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Residual stress and adhesion of thermal spray coatings: Microscopic view by solidification and crystallisation analysis in the epitaxial CoNiCrAlY single splat

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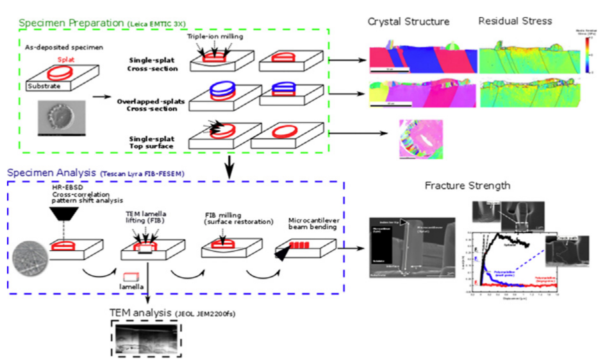
HIGHLIGHTS

- Crystallisation, residual stress and adhesion are characterised in thermally sprayed CoNiCrAlY single splats.
- HR-EBSD, TEM, FIB-milled cantilever beam bending and $\sin^2\Psi$ and pull-off method have been used.
- A dual epitaxial/polycrystalline structure and a gradient in fracture strength is observed in the single splats.
- Lower residual stress/adhesion in coatings vs single splat is linked to lower intersplat chemical-mechanical interaction.

GRAPHICAL ABSTRACT



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ABSTRACT

A new approach is proposed to achieve an in-depth understanding of crystallisation, residual stress and adhesion in epitaxial splats obtained by Combustion Flame Spray. Modelling of the fundamental process mechanisms is achieved with the help of experimental observations providing details with a sub-micrometre spatial resolution. At this scope, High Angular Resolution Electron Backscatter Diffraction and Transmission Electron Microscopy analysis are employed to provide insights into crystallisation and residual stress levels, while FIB-milled microcantilever beam bending is used for fracture strength measurements in the case of single splats. A comparison to fully-developed coatings is achieved by employing the X-ray Diffraction $\sin^2\psi$ technique and pull-off methods for residual stress and fracture strength, respectively. The methodology is applied to metallic CoNiCrAlY material sprayed onto a Ni-based superalloy substrate. The establishment of different crystallisation regions: epitaxial and polycrystalline, is the result of variations in the heat flux direction at the solidification front. Significant dislocation density is also reported, indicating the relevance of impact dynamics and plastic deformation mechanisms. The comparison with fully-developed coatings suggests a reduction in inter-splat bonding at splat overlapping.

1. Introduction

Thermal spray methods, e.g. plasma, combustion flame, and high-velocity oxyfuel, are widely used to produce thermal, oxidation and wear resistant coatings for many industrial applications, such as aerospace, electronics, energy systems and oil & gas [1-6]. The material to be deposited is fed, generally in powder form, through a jet stream which melts and accelerates it onto a substrate where it generates a thin layer as it spreads and solidifies. The simplicity and flexibility of the process, in terms of flame temperatures,

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chemistry and flow, makes it suitable to melt virtually any material and allows the coating properties (e.g. density and chemistry) to be tuned over a wide range. Many of the properties of thermally-sprayed coatings depend upon their microstructure (e.g. porosity, oxides, particles' overlapping), which in turn is dictated by the history of mechanical, thermal, and chemical phenomena involved during deposition. Due to the overlapping characteristics of the process and almost independent nature of each impact and solidification event, the problem can be reduced to the analysis of microstructure development at the single splat level (i.e. splat-substrate and successive splat-splat interaction) [7-9]. In this work, the word "droplet" refers to the in-flight molten particle while "splat" is the same droplet after substrate impact. A single droplet impact event can be described, in thermal spray, as a Rapid Solidification Process (RSP) since it allows continuous quenching while permitting material build-up [10]. The final properties of a solidified splat, namely crystal structure, shape/geometry, residual stress and adhesion are the result of several contributions.

1.1. Crystallisation and solidification

Although in RSP, due to the extreme temperature gradient and interfacial velocity of the solidification front, a planar or columnar front growth is often observed (as opposed to dentritic) [11], the specific crystallographic orientation of the splat grains and its relationship with the substrate orientation, if any, is complex to predict. It has been suggested that the establishment of epitaxy in thermally-sprayed materials depends on a combination of substrate crystalline structure, surface temperature and surface melting at droplet impact [12,13]. A number of studies have addressed the grain structure of thermally-sprayed single splats, mainly focussing on plasma spray and ceramic materials. The effect of substrate preheating on the splat grain structure has also been assessed by Yang et al. [14]. The authors observed that epitaxial Al_2O_3 splats can be formed on α-Al_2O_3 substrates at 900 °C. Co-Cr coatings have been analysed by Lau et al. [15], where a nanocrystalline structure of 21 nm average grain size was detected by analysis of X-ray diffraction lines. Nanocrystalline structures were also observed in the review of He and Schoenung on Cr_3C_2-NiCr and Inconel 625 materials [16], who concluded that the origin of these nanostructures still remained an open question. Gang et al. [17] addressed the topic by Transmission Electron Microscopy (TEM) analysis of the texture in FeAl splats from solid and molten powders, observing equiaxed 3D crystallites and elongated 2D nanograins respectively. TEM was also employed in the work of Chraska and King [18,19] on single and overlapping splats of Yttria Stabilised Zirconia (YSZ) ceramics, occasionally observing epitaxial columnar grains within the subsequent splat by depositing on a preheated stainless steel substrate.

