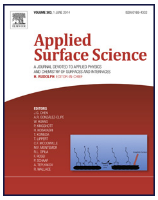
Applied Surface Science 303 (2014) 155–162

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc



Evaluation of the mechanical properties of plasma sprayed hydroxyapatite coatings

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ARTICLE INFO

Article history:
Received 30 July 2013
Received in revised form 21 February 2014
Accepted 21 February 2014
Available online 2 March 2014

Keywords:
Microhardness
Roughness
Plasma spray
Hydroxyapatite
Weibull modulus
Anisotropy

ABSTRACT

The mechanical behaviour of plasma sprayed hydroxyapatite coatings was evaluated using Vickers hardness measurements on the cross-section as well as on the top surface of coatings. The effects of applied load, measurement direction and indent location on the microhardness were investigated. Indentation was performed on dense and porous areas of the coatings. After Vickers indentation on the polished cross-section, the surface roughness on the indenter horizontal impression was measured to establish any influence on local surface tropology. The data was statistically analysed using the Weibull distribution to examine their variability and distribution within the coatings. It was found that the effect of lower applied loads (50 and 100 gf) and higher applied loads (300 and 500 gf) showed two distinct trends concerning the microhardness, indent roughness, and Weibull modulus of microhardness throughout the coating thicknesses in the dense area. Top surface microhardness was higher compared to the cross-section microhardness for 100, 300 and 500 gf whereas equal for 50 gf. The statistical analyses showed that the Weibull modulus of microhardness was related to the applied load and indent position. The Weibull moduli of microhardness were high on the dense areas of the coatings.

1. Introduction

Thermal spray coatings are widely used in various industrial sectors, such as agricultural implements; the automotive industry; aerospace engineering; primary metals processing; mining; chemical and plastics industries; the paper industry; oil and gas production; defence; electronics; energy; power generation; and biomedical applications [1–6]. For these applications the coating quality is of prime importance and an indentation test method is the commonly used technique to determine the coating quality [7].

Thermal spray coatings exhibit complex microstructures that consist of flat plate-like lamellae, cracks, pores, unmelted particles, weak interfaces between splats, and oxides: features that all contribute to highly heterogeneous and anisotropic behaviour [8–12]. Thus, it is important to consider the measurement direction, indent locations and applied load conditions when determining the microhardness of thermal spray coatings. Microhardness is reported in the literature as an average value of 10–30 random indents on the cross-section of the coating [13]. However, it is controversial whether such an average with an associated high standard deviation reflects the actual microstructural characteristics of thermal sprayed coatings. Also, there is some confusion arising, i.e., whether the indentation is located in a dense or porous area and, therefore the percentage of data collected from each region also needs to be unambiguously considered.

Lin et al. [14] and Leigh et al. [15] reported on the microhardness variation of thermal spray coatings with respect to applied load and measurement direction using Vickers indentation to understand relations with regard to the coating microstructure. A Knoop indentation technique was used to investigate elastic moduli and their variation across the surface and cross-section directions of the coatings [16,17]. The Weibull moduli were determined and statistic evaluation performed on various materials with different applied loads [7,10,18]. Lima et al. [19] demonstrated near-isotropic behaviour for plasma sprayed titania coatings. Yin et al. [13] examined the microhardness variation for alumina coatings with changing indent location throughout the coating thickness for a 50 gf load. However, more studies are necessary to investigate the effect of applied loads (e.g., 50, 100, 300, 500 gf) throughout the coating thickness; as well as taking into consideration the dense and porous areas, to fully understand the characteristics of thermal spray coatings.

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http://dx.doi.org/10.1016/j.apsusc.2014.02.125
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Table 1
Plasma spray parameters.

Parameters	Value
Power (kW)	40
Primary gas flow rate, Ar (slpm)	50
Secondary gas flow rate, He (slpm)	12
Carrier gas flow rate, Ar (slpm)	7
Powder feed rate (g/min)	27
Stand-off distance (cm)	11

In this study, hydroxyapatite (HA) coatings were air plasma sprayed onto mild steel substrates and microhardness was measured using the Vickers indentation technique. The indentation was performed on a dense and porous area and then the rule of mixtures was used to calculate the combined microhardness by varying the percentage contribution arising from the dense and porous regions. The indent roughness was measured using a 3D profilometer by varying applied load, measurement direction, and measurement location. The measured data sets were also statistically analysed to evaluate and understand their variability.

2. Experimental procedure and characterisation techniques

Plasma sprayed HA coatings were prepared using a SG 100 (Praxair Inc., USA) torch with a 175 sub-sonic nozzle. The process parameters are listed in Table 1. The starting powder was commercially available high crystalline HA powders, Captal 60-1 (Plasma Biotal, UK). The shape of the initial HA feedstock was typically spherical with a mean particle size of 45 ± 10 μm, *d*(10) of 20 ± 5 μm, *d*(90) of 80 ± 10 μm, Fig. 1. The mild steel substrates were of dimensions 40 mm × 30 mm × 3 mm. Prior to spraying, substrates were blasted with alumina grit and then air blasted to remove residual grit. The roughness of the substrate was 3.9 ± 0.47 μm.

