



Abstract: Environment barrier coatings (EBCs) have been developed to protect metallic and Si-based ceramic components of aerospace structures and turbine blades working under elevated temperatures and chemically harsh environment. Mullite-alumina based coatings were deposited on low carbon steel by a relatively simple and low-cost surface deposition technique based on the slurry spray technique (SST) developed for environmental barrier coating applications. Corrosion test is conducted with a Corrosive medium of 6 M Na_2SO_4 solution prepared by adding 16.8g of Na_2SO_4 powder in 100 ml of pure water, magnetic stirred for 30 min. It has been found that the developed coatings using SST demonstrate dense coating deposits with significantly less corrosion rate than that uncoated low carbon which is comparable with that produced from traditional techniques.

Key Words: slurry spray technique, corrosion rate, Environment barrier coating

Introduction

Environmental barrier coatings (EBCs) are fabricated to improve the stability and service life of the base material in destructive environments. Due to the superior properties exhibited at higher temperatures and in chemically severe environments, nonoxide and oxide ceramics are favorable materials to improve the resistance against degradation like corrosion and oxidation of metals (1). The application of EBCs can significantly increase the operating temperatures up to 1400-1500°C, increase efficiency and improve the durability of the components such as in oxidizing environment (2-4). There are many applications which have benefited from adopting EBCs including the aeronautical, aerospace, nuclear and automotive industries including heavy duty utilities such as diesel trucks (6-7).

The Slurry Spray technique for fabrication environmental barrier coating opts traditional wet powder spraying methods to deposit feasible coating materials onto target substrates. The process involves suspending the coating material within a fluid to form a slurry mixture that can be applied to a surface using common gravity fed air pressurized spray guns. Successive layers are then sprayed onto the substrate and dried using varying slurry compositions to produce a functional coating (5,8). After the desirable number of layers of the EBC is deposited the multilayered coating is loaded in a compression chamber to form a densified layer before being sintered in the furnace.

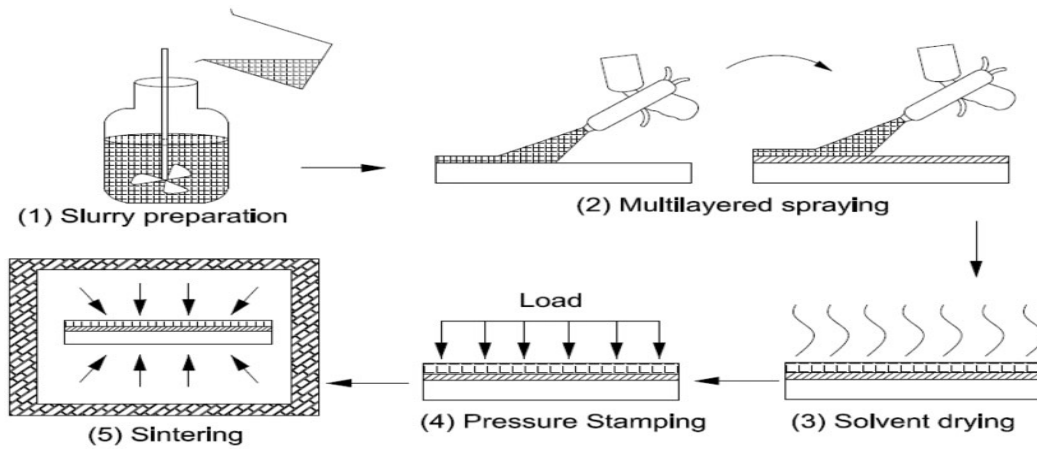


Figure 1. Stages in slurry spray technique (5)

The applied pressure varies depending on the number of coating layers, typically between 10 and 40 MPa (5). Coating fabrication in SST consists of the stages like slurry preparation, multilayered spraying, solvent drying, pressure stamping, sintering (Figure 1).

