Development and Characterization of Ni-YSZ Coating on Stainless Steel by HVOF Process

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Abstract— The objective of this study is to deposit oxide based cermet coating i.e. Nickel - yttria stabilized zirconia, Ni-YSZ on stainless steel (SS) by HVOF process. The YSZ content in the coating is 30 and 50 wt. %. The coating has been characterized by X-ray diffraction (XRD) and scanning electron microscopy (FESEM). XRD studies reveal the presence of Ni and YSZ in the coating. The microstructural studies of the Ni-YSZ coating revealed the presence of porosity and micro cracks. The pin-on-disc studies revealed that the wear resistance of Ni-30YSZ coating is better than that of Ni-50YSZ and bare SS substrate under dry sliding conditions. Also, the coefficient of friction is low for Ni-30YSZ coating compared to the Ni-50YSZ coating. The wear rate of Ni-30YSZ coating is equivalent to that of hard chrome coating suggesting it to be a probable substitute. It was seen from the polarization studies that the HVOF deposited Ni-YSZ coatings displayed poor corrosion resistance compared to the uncoated SS due to the presence of porosity and micro-cracks a characteristic feature of HVOF coatings.

Keywords— HVOF; XRD; Wear; Corrosion; Coating

I. INTRODUCTION
High velocity oxy fuel process has attracted a lot of interest because of the ability to obtain dense adherent coatings of composite and alloy powders. A lot of researchers have explored this process to obtain carbide based coatings like WC-Co, NiCr-Cr3C2 [1-7]. The potential of this process to obtain oxide based coatings have largely been ignored due to the low deposition temperatures unlike that of plasma spraying which is conventionally adopted for oxide coatings. The requirement for the deposition of an adherent coating by thermal spray process is the complete melting of the particle during the process. The maximum temperatures generated in the HVOF process using the propylene gas is 2896 oC and the melting point of oxide particles is high hence, scarce reports are available on the development of these coatings by HVOF process [8-12].

In the present study an attempt has been made to develop yttria stabilized zirconia (YSZ) based coatings by HVOF process. YSZ has been chosen as it is widely used as thermal barrier coating, TBC. Dobbins et al have deposited YSZ coating over plasma sprayed Ni-22Cr-10Al-1Y bond coat for TBC by HVOF process [8]. It is surprising to see how YSZ coating can be obtained without a metal binder as the operating temperature in the HVOF process is almost close to the melting point of YSZ and moreover, the low thermal conductivity of zirconia will affect the melting of the YSZ particles of size > 40µm. This problem has been overcome in the present investigation by adding Ni metal as the binder for the YSZ particles so that adherent coatings can be obtained using HVOF process. An alternate is to adopt air plasma spray or electron beam physical vapor deposition processes to obtain this coating wherein the operating temperatures are much higher compared to HVOF resulting in complete melting of the particles. Since both these processes are costly and time consuming as they require either a high voltage power supply or a high vacuum, HVOF process has been used in this investigation.

The Ni-YSZ coatings with YSZ contents of 30 and 50 wt. % has been developed and these coatings have been characterized and evaluated in detail.

II. EXPERIMENTAL PROCEDURE
Ni-YSZ coatings with YSZ contents of 30 and 50 wt. % have been developed on SS substrate by HVOF process. Commercially available nickel and YSZ powders were used as the starting material for depositing these coatings. Fig 1 (a), (b) shows the optical images of as received YSZ and Ni powders respectively. It is observed that the YSZ particles are spherical in shape with a size of ≈ 80µm. The size of the YSZ particles was reduced to 50 µm using ball milling with disc rpm 150 for 20 min under dry condition. The ball to powder ratio was maintained as 10:1. It is seen from Fig 1b that the Ni particles are irregular in shape with an average particle size of ≈ 30µm. These two powders were mechanically mixed in a ball mill under dry conditions at an rpm of 100 for 10 mins. and the milled powder mixture was sprayed on stainless steel substrate by HVOF process. The spray parameters adopted are listed in Table1.

In Fig. 1 Optical images for (a) Ni and (b) YSZ powders.

