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Optimizing adhesion strength of Environment Barrier coatingusing Slurry spray technique

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Abstract:—*Environment barrier coatings (EBCs) have been widely developed to protect metallic and Si-based ceramic components from high temperature environmental attack and thermal stresses in structures and machine components across a wide range of industries and applications.Zirconia-yttria based oxides and (Ba,Sr)Al2Si2O8 (BSAS)/mullite basedsilicates have been used as the coating materials. The objective of this paper is to optimize adhesion strength of Mullite based Ceramics – metal environment barrier slurry sprayed coating on mild steel while using a new, low cost and effective technique; Slurry Spray Technique(SST). The L9 Taguchi method has been used to develop the empirical models for response characteristics i.e Adhesion strength against three significance parameters sintering temperature, sintering time and Percentage additives with three levels have been employed.*

Key Words: *— Environment barrier coatings, Slurry Spray Technique, Mullite*

INTRODUCTION

Environment barrier coatings (EBCs) are essential structural components in current engineering applications associated with high temperatures or high thermal fluxes as well as in future developments. Several generations of super alloys have been developed over the past 20 years, so that these can with stand the hot gases in harsh environment (1). But the limits of stress rupture, surface protection and melting points creates difficulties ahead of these super alloys. In addition, the amount of air that can be used for cooling in high-performance engines is limited(2,3). These difficulties divert mind towards use of Environment barrier coatings (EBC's) as a protective against hot gases in various applications. These include thermal protection for rocket and scramjet engines, re-entry space vehicles, gas turbines, diesel engines, nuclear power plants and many other structures and
Machines (4).

In this present work a multilayer Mullite-Alumina based Environment barrier coatings (EBC) has been developed by SST technique using Tio$_2$ as sintering additive, Tetra Sodium Pyrophosphate as Dispersant and Hydro soluble polyvinyl alcohol as binder. The composition of the weight percentage of all the components for the graded coatings can be seen in Table1.

Table 1: Composition of slurry mixture

125g (1%) additive

Ceramic-Metal	Mullite (50%)	Alumina (50%)	Ni (100%)	**TiO2**(Additive)	Binder	Dispersant	Mix agent
	44%			1%	2.5%	0.5%	52%
60-40	16.5	16.5	22	1.25	3.125	0.625	65
30-70	8.25	8.25	38.50	1.25	3.125	0.625	65

SLURRY SPRAY TECHNIQUE

The literature survey has revealed that there are number of environment barrier coating techniques such as HVOF, APS and EV-PVD Techniques (5,6). But an advancement of wet powder spray, Slurry Spray Technique (SST) has been emerged as a low cost, simple and effective technique (5).The Slurry Spray Technique (SST) utilizes traditional wet powder spraying methods to deposit coating materials onto a target substrate to produce a functional coating. 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 spray guns as shown in figure 1. Successive layers are then sprayed onto the metal substrate and dried using varied slurry compositions. After depositing the desired no. of layers the multi-layered coating is loaded in a compression chamber to form a densified layer before being sintered with an acetylene torch or in a high temperature furnace. The applied pressure varies depending on the number of coating layers, typically between 10 and 40 Mpa.

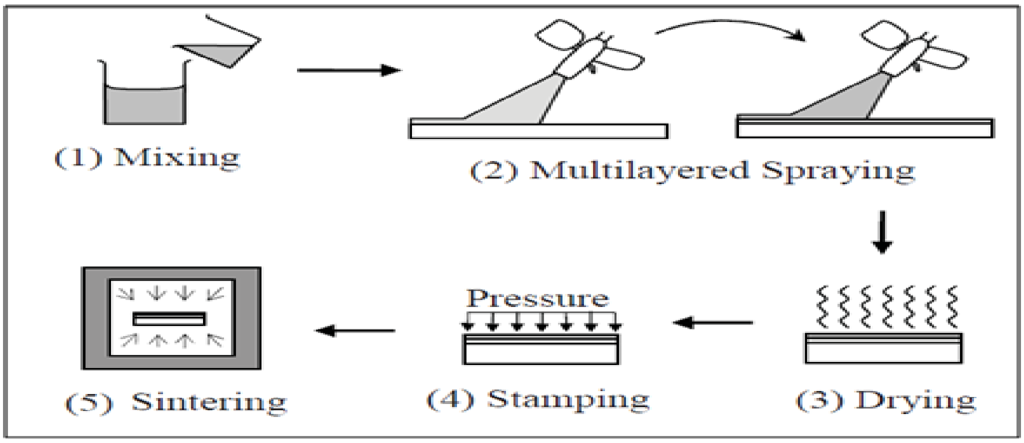

Figure 1: Process in SST

Taguchi modelling of Mullite-Nickel coating:

