) to 600 °C under dry lubrication conditions using a pin on disc high-temperature tribometer. Scanning Electron Microscopy (SEM), X-ray diffraction (XRD) and Energy Dispersive Spectroscopy (EDS) are used to characterize the coatings. Presence of cenospheres in these coatings might effectively reduce wear acting as localized regions accumulating wear debris. The result shows that wear rate of all the coatings are lower as compared to MDN 321 substrate at all the test conditions. NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaF<sub>2</sub> and NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaSO<sub>4</sub> coatings registered lower friction coefficient as compared to NiCrAlY/WC-Co/Cenosphere coating and MDN 321 substrate. Characterization of the NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaF<sub>2</sub> and NiCrAlY/WC-Co/Cenosphere/MoS<sub>2</sub>/CaSO<sub>4</sub> coatings worn out surface suggests that MoS<sub>2</sub> provides lubrication at 200 °C and formation of CaMoO<sub>4</sub>, MoO<sub>3</sub> through tribo chemistry reaction at higher temperature provides lubrication at 600 °C. SEM micrograph of worn surface demonstrates that the main wear mechanism is plowing and delamination.

## 1. Introduction

Components working at higher operating temperatures like in gas and steam turbines, propulsion bearings, air foil bearings and internal combustion engines are subjected to surface friction and wear [1–4]. Solid lubricant coatings are effective in minimizing friction and wear under harsh environments like high temperature, vacuum and high humidity where conventional lubricants such as oil and grease fail to sustain their integrity [5–7]. Commonly available solid lubricants are transition metal disulfides (MoS<sub>2</sub>, WS<sub>2</sub>, TaS<sub>2</sub>), soft noble metals (Au, Ag), inorganic fluorides (CaF<sub>2</sub>, BaF<sub>2</sub>, etc.) and metal oxides (NiO, Cr<sub>2</sub>O<sub>3</sub>, etc.) [8,9]. However, the sole lubricant may not provide the lubrication over a wide range of operating temperatures. For example, MoS<sub>2</sub> loses its lubricity above 300 °C owing to oxidation and cannot be used for high-temperature applications. CaF<sub>2</sub> and BaF<sub>2</sub> are effective at elevated temperature (> 500 °C) due to their low shear strength at higher temperature [10]. Many researches have incorporated these low and high-temperature solid lubricants in the coatings to achieve the synergetic effect over wide temperature range [8,11–13].

Plasma sprayed NiCrAlY coatings are widely used in the higher

temperature applications to combat the effects of oxidation, corrosion and wear resistance. However, these coatings find limitations in industries due to lower hardness as compared to carbides, ceramics, and oxides [14–16]. Nevertheless, wear resistance of these coatings can be significantly improved by reinforcing the hard phases like WC, Al<sub>2</sub>O<sub>3</sub>, Cr<sub>3</sub>C<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> etc. [17]. WC-Co possesses higher hardness, better ductility, chemical inertness and lower frictional values as compared to Cr<sub>3</sub>C<sub>2</sub> [18]. These properties of WC-Co improve the wear resistance of NiCrAlY coating when reinforced as a hard phase. Further, WC-Co coatings possess high frictional values at higher temperature necessitating the incorporation of solid lubricants in the coating. Chen et al. [1] studied the friction and wear behavior of plasma sprayed NiCrAlY-Ag-Mo coating at different temperatures and reported that, the addition of Ag and Mo decreased the friction coefficient and wear rate. Joel and Basil [19] investigated the WC-10Ni/MoS<sub>2</sub>/WS<sub>2</sub> coating by HVOF and they found 50% reduction in mass loss of the coating as compared to WC-10Ni. Bin C et al. [20] studied the tribological behavior of graphite/CaF<sub>2</sub>/TiC/Ni-base alloy coating by plasma spray and reported that friction coefficient and wear rate are reduced by 25.9–53% and 18.6–70.1% compared to Ni base alloy coating respectively. However,

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NiCrAlY and WC-Co powders are the quite expensive demanding development of low cost cenosphere coatings. Incorporation of the inexpensive hollow cenospheres in the coatings might effectively reduce the wear by opening up the built-in porosity which can act as localized regions for wear debris accumulation. Elemental constituents of cenospheres can reduce wear further.