1.2. Residual stress

Residual stresses developed in thermal spray can be attributed to two main contributions [20, 21]: quenching stresses, always tensile in nature and generated as the impacting splat contracts during cooling to the substrate temperature, and differential thermal contraction stresses, generated due to differences in thermal expansivity as the splat/substrate system cools-down to ambient temperature. Despite residual stress values having been extensively analysed at a macroscopic level in fully developed coatings, very few studies address the problem at a single splat level. Attempts have been made by Matejicek and Sampath [22] by using X-ray microdiffraction with a 800 μm collimator on plasma- and cold-sprayed Mo and Cu single splats respectively. Residual stresses ranging from 50 to −1050 MPa were measured, increasing in compressive magnitude at higher substrate temperatures. Although X-ray microdiffraction is a well established technique for residual stress measurement, it suffers from being limited to interaction volumes of 50 to 800 μm [23], thus making difficult to reproduce an accurate focus onto small thermally-sprayed splats and assess the stress at an intergranular level (nm in thermally-sprayed splats). Moreover, the residual stress signal acquired in this way is the average from an interaction volume, making it virtually impossible to separate the contributions in different sample directions. More recently, Sebastiani et al. [24,25], used a novel ring-core drilling method based on Focused Ion Beam (FIB) milling to measure in-plane, depth-resolved residual stresses on ceramic and metallic thermally-sprayed splats. With this technique, residual stresses can be sampled in areas of 3 to 5 μm in diameter, thus considerably smaller than X-ray microdiffraction, although limited to the in-plane direction.

1.3. Adhesion

The development of adhesion strength in thermally sprayed single splats can be attributed to three main bonding mechanisms [26-28]: mechanical interlocking of the splat solidifying within substrate irregularities, chemical bonding due to interdiffusion mechanisms between splat and substrate and physical (or van der Waals) bonding, i.e. weak contributions given by the atomic attraction between splat and substrate. Although mechanical interlocking is often addressed as the major contribution to the overall adhesion strength, the final value of this quantity is also affected by other mechanisms as oxidation, substrate condition and residual stresses. Due to the intrinsic small dimensions of thermally sprayed single splats (~μm), few attempts have been made in research to measure adhesion in these systems, mainly focusing on the application of known techniques generally used for thin films analysis. Balic et al. [29] reported an average strain energy release rate of 80 J/m^2 measured on single splats of Vacuum Plasma Sprayed (VPS) Al_2O_3 material. The value was measured after following the interfacial splat-substrate crack propagation, obtained by indenting the splat via a specifically designed experimental setup. Although a good match was reported in respect to adhesion values experimentally measured by other research groups, the technique is expected to fail in case of strongly adhered splats of ductile materials. A modified ball bond shear test was employed by Chromik et al. [30] to measure fracture strength of Ti splats. Values of up to 240 MPa were measured, largely depending on the droplets' impact velocity. The developed technique is simple as it required little setup, however due to the quite spherical shape of cold sprayed splats (in-flight droplets are largely unmolten at impact with the substrate in cold spray), doubts arise on the application to flat-shaped and/or severely microcracked ceramic splats obtained by other thermal spray techniques. Guetta et al. [31], studied the adhesion of cold sprayed Cu single splats on Al substrates by debonding via a novel LAser Shock Adhesion Test (LASAT) coupled to numerical simulations, obtaining maximum values of 350 MPa. The study demonstrated the development of a quite innovative method for adhesion measurement, although the technique results are difficult to replicate as it requires specific hardware and a calibration step based on finite element analysis.

Thus, although several attempts have been reported in literature to measure crystallisation, residual stress and adhesion in thermally-sprayed single splats, no extensive analysis at a (sub)micrometer spatial resolution for the whole single splat area has yet been carried out. In this paper, a multi-scale approach to the analysis of the crystallisation, residual stress and adhesion is performed, on splat-substrate and splat-splat systems, by using High Angular Resolution Electron Backscatter Diffraction (HR-EBSD) and TEM for crystal orientation, residual stress and geometrically necessary dislocation (GND) analysis and a FIB-milled cantilever beam bending technique for adhesive strength analyses. In this latter case, fracture strength has been probed at different locations in the splat-substrate interface by using an in-situ indenter inside the SEM chamber. For comparison purposes, residual stress and adhesive strength results are also measured on full