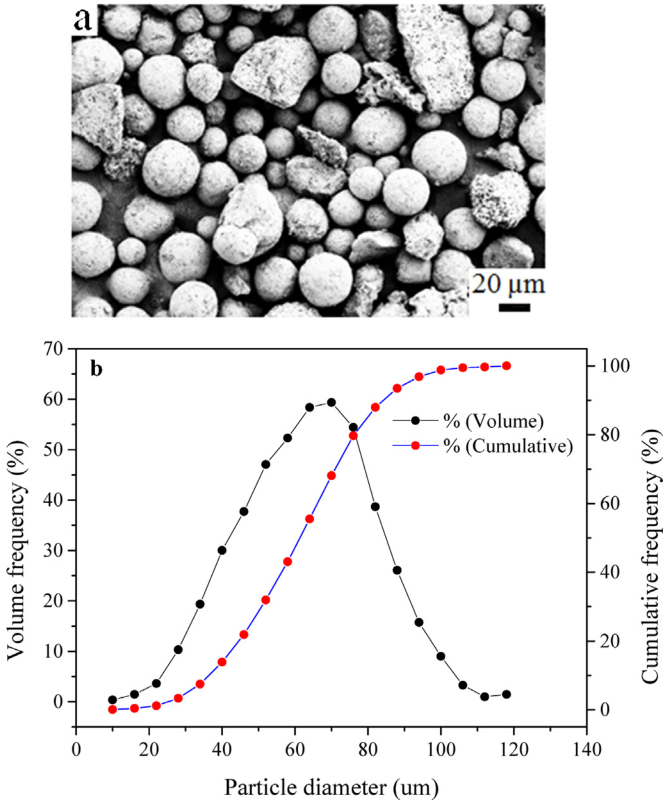

Fig. 1. Hydroxyapatite powders (a) particles image (b) particles distribution.

The microhardness measurements were performed using a Vickers microhardness tester (BUEHLER Micromet 2100 Series) with an integral microscope at 400×. The measurements were executed on the top surface and cross-section of the coatings by indenting at loads of 50, 100, 300, and 500 gf for a dwell time of 15 s. Indentations were performed on the cross-section of coatings at locations of 75, 175, and 275 μm distance from the substrate-coating interface for all loads to avoid the effect of impinging stress fields, Fig. 2(a). Twenty readings were taken along each region of interest, considering dense and porous areas for each indenter load, testing direction, and indent location; Fig. 3. The distance between each indentation was three times greater than the indent diagonal [20]. Porous area means area which contains lots of pores and dense area means relatively dense compared to the porous area which contains very few pores.

The Vickers microhardness measurement can be obtained from the following formula

$$\mathrm{VHN} = 1854.4\frac{P}{d^2} \tag{1}$$

the detail of which is diagrammed in Fig. 2(a), where *P* is the applied load in grams-force, *d* is the indentation diagonal length in micrometres, and VHN is the microhardness.

The rule of mixtures was used to determine a composite microhardness from dense and porous areas with varying percentage contributions (25%, 50%, and 75%). The composite microhardness can be calculated from:

$$H_c = dH_d + (1-d)H_p \tag{2}$$

where *d* is the percentage contribution of the dense area, and H_c, H_d, H_p are the composite microhardness, dense microhardness and porous microhardness, respectively.

The surface roughness was measured using a 3D profilometer (Bruker AXS ContourGT-K) on the polished cross-section and top surface after Vickers indentation. Roughness was measured from the corner of the horizontal indent impression to a 5 μm distance along the horizontal direction. This procedure is denoted as "indent roughness" in this manuscript. For each indent, roughness was measured at the two corners of the horizontal indent impression, and the averages of these two indent roughnesses are presented. The intent of this experimental study was to gauge the influence of the indent experiment on the surface deformation near the indent. For example, it may be possible to detect splat movement and crack features.

3. Statistical analysis

The Weibull distribution has been applied to characterise the mechanical properties and reliability of brittle materials [18]. There are also other statistical studies available, such as the normal and lognormal distributions [21]. However, the Weibull modulus is the most convenient and commonly used technique for describing property distributions within thermal spray coatings that may be highly skewed or broadly distributed [10,18]. The Weibull distribution for a two-parameter form is given as follows:

$$F(x) = 1 - \exp\left[-\left(\frac{x}{x_0}\right)\right]^m, \tag{3}$$

where *m* is the Weibull modulus or shape factor, *x* is the microhardness data, x_0 is the characteristic value (or scale parameter) below which 63.2% of the data lie, and *F*(*x*) is the cumulative density function of probability.

The Weibull plot is the most commonly used technique to obtain Weibull parameters and is obtained by rearranging equation 3 and taking natural logarithms twice [22,23].