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Characterization of Slurry Sprayed Mullite-Alumina based Thermal and Environment Barrier Coating

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Abstract: *Mullite-Alumina-based coatings were deposited on mild steel by a relatively simple and low-cost surface deposition technique based on the slurry spray technique (SST) developed for thermal and environmental barrier coating (TEBC) applications. There are many techniques to fabricate TEBC such as flame spray, HVOF, cold spray, Electric arc spray, EB-PVD and APS coatings.* ***.*** *Thus, in this article mullite-alumina based TEBC is fabricated using SST process while maintaining its simplicity and cost effectiveness of fabrication technique. Properties like porosity, thickness of coating, hardness of coating are characterized according to Taguchi's experimental design based on L9 orthogonal array. Microstructures of sprayed coating is studied by SEM and XRD. This mullite-alumina based Thermal and Environment Barrier coatings using SST reveal uniform coating deposits with adequate adhesion strength which is comparable with that produced from traditional techniques.*

Keywords:

Mullite-Alumina coatings, slurry spray technique, adhesion strength.

Introduction

Modern engine constructions together with the technological advancement lead to the evolution of new thermal and environmental barrier coating types and to the improvement of the formerly used coatings. Thermal barrier coatings were first successfully tested in the turbine section of a research gas turbine engine in the mid- 1970s (1). For many decades, the development of TBC remains an attractive research area due to its applications in various automobiles engines and gas turbines. Thermal Barrier Coatings (TBCs) are surface coatings in which high insulating materials, like ceramics forms the top layer. A thermally grown oxide is sandwiched between top coat and bond coat. This bond coat is bonded to a metal substrate, which is usually metal. This bond coat protects the metal load carrying structure during temperature excursions. The application of TBCs can significantly increase the operating temperatures up to 1400-1500ºC and increase thermal efficiency.

Ceramic TEBC's are considered technologically important because of their ability to increase gas turbine engine operating temperatures and reduce cooling requirements, thus help achieving engine performance goals..The advantages of using TEBCs include higher engine efficiency by increasing gas temperatures, and improved engine reliability by reducing engine hot-section component temperatures. The development of advanced ceramic barrier coatings is currently aimed at significantly increasing engine operating temperature and simultaneously reducing air cooling, in order to meet future engine lower emission, higher efficiency and improved reliability goals in various applications. The environment barriers found application as protective layers for steel surfaces of pistons and cylinders in Diesel engines and in case of elements of compressors housing for aircraft engines, made of titanium alloys, and working surfaces of exhaust nozzles, made of niobium alloys. Along with these applications, TEBC's also successfully applying in following areas.

- aerospace and aeronautical applications
- rocket combustors
- gas turbines diesel and high duties engines(2,3,4)

Characterization of Coatings:

5.1 *Micro-hardness*:

Micro-hardness of samples of mullite-nickel coating for different compositions is determined on Vickers Micro-hardness Tester. The polished samples of 25mm length of square pieces are prepared. Load of 100gm for 15 seconds on specimen is applied for indentation. Hardness results for coated samples of different compositions are:

Table 5.1 Hardness of the coating at different sintering temperature, sintering time and percentage of additive:

Sr. No.	Material Type	Sintering Temp. (°C)	Sintering Time (min.)	Additive (%)	Hardness (Hv)
1.	Mild Steel	850	15	1	485.09
2.	---do---	850	30	3	565.66
3.	---do---	850	45	5	601.69
4.	---do---	950	15	3	635.99
5.	---do---	950	30	5	685.94
6.	---do---	950	45	1	499.85
7.	---do---	1050	15	5	543.88
8.	---do---	1050	30	1	525.18
9.	---do---	1050	45	3	521.20

From the table 5.1, it is clear that the coating hardness is maximum at 950°C, sintered for 30 minutes with the addition of 3% additive and relatively low at 850°C, sintered for 15 minutes with 1% additive.

5.2 *Thickness of the coating*:

Coating thickness of mullite-nickel powder was measured on the polished cross-sections of the samples, using an optical microscope. The thickness values obtained for coatings deposited at different power levels for Mild steel is presented in Table 5.2. Each data point is the average of four readings.