Fly ash cenospheres are industrial waste from thermal power plants, generated due to combustion of coal. These fly ash cenospheres are spherical in shape, readily available in powder form, inexpensive and possess superior mechanical properties [21–26]. The main constituents of fly ash cenospheres are oxides of silicon ( $\text{SiO}_2$ ), aluminium ( $\text{Al}_2\text{O}_3$ ), iron ( $\text{Fe}_2\text{O}_3$ ) and mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) [27–31]. Among them, aluminium oxide and mullite possess high-temperature stability, wear, erosion, and corrosion resistance [32,33]. These properties can be well exploited by incorporating fly ash cenospheres in the coatings. Some researchers have developed the coatings using fly ash and their study reveals that fly ash can be effectively used as feedstock for plasma spray coatings [34,35]. Rama Krishna et al. [36] investigated the wear response of detonation sprayed fly ash coating on mild steel. They found that fly ash coating has a lower coefficient of friction compared to mild steel. Sidhu et al. [37] studied the wear, oxidation and salt corrosion behavior of fly ash coating deposited using plasma spray. They reported better oxidation and salt corrosion resistance of fly ash coating as compared to carbon steel substrate. Behera and Mishra [38] investigated the fly ash composite coating deposited on the copper substrate by using plasma spray technique and results reveal better interfacial adhesion between substrate and coating. Available literature is relatively scarce on the usage of fly ash composite coatings using solid lubricants for high temperature wear resistance applications. This fact necessitates the study of elevated temperature wear behavior of composite coatings containing cenospheres and solid lubricants. Furthermore, understanding the wear mechanism of metallic matrix coatings with hollow cenospheres is an interesting and worth investigating task. Usage of such environmental pollutants in coatings might further reduce landfill burden and if developed successfully are eco-friendly as well.

In the present work, NiCrAlY-WC-Co/Cenosphere/ $\text{MoS}_2$ / $\text{CaF}_2$  (designated as NiCaF<sub>2</sub>), NiCrAlY-WC-Co/Cenosphere/ $\text{MoS}_2$ / $\text{CaSO}_4$  (designated as NiCaSO<sub>4</sub>) and NiCrAlY-WC-Co/Cenosphere (designated as Ni-WC) coatings are deposited on MDN 321 steel using plasma spray process. Sliding wear behavior of MDN 321 steel and coatings are evaluated at room temperature (RT), 200, 400 and 600 °C using a pin on disc high-temperature tribometer. The worn surfaces are analyzed and discussed in detail.

## 2. Materials and methods

### 2.1. Materials

MDN 321 steel is used as a substrate which is procured from Mishra Dhatu Nigam Ltd., Hyderabad, India, having chemical composition is Fe-0.1C-1.46Mn-18.13Cr-10.36Ni-0.62Ti-0.55Si (wt%). The substrate is trimmed to the dimension of  $12 \times 12 \times 4$  mm using a diamond saw prior to plasma spraying. Commercially available gas atomized NiCrAlY, agglomerated and sintered WC-Co (Powder Alloy Corporation, USA), fly ash cenospheres (Cenospheres India Pvt. Ltd., Kolkata, India) and laboratory reagent grade  $\text{MoS}_2$ ,  $\text{CaF}_2$  and  $\text{CaSO}_4$  (Loba Chemie Pvt. Ltd., Mumbai, India) solid lubricants are utilized to prepare coating feedstock. Laser diffraction technique (Cilas, 1064, France) is used to measure the nominal size distribution of the powders and is reported in Table 1. These powders are blended mechanically with predetermined mass fraction before getting sprayed. Compositions of the coatings are listed in Table 2. Fig. 1a and b shows the SEM (JSM 6380LA, JEOL, JAPAN) micrographs of the blended NiCaF<sub>2</sub> and NiCaSO<sub>4</sub> mix respectively. It is observed from the micrographs that all the powders are mixed uniformly. NiCrAlY, WC-Co, and cenospheres are in spherical

**Table 1**

Particle size ( $\mu\text{m}$ ) distribution of powders.