Corrosion test:

Corrosion test is conducted on the 25X25mm mild steel samples coated by mullite based coating using slurry spray technique (SST) by subjecting these samples in corrosive medium for 30 days. Corrosive medium is 6 M Na₂SO₄ solution prepared by adding 16.8g of Na₂SO₄ powder in 100 ml of pure water . This mixture is rotated in magnetic stirrer for 30min. using a magnetic bid. After preparation of this solution different samples are subjected in solution poured in 100 ml glass vessel. Surface macrographs of the slurry spray coated mild steel samples subjected to corrosion in (0.6 M Na₂SO₄) for 30 days have been presented in figure 4.8(a-j). Weight loss per sample is measured daily and corrosion rate is calculated in mils per year (mpy).The corrosion rates are calculated using the loss in mass of a specimen. The density of the metal, the area of the test specimen and duration of the test are taken into account when converting mass loss into corrosion rates. The expression mils per year (mpy) is the most desirable way of expressing corrosion rates. The expression is readily calculated from weight loss of the metal specimen during the corrosion test by the formula (9) given below .

$$\text{Corrosion rate in (mpy)} = \frac{534 W}{DAT}$$

where, W = weight loss, mg

D = density of specimen, gm/cm³

A = area of specimen, sq. inch.

T = exposure time, hrs

Also the Inhibition Efficiency (%IE) of the coated specimen was evaluated using the following equation(10):

$$(\%IE) = \frac{(CR_u - CR_c)}{CR_u} \times 100$$

Where, CR_u is the corrosion rate of mild steel substrate in absence of coating and CR_c is corrosion rate of mild steel in presence of coating. Values of corrosion rate and %IE is given in table 3.5 at different temperature, sintering time and % additives.

Table 5.4 Corrosion rate and % IE at different temperature, sintering time and % additives.

Sr. No.	Material Type	Sintering Temp. (°C)	Sintering Time (min.)	Additive (%)	Corrosion rate(mpy)	Inhibition efficiency(%IE)
1.	Mild Steel	850	15	1	247	96
2.	---do---	850	30	3	3.811	94.55
3.	---do---	850	45	5	9.375	86.60
4.	---do---	950	15	3	4.533	93.52
5.	---do---	950	30	5	3.5028	94.99
6.	---do---	950	45	1	1.7514	97.49
7.	---do---	1050	15	5	10.40	85.14
8.	---do---	1050	30	1	4.7391	93.22
9.	---do---	1050	45	3	7.1086	89.84
Uncoated	---do---	-	-	-	70	

From the table 5.4 we can conclude that corrosion rate of mullite based slurry coated mild steel is very less in comparison to uncoated mild steel and corrosion rate of coated mild steel is highest and % IE is lowest at 850°C, 45 min. sintering time and 5% additives. While corrosion rate is lowest and %IE is highest at 950°C, 45 min. and 1% additives

Table 5.5 Weight loss/area(mg/cm²) during 30 days.

S.NO	S-1 (mg/cm ²)	S-2 (mg/cm ²)	S-3 (mg/cm ²)	S-4 (mg/cm ²)	S-5 (mg/cm ²)	S-6 (mg/cm ²)	S-7 (mg/cm ²)	S-8 (mg/cm ²)	S-9 (mg/cm ²)	uncoated (mg/cm ²)
1	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0
3	0.16	0.32	0.16	0.32	0	0.16	0.32	0.32	0.64	32
4	0.16	0.32	0.32	0.48	0	0.16	0.32	0.48	0.64	107.52
5	0.64	0.48	0.86	0.48	0.16	0.32	0.86	0.48	0.8	80
6	0.8	0.8	1.6	0.8	0.48	0.32	1.6	0.8	1.12	16
7	0.8	1.12	1.6	1.12	0.48	0.16	2.4	0.8	1.6	0.8
8	1.6	2.4	3.8	3.2	0.52	0.32	9.92	1.12	1.6	0.64
9	1.92	3.2	9.92	3.36	0.64	0.32	11.52	1.28	2.4	0.32
10	1.44	3.52	3.52	0.32	0.8	0.32	8.3	1.12	3.5	0.32
11	1.12	2.08	2.72	0.8	1.44	1.44	3.52	1.6	1.44	0.48
12	0.96	1.44	1.44	0.8	0.8	0.8	1.12	0.48	0.8	0.8
13	0.8	0.8	0.86	0.86	1.6	0.86	0.8	0.8	1.12	0.48
14	0.64	0.96	2.08	0.96	2.08	1.44	0.48	1.12	5.92	0.32