Table 5.2 Thickness of the coating at different sintering temperature, sintering time and percentage of additive:

Sr. No.	Material Type	Sintering Temp. (°C)	Sintering Time (min.)	Additive (%)	Thickness (μm)
1.	Mild Steel	850	15	1	287
2.	---do---	850	30	3	380.33
3.	---do---	850	45	5	232.66
4.	---do---	950	15	3	153.06
5.	---do---	950	30	5	158.66
6.	---do---	950	45	1	135
7.	---do---	1050	15	5	176.66
8.	---do---	1050	30	1	171.45
9.	---do---	1050	45	3	100.52

From the table 5.2, it is clear that the coating thickness is high at 850°C at 30 min. sintering time and 3% additives. While coating thickness relatively low at 1050°C. It is clear that with the increase in sintering temperature, there is rise in enthalpy and thereby the particle temperature. Hence more number of particles gets melted during high temperature. When these molten species hit the substrate, get flattened and adhere to the surface forming big splats. If the inter-lamellar bonding between these splats is strong and the area of contact between the lamellae is more, leads to less amount of porosity. Hence, although there is decrease in coating thickness but a dense coating is formed.

5.3 *Coating Porosity*:

Measurement of porosity is done using the image analysis technique. The polished surfaces of various coatings under this study are kept under a microscope (Neomate) equipped with a CCD camera (JVC ,TK 870E). Porosity on coating surfaces for different specimen are tabulated in the table below.

Table 5.3 Porosity of the coating at different sintering temperature, sintering time and percentage of additive:

Sr. No.	Material Type	Sintering Temp. (°C)	Sintering Time (min.)	Additive (%)	Porosity (%)
1.	Mild Steel	750	30	1	11.90
2.	---do---	750	45	3	11.60
3.	---do---	750	60	5	12.12

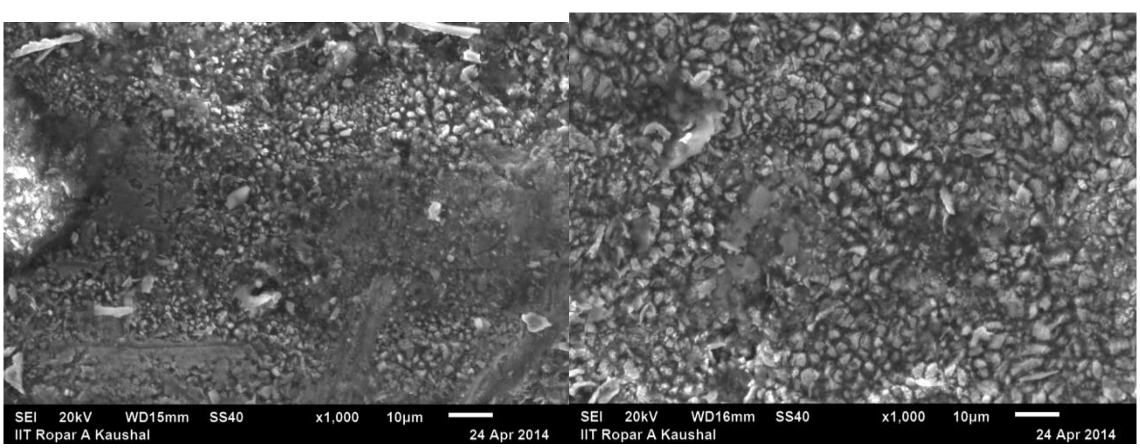
4.	---do---	800	30	3	10.84
5.	---do---	800	45	5	9.53
6.	---do---	800	60	1	12.94
7.	---do---	850	30	5	17.34
8.	---do---	850	45	1	19.85
9.	---do---	850	60	3	24.56

From the digitized image obtained by this system, coating porosity was determined using VOIS image analysis software. The results are tabulated in table 5.3. It was observed that, porosity volume fraction of these coatings lie in the range from 10% to 25%. The amount of porosity is more in case of the coatings of all compositions, made when the additive percentage is more (i.e. at 5%). Porosity level is relatively low in case of coatings when the additive percentage is 3%. Porosity level was also low at the sintering temperature 800°C and higher at 850°C.

Porosity analysis revealed that coating with 1% & 3% of TiO_2 of the total weight of the mixture of mullite-nickel powder at 850^0C showed a good level of decrease in the porosity as compared to other samples of 1,3 and 5% sintered at 850^0C which was also confirmed by the Scanning electron micrographs (SEM).

