INDO SWISS JOINT RESEARCH PROGRAMME (ISJRP)

JOINT UTILISATION OF ADVANCED FACILITIES

EXCHANGE GRANT REPORT

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Part 1 - General Information

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<th>Project Title:</th>
<th>Measurement of residual stress in thermally sprayed ceramic splats</th>
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Part 2 - Exchange Participant(s) Details

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3.1 Purpose of visit

Thermally sprayed coatings are widely used for wear, thermal barrier, decorative or other applications. Such coatings are built up by frozen deformed individual particles of the coating material known as splats. Figure 1 shows a coating formed by individual splats (a) and isolated splats (b) as well.

Figure 1. (a) A coating in cross section showing individual splats building up the coating and (b) a few isolated splats shown from top.

The first layer of splats determines the adhesion of the coating with the substrate. Such splats are subjected to a residual stress owing to rapid quenching associated with the process and the differential thermal expansion between the substrate and the coating. A high magnitude of residual stress can cause splat cracking and/or delamination of the splat from the substrate. Hence, residual stress constitutes an important aspect of splat-substrate adhesion and it is important to measure such stress. The objective of the present work was to measure the residual stress in ceramic splats using special techniques like micro-Raman or micro FTIR (Fourier transformed infra red) spectroscopy.

3.2 Short description of work carried out during the visit

A thermally sprayed ceramic coating is not usually deposited on to a standard substrate like steel directly. First an interlayer of a bond coat material is sprayed and the ceramic top coat is deposited on the bond coat. A bond coat improves the wetting of ceramics and reduces the thermal expansion mismatch between steel and ceramic.
Two ceramic powders (coating consumables) were used in this investigation, namely, alumina and zirconia. The former is well known for its wear resistance and the latter is a widely accepted thermal barrier coating material. The following bond coats were used in this study: Ni-5 wt% Al, Ni-20 wt% Cr and Ni 17Cr 6Al 0.5Y. Since this is an academic exercise, a few splat samples were deposited directly on polished steel surface as well.

At first a 100 µm layer of bond coat have been deposited on steel substrate. It was polished using various grades of polishing papers and diamond pastes so as to finally obtain a surface having a surface roughness of 0.3 µm. The splats were deposited on the polished bond coat. The purpose of polishing the bond coat is to create well shaped splats which can be further studied using the above mentioned characterisation techniques [Sabiruddin, 2011].

Raman spectroscopy was carried out using a Dilor XY 800 triple monochromator micro Raman spectrometer equipped with a Ar laser source generating laser of 514.5 nm wavelength and a CCD camera for collecting the Raman signal. FTIR microscopy has been undertaken using a Bio Rad FTS 175C instrument equipped with a UMA 500 (MCT-Detector) in reflectance mode. For each measurement an area of 200 x 200 µm has been selected. More than one set of measurements have been taken for each bond coat.

3.3 Outcomes

Figure 2 shows an alumina splat (a) and the corresponding Raman signals.

![Figure 2. An alumina splat (a) and the corresponding Raman signals (b).](image)

It can be observed from figure 2 (b), that alumina failed to generate any Raman signal initially (blue curve). Some signals appeared after two minutes of exposure (green curve). The alumina present in the coating belonged to the $\gamma$ phase which was not Raman active. On prolonged exposure to laser this phase was transformed to the Raman active $\alpha$ phase and signals appeared [Krishnan, 2003]. This leads to
construe that this material should be studied by some other techniques like micro FTIR which can generate a signal from γ alumina.

Figure 3(a) and (b) show the Raman spectra of an as-received zirconia powder and deposited splat, respectively.

Figure 3. Raman spectra of (a) an as-received zirconia powder and (b) a splat.

The peak present in the powder at 638.9 cm$^{-1}$ was found to shift to 634.8 cm$^{-1}$ in the splat. This shift is attributed to residual stress. The average Raman shift for this peak as obtained from five measurements for powders and splats were 638.5 cm$^{-1}$ and 634.0 cm$^{-1}$, respectively. This corresponds to a tensile stress of around 1 GPa [Trexeira, 1999]. This result is in agreement with splat residual stress measured using micro x-ray diffractometry [Matejicek, 2001].

Figure 4 shows the FTIR spectra of alumina splats deposited on Ni-Cr-Al-Y bond coat. In this plot four instances of the measurement are shown. In each case measurement was undertaken over an area of 200 X 200 μm. So it is expected that some signal from the bond coat has also been picked up during the measurement. Some peak shifts have also been noted. Further analysis of these results is underway.

Figure 4. FTIR spectra of alumina splats deposited on Ni-Cr-Al-Y bond coat.
3.4 Future collaboration with host institution

While this small project was a good opportunity to undertake a pilot project, intense follow up activity is necessary to supplement the initial encouraging results. The future collaboration will address the following:

- Effect of bond coats on residual stress in splats.
- Effect of process parameters like preheating temperature.
- Effect of controlled substrate topography (laser micropatterning).

3.5 Various comments

This scheme provides an excellent opportunity to initiate a work.

References