Taguchi method is a powerful statistical tool used for the design of experiments (DOE) can be effectively utilized to optimize process/ product, which involves various steps of planning,conducting and evaluating results of orthogonal array experiments to determine the optimum levels of usage parameters under very noisy environment(7).

The paper deals withmodelling of significant parameters such as sintering time, sintering temperature and percentage additives, using L_9 Taguchi technique. ANOVA is used to determine the most significant parameter affecting the output quality and characteristic using the quantities such as degree of freedom (f), sum of squares (SS), variance (V), percent contribution of each parameter (F-ratio) and then contribution ratio or contribution of variance is determined.

The**Table 2** represents experimental Observation of the control factors for sintering of mullite-nickel coating. As we have three control factors and three levels per factor, according to Taguchi method we choose L9 Taguchi design. In L9 Taguchi design, we use orthogonal arrays instead of standard factorial design. This design reduces the number of experiments.Experimental Results of Various Response Characteristics is shown in **Table 3.**

In our case the response is bond strength. It would be the best if bond strength is maximum. So as the objective is to maximize the bond strength, we select Signal-to-Noise ratio to larger the Better (LTB) quality. For Larger the Better the Signal to Noise ratio is given as,

(S/N)HB = -10 log (1/r Σ yi2), where, yi = each observed value(8).

Table 2. Experimental Observation:

TRAIL NUMBER (RUNS)	**CONTROL FACTORS**		
	Sintering Temperature (T °C)	**Sintering Time (min.)**	**Additives (% by weight)**
1	850	15	1.25
2	850	30	3.75
3	850	45	6.25
4	950	15	3.75
5	950	30	6.25
6	950	45	1.25
7	1050	15	6.25
8	1050	30	1.25
9	1050	45	3.75

Table 3.Experimental Results of Various Response Characteristics:

Exp. No.	Bond strength(MPa)			S/N Ratio (dB)
	R1	R2	R3	
1	13.1	14.9	14	22.92
2	14.5	14.8	15.2	23.42
3	14.2	11.5	13.5	22.32
4	17.4	18.5	17.6	25.05
5	18	16.5	15.5	24.43
6	16.4	13.5	15	23.50
7	17.1	18	15.5	24.54
8	15.5	16.5	14	23.71
9	16.1	15.2	17.5	24.22
	142.3	139.4	137.8	
Overall mean of BS = 15.537				

Effects on bond strength:

The average values of bond strength (BS) and S/N ratio for each parameter at levels L_1, L_2 and L_3 are calculated and given in Table 4

Table 4 Average Values and Main Effects: Bond Strength, BS (in MPa)

Process Parameter	**Level**	**SINTERING TEMP. (T)**		**SINTERING TIME (S)**		**ADDITIVE (A)**	
Type of Data		**Raw Data (°C)**	**S/N Ratio (dB)**	**Raw Data (minutes)**	**S/N Ratio (dB)**	**Raw Data (percent age)**	**S/N Ratio (dB)**
Average Values (BS)	L_1	13.966	22.890	16.233	24.162	14.766	23.379
	L_2	16.488	24.321	15.611	23.350	16.311	24.159
	L_3	16.155	24.159	14.766	23.350	15.533	23.766
Main Effects (BS)	L_2-L_1	2.522	1.431	-0.622	-0.304	1.544	0.845
	L_3-L_2	-0.333	-0.161	-0.844	-0.507	-0.777	-0.458
DIFFERENCE $\{(\mathbf{L_3}-\mathbf{L_2})-(\mathbf{L_2}-\mathbf{L_1})\}$		-2.855	-1.592	-0.222	-0.203	-2.322	-1.304
L_1, L_2 and L_3 represent levels 1, 2 and 3 respectively of parameters. L_2-L_1 is the average main effect when the corresponding parameter changes from level 1 to level 2. L_3-L_2 is the main effect when the corresponding parameter changes from level 2 to level 3.							