The phase purity of the powders and coatings was examined by X-ray diffraction (XRD) studies (Bruker D8 Advance) using CuKα (λ = 1.541 Å) radiation. The roughness of the coatings was measured using 3D Profilometer (Nano map 500LS USA). The surface morphology of the coating was...
examined using field emission scanning electron microscope (FESEM). The dry sliding wear behavior of Ni-YSZ coatings was studied using pin-on-disc wear tester TR-20L-PH 200, DUCOM. Uncoated and Ni-YSZ coated SS pins were tested against Al2O3 disc. The testing parameters adopted comprised of 5N load, 50 rpm sliding speed, 750 m sliding distance, 40 mm track radius and the duration of testing was 1h. All the wear tests were performed under ambient conditions.

### TABLE. I Spray parameters for Ni/YSZ coating

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen pressure</td>
<td>8 Kg/cm²</td>
</tr>
<tr>
<td>LPG pressure</td>
<td>6 Kg/cm²</td>
</tr>
<tr>
<td>Nitrogen pressure</td>
<td>6 Kg/cm²</td>
</tr>
<tr>
<td>Air pressure</td>
<td>5 Kg/cm²</td>
</tr>
<tr>
<td>Spray distance</td>
<td>300 mm</td>
</tr>
<tr>
<td>LPG flow</td>
<td>60 LPM</td>
</tr>
<tr>
<td>Oxygen flow</td>
<td>250 LPM</td>
</tr>
<tr>
<td>Nitrogen flow</td>
<td>20 LPM</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>38 g/min</td>
</tr>
</tbody>
</table>

### III. RESULTS AND DISCUSSION

#### A. XRD Studies

The typical X - ray diffraction patterns for (a) YSZ powder, (b) Ni powder, (c) Ni-30YSZ and (d) Ni-50 YSZ coatings are shown in Fig. 2a. The diffractogram of YSZ feedstock powder shown in Fig. 2a displays diffraction peaks at 2θ = 30.13, 34.91, 50.28, 59.71, 62.45, 63.75, 81.59, 84.33 and 94.63 degrees corresponding to miller indices (111), (200), (220), (311), (222), (400), (331), (420) and (422) respectively indicating that YSZ has a cubic structure (JCPD No.#30-1468). It is seen from Fig. 2(b) that the five characteristic peaks at 2θ = 44.37, 51.82, 76.40, 92.98, and 98.57 degrees is attributed to Ni in the crystalline form. The corresponding miller indices (111), (200), (220), (311), (222), (400), (331), (420) and (422) respectively indicating that YSZ has a cubic structure (JCPD No. #04-0850). The phase formation in Ni-YSZ coatings are shown in Fig. 2 (c and d). The XRD pattern shows prominent peaks corresponding to both YSZ and Ni along with a small amount of NiO at 2θ corresponding to 37.24 and 43.38 degrees. These peaks are indexed to (111) and (200) respectively suggesting NiO has a cubic structure (JCPD No. #73-1519). The small amount of NiO can be associated with the oxidation of Ni during the spraying process. Ni-50YSZ coating displayed intense YSZ peaks and low intensity Ni peaks compared to Ni-30YSZ coating. This can be associated with the increase in the YSZ content from 30 to 50 wt. %. Ni-30YSZ coating exhibits Ni peak as the major peak while Ni-50YSZ coating shows YSZ as the major peak. It is observed that the cubic structure of YSZ is retained in the Ni-YSZ coatings deposited by HVOF process.

#### B. Surface Morphology and Composition

Fig. 3. shows the surface morphology of the HVOF coated Ni-YSZ coatings obtained using FESEM. The morphology of the Ni-30YSZ coating contains porosity while that of Ni-50YSZ coating appears to contain porosity and micro-cracks in addition to some un-melted or partially melted YSZ particles. Dobbins et al have reported that YSZ coating deposited by HVOF method exhibits fine interlamellar porosity of vol. % 21 ± 6.3 [8]. Table.2 displays the elemental composition of the coating obtained using EDX analysis. It is observed that the amount of YSZ in the Ni-YSZ powder mixture is retained in the sprayed coating as well. This conveys the uniformity of the feedstock in powder mixture. The average thickness of the coating is around 100 ± 20µm and is examined by optical microscopy and is shown in Fig 3(e).

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**Fig. 2.** XRD pattern for as received (a) YSZ powder, (b) Nickel powder, (c) Ni-30YSZ coating and (d) Ni-50YSZ coating.

**Fig. 3.** FESEM images for HVOF coated samples at different magnifications (5X, 10X) of Ni-30YSZ (a, b), Ni-50YSZ (c, d) coated samples and (e) cross sectional image for Ni/50YSZ coating, respectively.
TABLE 2. Elemental composition of Ni-30YSZ and Ni-50YSZ HVOF coated samples.