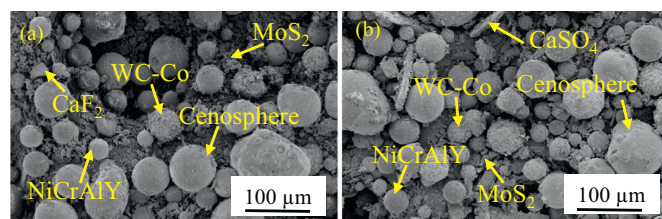
Particle size	NiCrAlY	WC-Co	Cenospheres <sup>a</sup>	$\text{MoS}_2$	$\text{CaF}_2$	$\text{CaSO}_4$
D (0.1)	41.88	23.52	–	0.63	4.27	8.72
D (0.5)	70.75	38.78	–	5.95	57.52	63.79
D (0.9)	120.33	64.11	–	29.45	115.52	132.57
0–10%	–	–	106	–	–	–
70–90%	–	–	63	–	–	–
0–30%	–	–	53	–	–	–
Mean	76.54	41.71	65	10.62	60.30	70.00

<sup>a</sup> As specified by Cenosphere India Pvt. Ltd., Kolkata, India.

**Table 2**

Composition of coatings (wt%).

Coatings	NiCrAlY	WC/Co	Cenospheres	$\text{MoS}_2$	$\text{CaF}_2$	$\text{CaSO}_4$
NiCaF <sub>2</sub>	37.5	12.5	30	10	10	–
NiCaSO <sub>4</sub>	37.5	12.5	30	10	–	10
Ni-WC	52.5	17.5	30	–	–	–



**Fig. 1.** Morphology of as blended (a) NiCaF<sub>2</sub> and (b) NiCaSO<sub>4</sub> powders.

shape as shown in Fig. 1 and  $\text{MoS}_2$ ,  $\text{CaF}_2$  and  $\text{CaSO}_4$  are irregular in shape.

### 2.2. Coating deposition and characterization

Atmospheric plasma spray technique is used to deposit NiCaF<sub>2</sub> and NiCaSO<sub>4</sub> blend using METCO USA 3 MB equipment (M/s. Spraymet Surface Technologies Pvt. Ltd., Bangalore, India). Substrates are grit blasted using alumina powder of 150  $\mu\text{m}$  size before spraying to ensure better adhesion between the coating and substrate. NiCr bond coat is applied on the substrate prior to the spraying of NiCaF<sub>2</sub> and NiCaSO<sub>4</sub> coatings. Details of spray parameters are listed in Table 3. During spraying blended powder is loaded into the hopper which flows through the feeder and gets mixed with argon gas flowing from the compressor at a predefined pressure. This mixture flows towards the plasma stream and gets deposited on to the substrate. Deposition efficiency of coatings is about 40–45% and deposition per pass is in the range of 12–15  $\mu\text{m}$ . SEM is used to measure the thickness of the developed coatings. Porosity of coatings is computed using optical microscope supported with biovis image analyzer (ARTRAY, AT 130, JAPAN). Twenty field of views are analyzed and average values are reported. XRD (DX GE-2P, JEOL, Japan) is employed to analyse the phases in blended powder and

**Table 3**

Plasma spray process parameters [41].<sup>a</sup>

Argon	Pressure	0.75 MPa
	Flow	40 lpm
Hydrogen	Pressure	0.35 MPa
	Flow	7 lpm
Current		490 A
Voltage		60 V
Powder feed rate		60 g/min
Stand of distance		100–125 mm

<sup>a</sup> As provided by Spraymet Surface Technology Pvt. Ltd., Bangalore, India.

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