15	0.48	1.6	1.8	0.8	1.44	1.8	0.96	0.64	0.48	0.64
16	0.48	0.8	1.44	0.8	1.44	0.8	1.12	0.32	0.64	0.48
17	0	1.12	1.28	1.12	0.32	1.44	0.96	1.44	0.32	0.32
18	0.16	0.8	1.44	0	1.44	0.32	1.44	1.12	2.4	0.16
19	0.64	0.64	0.8	0.32	0.16	0.64	0.64	1.28	0.8	0.48
20	0.48	0.8	0.32	0.48	0.32	0.96	0.48	0.8	0.42	0.8
21	0.32	0.48	0.8	0.64	0.8	0.8	0.32	2.4	0.32	0.48
22	0.16	0.32	0.96	0.16	0.32	0.64	0.16	1.12	0.64	0.32
23	0	2.4	0.8	1.12	0.32	0.8	1.21	2.88	0.32	0.48
24	0.16	1.52	0.8	1.28	0.32	0.96	2.4	0.64	0.64	0.16
25	0.32	1.6	0.96	0.64	0.08	2.58	2.3	0.32	0.32	0.32
26	0.16	0.8	1.44	0.64	0.16	0.96	0.64	1.44	0.8	0.16
27	0.24	0.6	0.96	0.32	0.16	1.44	1.4	0.64	1.6	0.16
28	0.32	0.48	0.96	0	0.16	1.44	1.504	0.64	0.48	0
29	0.48	0.64	0.7	0.8	1.6	1.12	0.8	0.8	1.44	0.16
30	0.96	0.8	0.8	1.6	3.52	0.32	0.64	1.44	1.92	0.32

On the other hand we also study the weight loss per unit area (mg/cm^2) of each sample during all 30 days. Figure 4.12 shows the condition of corrosion samples at different days, 1st day, 15th day and 30th day. Two samples of each composition as per L9 Taguchi table has been investigated and average has been taken for calculations. Weight loss is calculated per day by subtracting weight gain of consecutive two days. Weight loss/area for each sample is shown in table 5.5.

Figure 5.1 shows the graph of Weight loss per unit area with regarding 30 days of month at different temperature. According to graph weight loss/area value of coated sample with respect to uncoated sample is less and less varies with respect of the days. In the start of experiment wt/area value sharply increase ($107 \text{ mg}/\text{cm}^2$ for the 4th day) for the uncoated sample, but as the days pass value of wt/area is decrease in comparison to 4th day. While or coating samples values does not differ so much and value lies approximately in between 0,1 and 2.88.

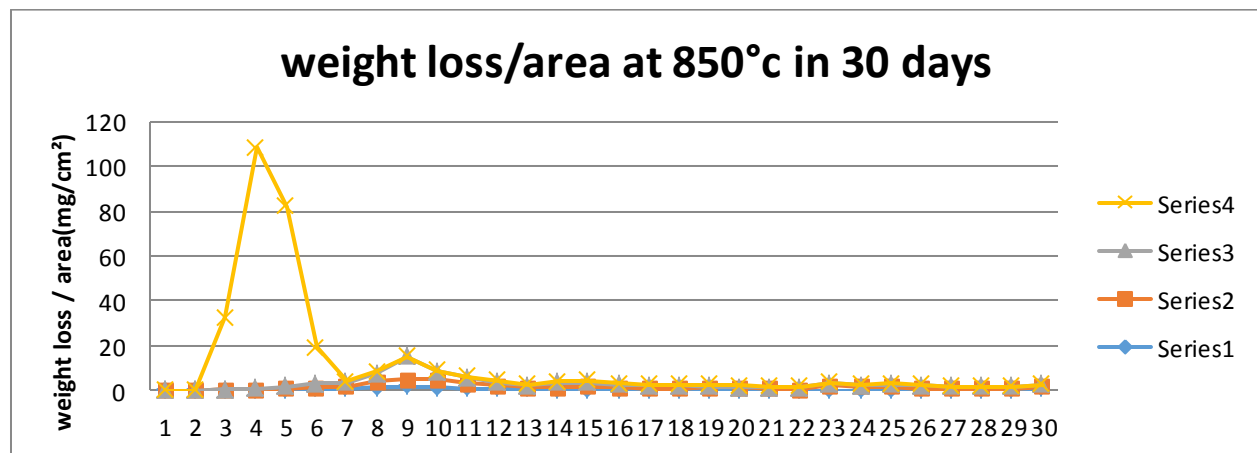


Figure 5.1 shows the graph of Weight loss per unit area with regarding 30 days of month at 850°C temperature. where series 4 refers to uncoated and series 1, 2, 3 are for coated samples.