5.5 Scanning Electron Microscope (SEM) Analysis:

Microstructures of sprayed coated specimen were studied by S-3400N made by HITACHI scanning electron microscope mostly using the secondary electron imaging. The surface morphology of all coatings was observed under the microscope. Small specimens were sliced from the coated samples and coating cross-section was polished using the gold coating for 20 second for coating interface analysis under SEM. SEM images are shown in the figure below



(a) (b)

Fig 5.4: SEM images sintered at 850°C with (a) 1 % additive for 15 minutes, (b) 3% additive for 30 minutes

3% additive for 15 minutes

Figures above show the scanning electron micrographs of 1% & 3 % of magnesium oxide of the total weight of the mixture of mullite-nickel powder coating. From the micrographs it is clear that samples coated with 1% and 3% TiO_2 sintered at 850^0C shows a uniformly deposited layer of the composite. SEM figures reveal that sintering has a great impact on the grain boundary area. Grain boundary started to expand on the surface of substrate as the sintering temperature increased from 850°C to 950^0C which may be attributed to the mechanics behind the sintering effect that is driven by the reduction in surface energies of the individual particles by coalescing to form a continuous medium with increased grain boundary areas and reduction in the porosity. But it was also observed that sintering with further increase in temperature at 1050^0C resulted in the formation of cracks on the coated surface of substrate which evokes the failure of the coating above the sintering temperature of 850^0C.

5.6 X-Ray Diffraction Analysis

The XRD patterns of mullite powder showed the presence of aluminum oxide and silica powders with some traces of a-quartz. XRD patterns of mullite based coating of the two specimens at different temperature and sintering time and % additives has been tested shown in Fig. 5.9 and Fig.5.10, has sharp and intense peaks of mullite, the major phase and also the presence of Al_2O_3, the minor phase and traces of SiO2 in the coating. Peaks pertaining to silica (quartz or crystalline) were not seen in the as-sprayed coating, and this absence of the XRD signature of silica may be due to low relative proportion in the sample. A few small peaks of low intensity were unidentifiable. The fairly sharp peaks in the XRD pattern clearly indicate that the duplex coating generally has a crystalline microstructure. But due to the presence of some amorphous matter (note the hump in the pattern between 20 and 35—2h values in Fig. 5.9), the microstructure may not contain fine particles. Slurry sprayed mullite coatings, which are to a large extent amorphous, will result in coating spallation. Complete crystallization and a finer microstructure can be achieved if the coating is heat treated between 750 and 1000 _C (the recrystallization temperature range for mullite).

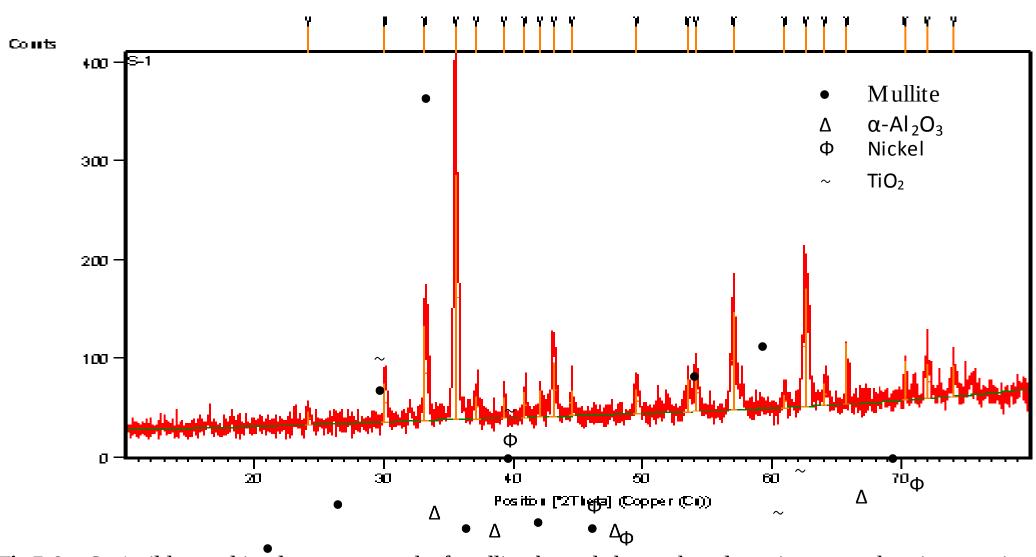


Fig5.9 : S- 1mild steel is slurry sprayed of mullite based thermal and environment barrier coating at 850°c,1% additive and 15min. sintering time.

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. Microhardness is maximium(685.94) at 950^0c sintering temperature, 30 min. of sintering time, and 5% percentage of additives.
3. Thickness of coating is maximum (380.33) at 850^0c sintering temperature, 30 min. of sintering time, and 3 % percentage of additives.
4. Porosity of the coating is minimum at 950^0c sintering temperature, 45 min. of sintering time, and 5 % percentage of additives.
5. 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%.
6. In conclusion, SEM and XRD results shows uniform deposition of Mullite based slurry spray environment barrier coating using SST, represent a rapidly developing area of science and engineering with numerous practical applications.

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