The main effects of different process parameters on the bond strength(BS) are plotted as Figures 2 (a, b, c).

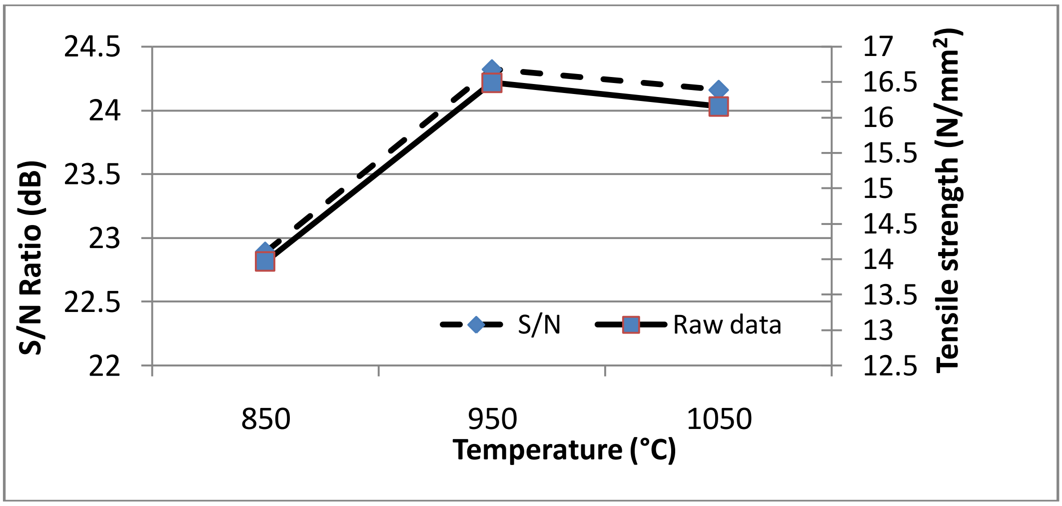

Figure 2 (a) Effect of sintering temperature on (S/N Ratio & raw data)

From figure 2(a) It is evident that bond strength increases from 13.96 MPa to 16.49 MPa, with increase in sintering temperature from 850°C (level 1) to 950°C (level 2), however slight decrease in bond strength is recorded beyond 950°C. Thus 950°C is found to be the optimal sintering temperature for mullite-nickel system with the aid of TiO_2 as sintering additive, which is lower than the conventional sintering temperature of mullite i.e. 1350°C which might be attributed to the effect of liquid phase sintering additive (Tio_2). Overall, the effect of sintering temperature on bond strength is significant as observed in both the ANOVA tables for the raw data and S/N ratio data.