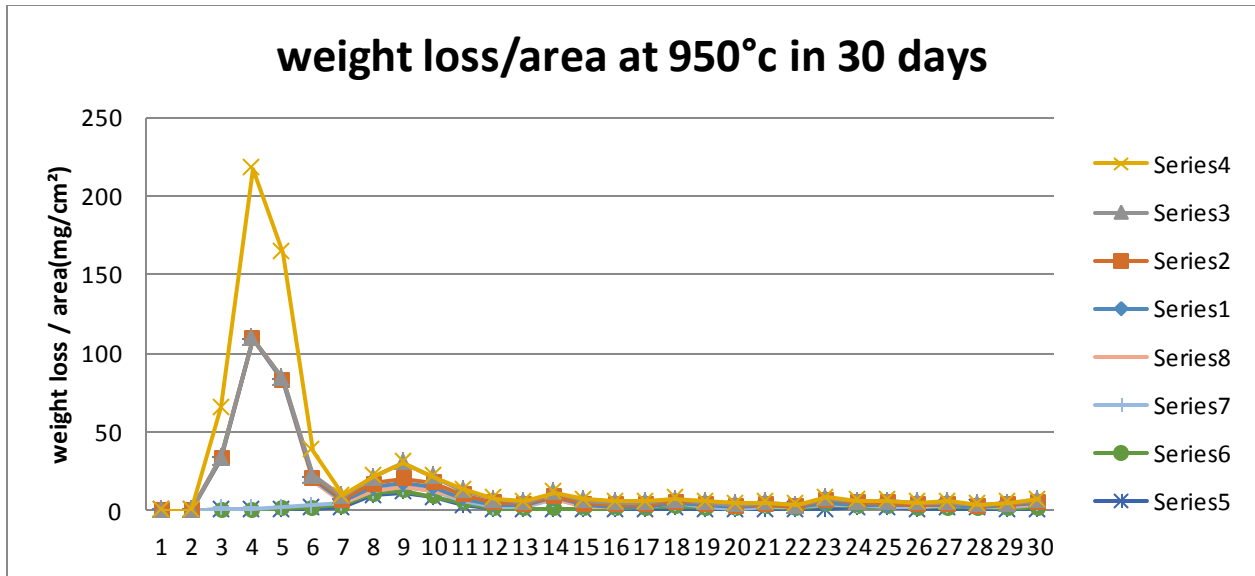


Figure 5.2 Shows the graph of Weight loss per unit area with regarding 30 days of month at 950°C temperature. where series 4 refers to uncoated and series 1, 2, 3 refers to coated samples.

