Preparation, Deposition and Characterization of Solution Precursor

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Abstract: The Solution Precursor Plasma Spray (SPPS) process has been used for Thermal Barrier Coatings (TBCs). In this work Zirconyl nitrate is used as solution precursor. It has been prepared in house for plasma spray. The prepared solution has successfully deposited on the substrate by SPPS process. The deposited coatings were characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD).

1. Introduction:
The thermal spray processes have been using the different kind of powder feed stocks such as for the coating. It has been widely used for various purposes like to protect from corrosion, improve durability and oxidation resistance properties at high temperature and it can reduce the metal service temperature [1-2]. The Solution Precursor Plasma Spray (SPPS) is recently developed for the fine spray. It is same as the existing conventional spraying processes with the only difference that instead of powders the feed is in the form of solution precursor. The liquid solution precursor material is injected into the plasma jet by a nozzle. Rapid heat-up and vaporization of precursor droplets in the formation of particles, which will be heated and accelerated to the substrate to generate coatings. In order to gain a better quality and performance of the coating, liquid precursors are sprayed into the plasma jet to generate finely structured coatings [3]. Deposition of small, melted particles leads to fine microstructure with the improvement in certain mechanical properties like hardness and strength. The different kinds of solutions or suspension precursors have been used for the different purposes. With normal APS process it is not possible to feed powder with size finer than 5-10\textmu m due to the effects of surface forces on powder flow [4]. Important thing to be noted that the SPPS coating has lower conductivity than EB-PVD coatings but in the upper range found in APS coatings. Mechanical properties of SPPS coatings like fracture toughness and hardness are measured to be higher than APS coatings. Both in plane and out of plane compressive strength for SPPS are lower than the APS but in plane elastic modulus is higher and out of plane is lower for SPPS than APS [5]. In general, SPPS coatings have a very fine grain structure [6]. Compared to conventional materials the Nano grained deposits and Nano sized particles have superior properties [7]. Thermal spraying can also use in the Automobile Industry [8]. The preliminary investigations have been carried out about the effects of processing parameters in the SPPS and to understand their implications and improving of this process.
2. Experimental setup:

2.1 Preparation of Solution Precursor:
In the preparation of zirconyl Nitrate the following process has been carried out. The 20 gm of Zirconium Oxide (ZrO₂) and 80 gm. of Potassium bisulphate (KHSO₄) are mixed together in a crucible. This mixed material is heated at the temperature of 600°C in muffle furnace about 15 minutes then the mixed powder is formed as a solid. After cooling, double distilled water and small quantity of sulphuric acid (H₂SO₄) is added to the mixed powder and heated on a Hot plate about three hours to dissolve and that has formed as liquid.

The Ammonia solution 25% (NH₃) is added to above liquid, then the solution will be precipitated and heated on a Hot plate for few minutes and filtered it about 10 times by adding double distilled water to remove the sulphates to obtain zirconium oxide.

Immediately after filtration some of double distilled water and Nitric acid (HNO₃) is added and heated on a Hot plate for few minutes to dissolve then it has formed as Zirconyl Nitrate of 500 ml. 500 ml of double distilled water is added to the above solution to make 1 liter solution.

2.2 Determination of Zirconium content in the solution:
10 ml of Zirconyl Nitrate is taken from the above 1 liter solution and Ammonia solution 25% is added and allowed to precipitate and heated on a Hot plate for 10 min.

The solution is filtered about 10 times to obtain Zirconium oxide powder and after drying for some time the Zirconium oxide powder is taken into a small crucible heated up to 800°C. After burning of this
material, the mole wt% of ZrO$_2$ is calculated. Now Y$_2$O$_3$ is added with Nitric acid and some quantity of Zirconyl Nitrate then heated on the Hot plate for 30 minutes to dissolve. This solution is added to Zirconyl Nitrate solution. Finally the solution will be formed as 8M%Y$_2$O$_3$.ZrO(NO$_3$)$_2$.

The typical Flow Chart of the processing of Preparation of Liquid precursor

![Flow Chart](image-url)
2.3 Coating processes:

Coatings were sprayed by using a direct current (DC) plasma torch (Metco 9 MB, SulzerMetco). The Ar was used as the primary and H₂ was used as secondary plasma gases. Compressed air was used as the solution precursor atomizing gas. The precursor used here was zirconyl nitrate [8M%Y₂O₃.ZrO(NO₃)₂] in this work. The substrate was characterized using a scanning electron microscope (SEM) and X-ray diffraction (XRD) and EDS.

The standard substrate is a square shape of 50x50 mm² with thickness 5 mm. Stainless-Steels of grade 304 was considered as substrate. Prior to the coating deposition, the substrates were grid-blasted in a closed chamber using aluminium oxide particulates of size-class FEPA 36 with particle size of 425 to 600 μm.

3. Results and Discussions:

| S.No | Plasma conditions          | Distance (mm) | Robot               |
|------|---------------------------|---------------|---------------------|
| 1    | Argon - 80                | 70            | Rastering profile - 70 mm/sec Movement - H&V |
|      | Hydrogen - 20             |               |                     |
|      | Current - 400 amps        |               |                     |
|      | Voltage - 50 volts        |               |                     |

SEM – MAG:
Fig. 6 SEM images at different magnification

EDX:

| Element | Weight % | Atomic % |
|---------|----------|----------|
| O K     | 47.73    | 83.85    |
| Y L     | 6.28     | 1.99     |
| Zr L    | 45.99    | 14.17    |
| Total   | 100      |          |

Fig. 7 EDS of the coated sample
4. Conclusion:

YSZ coatings by SPPS method was successfully obtained. At the 70 mm/sec of rastering speed and 70 mm spray distance, the coatings were done and characterized the surface. XRD analysis showed the formation of YSZ from liquid precursor. SEM observations showed significant effect of processing conditions on the microstructure of YSZ coatings. The prepared solution can be used for the surface coating.

5. References:

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