<table>
<thead>
<tr>
<th>Name of the Coating</th>
<th>O Wt. %</th>
<th>Ni At. %</th>
<th>Y Wt. %</th>
<th>Zr At. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-30YSZ</td>
<td>11.36</td>
<td>33.60</td>
<td>71.01</td>
<td>15.43</td>
</tr>
<tr>
<td>Ni-50YSZ</td>
<td>15.77</td>
<td>44.55</td>
<td>49.86</td>
<td>30.68</td>
</tr>
</tbody>
</table>

C. Roughness

Fig. 4 shows the 3D surface roughness of the (a) uncoated sand blasted SS, (b) Ni-30YSZ and (c) Ni-50YSZ coated samples, respectively. The average roughness of the sand blasted substrate by profilometry was found to be 1.8 μm whereas the roughness of the HVOF coated samples are 7.77 μm and 9.2 μm for Ni-30YSZ and Ni-50YSZ coated samples respectively. The roughness of the coating is higher than the uncoated substrate because of the partially melted or semi molten Ni and YSZ particles distributed throughout the surface, which can be clearly observed from the surface morphologies. The roughness is observed to increase from 7.77 μm to 9.2 μm with an increase in the YSZ content from 30 to 50 wt. %.

Dobbins et al have reported an average roughness of 13.10 μm for HVOF YSZ coating. The lower roughness of the Ni-YSZ coating developed in the present study compared to the reported YSZ coating can be related to the higher impact velocity resulting in the melting of the particles.

D. Wear and Friction

The dry sliding wear response of uncoated SS and Ni-YSZ coated samples tested against alumina disc at 5N load is shown in Fig. 5(a). The tests were performed for a sliding distance of 750 m. The results showed that the uncoated SS sample suffered a wear loss of ≈ 225 μm, whereas, Ni-50YSZ and Ni-30YSZ coated samples exhibited a wear loss of ≈ 100 μm and ≈ 27 μm, respectively. In other words, the wear rate of uncoated SS, Ni-30YSZ and Ni-50YSZ coated SS are 2.61 x 10^{-4}, 2.66 x 10^{-4} and 7.61 x 10^{-5} mm²/Nm respectively. The wear rate of Ni-30YSZ coated SS is better than that of hard chrome coated SS (9-11x 10^{-6} mm²/Nm). Thus, modifying the surface of SS with Ni-30YSZ coating enhances its wear resistance by two orders of magnitude. Turunen et al have reported a wear loss of 31 μm for 7wt% YSZ coating obtained by HVOF process [12]. A sudden increase in wear loss is observed initially in all the samples and this is due to the running-in wear. In other words, a high localized contact pressure at the real contact point between the asperities of coated sample and alumina is responsible for the high wear loss. Subsequently, the wear loss stabilizes as a steady state condition is established. The low wear loss in case of Ni-30YSZ coated sample can be associated with the optimum YSZ content in the coating resulting in its enhanced wear resistance. An increase in wear loss with increase in YSZ content to 50 wt. % is observed and this can be associated with the weak bonding between the Ni binder and the YSZ particles leading to their poor wear resistance.

During dry sliding wear test, the height loss of sample measured by LVDT is also supported by measuring simultaneously the friction coefficient. The COF values of uncoated SS, Ni-30YSZ and Ni-50YSZ coated samples is shown in the Fig. 5(b). The coefficient of friction of uncoated sample is 0.7 while it is 0.45 and 0.25 for Ni-50YSZ and Ni-30YSZ coatings respectively. The results clearly demonstrate that optimum Ni and YSZ contents are required for the coating to display enhanced tribological properties.

III. CONCLUSIONS

Oxide based Ni-YSZ coatings were deposited successfully under optimized conditions by HVOF process. The XRD studies revealed the presence of prominent Ni and YSZ peaks along with a small amount of NiO. The presence of NiO might be due to the oxidation of Ni during the spraying process. The surface morphology of the coatings revealed the presence of porosity and micro cracks on the surface. The corrosion studies revealed that the corrosion resistance of bare SS substrate is better than the coated ones and this is due to the porosity and micro cracks present in the coating. The Ni-30YSZ coating displayed low wear loss and coefficient of friction thereby exhibiting improved wear resistance compared to the Ni-50YSZ coating. Thus, Ni-YSZ coating can be used for wear resistant applications.

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