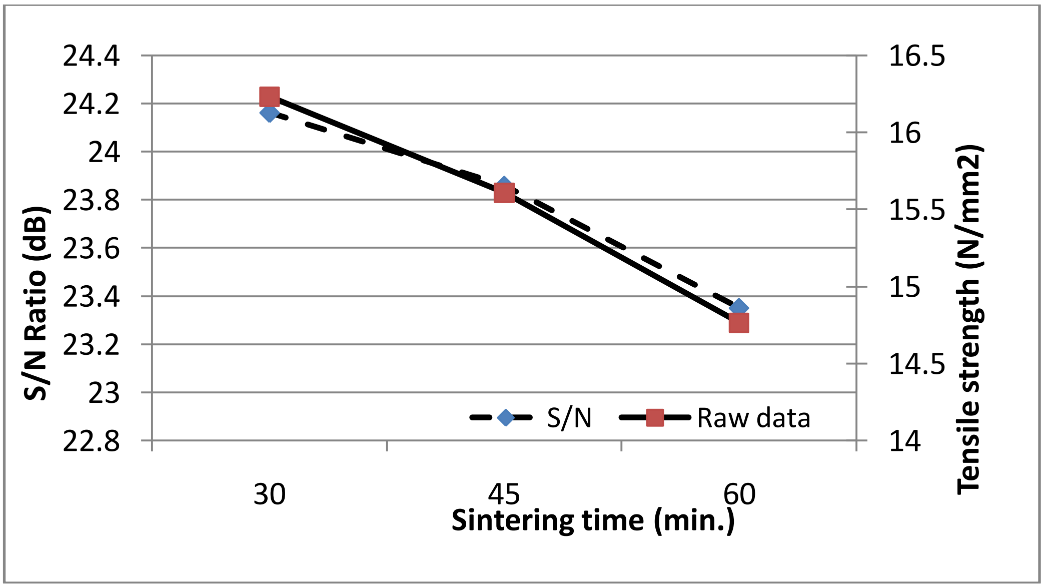

Figure 2(b) Effect of sintering time on (S/N Ratio & raw data)

It is noticed from the Figure 2(b) that the average values and main effect (S\N Data) of s*intering time* on bond strength. The maximum value of bond strength (i.e. 16.2 MPa) of the coating is achieved at 15 minutes (level 1) however with the increase in sintering time from 15 minutes to 45 minute there is constant decrease in adhesion strength. However, sintering time is not the largest contributing factor as evident from the percentage contribution of the sintering time i.e. 17.34%, considering the given parameters and conditions.

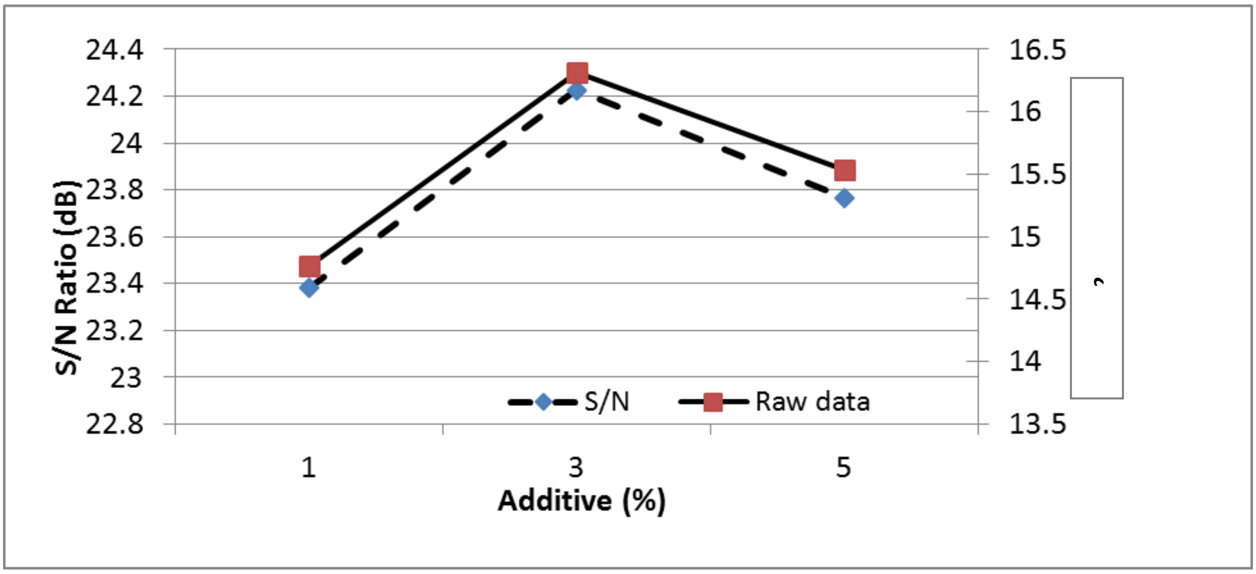

Figure 2(c) Effect of additive on (S/N Ratio & raw data)

Figure 2(c) shows that the average values and main effect (S\N Data) of *sintering additive* on bond strength. It is evident that bond strength increases from 14.76 MPa to 16.31 MPa, with increase in percentage of TiO_2 from 1% (level 1) to 3% (level 2), however there is decrease in bond strength recorded beyond 3% addition of TiO_2. Thus 3% TiO_2 as liquid phase sintering additive is found to be the optimal amount of sintering additive for mullite-nickel system. The increase in TIO_2 increases the grain size of mullite. Moreover, when a larger amount of TIO_2 powder was added during the pilot experiments, the large defects appeared as the large amounts of glassy phase concentrated at grain boundaries. The large defects in ceramic materials play a main role on the mechanical properties.