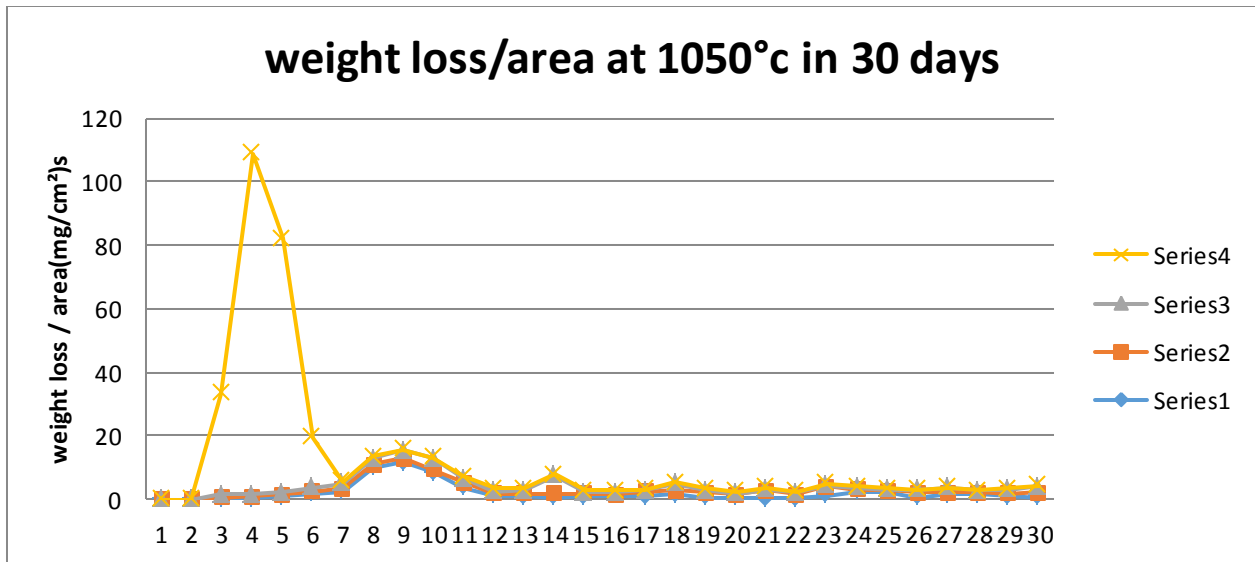


Figure 5.3 Shows the graph of Weight loss per unit area with regarding 30 days of month at 1050°C temperature. where series 4 refers to uncoated and series 1, 2, 3 refers to coated samples.

Conclusion:

Based on the experimental results during the slurry spray coating technique based on mullite-alumina cermet, the following conclusions have been made as listed here.

1. The slurry spray technique has been validated to be capable of producing resilient coating of satisfactory low corrosion rate, which is comparable with that produced from traditional techniques such as Air plasma spray.

2. The corrosion rate of mullite based slurry coated mild steel is very less in comparison to uncoated mild steel and corrosion rate of coated mild steel is highest and % IE is lowest at 850^oc, 45 min. sintering time and 5% additives. While corrosion rate is lowest and %IE is highest at 950^oc,45 min. and 1% additives
3. According to graph weight loss/area value of coated sample with respect to uncoated sample is less and less varies with respect of the days.
4. In the start of experiment weight/area value sharply increase (107 mg/cm²for the 4th day) for the uncoated sample, but as the days pass value of weight/area is decrease in comparison to 4th day.
5. While for mullite-alumina coated samples values of corrosion rate does not differ so much and value lies approximately in between 0,1 and 2.88.

References:

1. Gunthner M, Schutz A, Glatzel U, et al. High performance environmental barrier coatings, Part I: Passive filler loaded SiCN system for steel. *J Eur Ceram Soc* 2011; 31:3003–3010.
2. Ho SY and Paul A. Coupled thermal, structural and vibrational analysis of a hypersonic engine for flight test. *Aerosp Sci Technol* 2006; 10: 420–426.
3. Thome L and Garrido F. Application of ion beams to nuclear waste issues: Evaluation of nuclear ceramics. *Vacuum* 2011; 63: 619–626.
4. Koizumi M. FGM activities in Japan. *Compos Part B:Eng* 1997; 28: 1–4.
5. Nguyen P, Harding S and Ho SY. Experimental studies on slurry based thermal barrier coatings. In: 5th Australasian congress on applied mechanics, Brisbane, Australia, 10–12 December 2007, pp. 545–550.
6. Y.L. Xi, D.L. Chai, W.X. Zhang and J.E. Zhou: *Scripta Materialia* 54 (2006) 19.
7. Zhang Xiuqing, Liao Lihu, Ma Naiheng and Wang Haowei // *Composites: Part A* 37 (2006) 2011.
8. Wei Cao, Congfa Zhang, Tongxiang Fan and Di Zhang // *Materials Science and Engineering A* 496 (2008) 242.
9. Zhiming Shi, Ming Liu and Andrej Atrens: Measurement of the corrosion rate of magnesium alloys using Tafel extrapolation, Volume 52, Issue 2, February 2010, Pages 579–588.
10. Frank Wittea, Jens Fischera, Jens Nellesenb, Horst-Artur Crostackb, Volker Kaesec, Alexander Pischd, Felix Beckmanne and Henning Windhagena: In vitro and in vivo corrosion measurements of magnesium alloys, *Biomaterials* 27 (2006) 1013–1018.