The optimum levels of the parameters for the higher bond strength are the, second level of sintering temperature (T_2), first level of sintering time (S_1) and second level of percentage of additives (A_2), corresponding to sintering temperature of 950°C, sintering time of 15 minutes, and percentage of additives of 3%.

Selection of optimum levels:

In order to study the significance of the process parameters towards the BS, analysis of variance (ANOVA) was performed. The pooled versions of ANOVA of the raw data and the S/N data for BS are given in Tables 5 &6. From these tables, it is clear that parameters T (sintering temp.), S (sintering time) and A (additive) significantly affect the mean and variation in the BS values. The percentage contribution of sintering temp. is highest (63.81%) for bond strength followed by percentage of additive (17.34%), and sintering time (18.47%). Bond strength is the "higher the better" type of quality characteristic. Therefore, higher values of BS are considered to be optimal.It is clear from the figure2 that the BS for raw data is highest at the second level of sintering temperature (T_2), first level of sintering time (S_1) and second level of percentage of additives (A_2), corresponding to sintering temperature of 950°C, sintering time of 15 minutes, and percentage of additives of 3%.

Table 5ANOVA (Raw Data, BS)

SOURCE	SS	DOF	V	P%	F-RATIO
SINTERNG TEMP	33.7918519	2	16.89593	43.63450281	14.58874
SINTERING TIME	9.75407407	2	4.877037	12.5951716	4.211065
ADDITIVE	10.7340741	2	5.367037	13.86061905	4.634154
Error	23.162963	20	1.158148	29.90970655	
Total (T)	77.442963	26			

*Significant at 95% confidence level, $F_{critical}$ =3.49

SS-Sum of Squares, DOF- Degree of Freedom, V-Variance

The S/N ratio analysis (refer Table 5.5 and Figure 5.11 (a, b, c) suggests ($T_{2,}S_{1,}A_2$) levels of parameters as the best levels for maximum BS of mullite-nickel coating

Table 6ANOVA (S/N Ratio Data, BS)

SOURCE	SS	DOF	V	P%	F-RATIO
SINTERING TEMP	3.68611452	2	1.843057	63.31276398	72.89868
SINTERING TIME	1.00966948	2	0.504835	17.34209969	19.96779
ADDITIVE	1.07572281	2	0.537861	18.4766327	21.2741
Error	0.0505649	2	0.025282	0.868503631	
Total (T)	5.82207171	8			

*Significant at 95% confidence level, $F_{critical}$ = 19

SS-Sum of Squares, DOF- Degree of Freedom, V-Variance

Conclusions

The following conclusions have been made from the above investigation:

1. The slurry spray technique has been demonstrated to be capable of producing durable coating of satisfactory bond strength, which is comparable with that produced from traditional techniques such as flame spray method, APS, HOVF and EV-PVD.
2. All the three selected parameters viz. sintering temperature, sintering time, and percentage of additives significantly affect the coating bonding adhesion strength.
3. With the increase in sintering temperature, coating bonding adhesion strength increases upto 950°C and a slight decrease on adhesion strength is recorded for sintering temperature of 1050°C. Moreover, sintering temperature is found to be the most dominating factor for coating adhesion strength, having the highest contribution of the order of 63.81%.
4. As the sintering time is increased from 15 minutes to 30 minutes and further to 45 minutes slight decrease in coating adhesion strength is observed.

In conclusion, Mullite based slurry spray environment barrier coating using SST, represent a rapidly developing area of science and engineering with numerous practical applications. The research needs in this area are uniquely numerous and diverse, but Ceramic coatings promise significant potential benefits that fully justify the necessary effort

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