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Depth Sensing Nano Indentation of Oxidized Plasma Sprayed CoNiCrAlY Coatings

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ABSTRACT

The aim of the present work is to analyze the evolution of microstructural and mechanical properties of Air Plasma Sprayed (*APS*) *CoNiCrAlY coatings after early stage high-temperature oxidation. Phase analysis and oxide scale characterization were performed by using X-Ray Diffraction* (*XRD*). *The microstructural features of CoNiCrAlY coatings were analyzed by Scanning Electron Microscopy* (*SEM*), *while Nanoindentation* (*NI*) *technique was employed to study the evolution of the mechanical properties.*

Keywords**:** *Air Plasma Spraying*, *Nanoindentation*, *Bond Coat*

1. Introduction

Thermal Spraying is a cost-effective technique for fabrication of MCrAlY (M = Ni, Co) overlay coatings for protection of Ni-based hot section components of turbine engines from high-temperature oxidation and hot corrosion [1]. Past researches focused on the analysis of microstructure of as-produced and oxidized High Velocity Oxygen Fuel (HVOF) and Vacuum Plasma Spray (VPS) MCrAlY coatings and less attention has been paid to the corresponding mechanical properties [2]. Therefore, the aim of this work is to complement the available data studying the mechanical properties of as-sprayed and oxidized CoNiCrAlY coatings produced by Air Plasma Spraying.

2. Experimental Procedure

The feedstock used for coatings fabrication was a CoNiCrAlY powder (Amdry 995C, Sulzer Metco), with particles size from 45 to 75 µm. The coatings were deposited on stainless steel substrates (final thickness 150 µm). Before deposition, the substrates were gritblasted with alumina to increase surface roughness and ultrasonically cleaned. Then they were placed on a rotating sample holder and coated. The process parameters have been reported in a previous work [3].

CoNiCrAlY coatings were isothermally treated in an air furnace at 1110˚C for 2 and 24 h, respectively, at heating rate of 6˚C/min. As-sprayed and annealed coatings were cut, ground and polished[2]. Cross sectional microstructural analyses were performed using Scanning Electron Microscopy (SEM) and Image Analysis.

Nanoindentation (NI) tests were performed on the polished cross section of as-sprayed and annealed coatings, by using a NanoTest 600 platform (Micro Materials Ltd) equipped with Berkovich indenter. The indentations were performed perpendicularly to the coating/substrate interface. The distance between the indentations was kept equal to 30 µm, to avoid a mutual influence of consecutive responses. The loading rate was 3 mN/s. The holding time at maximum load was adjusted to avoid the influence of nanoindentation creep on the results. In particular, the nanoindentations were performed at different dwell times, *i.e.* 15, 60 and 120 s and, finally, a dwell time of 60 s was set for the subsequent tests. In addition, multistep indents were done to assess the variation in the elastic modulus and the hardness with the indentation load.

3. Results and Discussion

As shown in **Figure 1**, as-sprayed CoNiCrAlY coating shows a lamellar microstructure, with a porosity of 3.3 ± 1.2% and an oxide content of 4.5 ± 1.7%. After thermal aging, a partial densification of the microstructure oc-

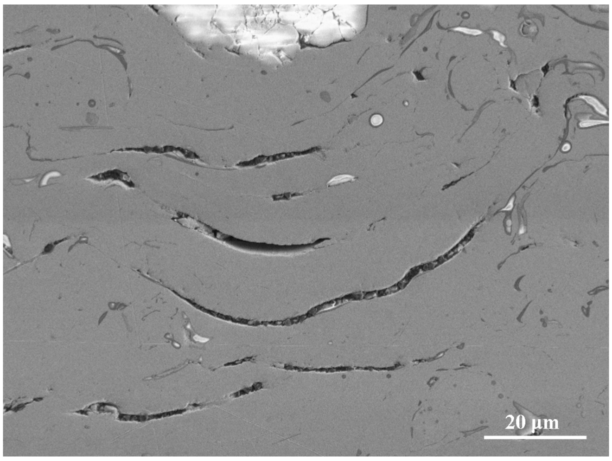


Figure 1. SEM image showing cross sectional microstructure of as-sprayed CoNiCrAlY coating.

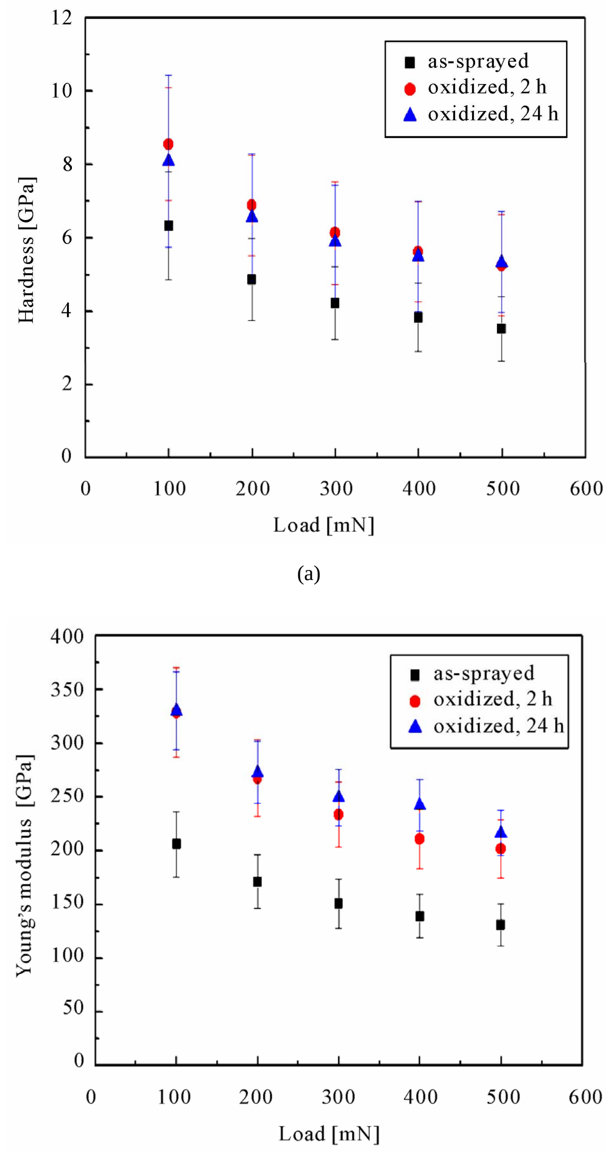
curred due to the closure of fine pores and splat boundaries: the porosity decreased to 2.4 ± 0.2% after 2 h and to 2.1 ± 0.2% after 24 h, while a double oxide layer (alumina + spinels) gradually grew on coating top-surface.

The determination of mechanical properties from NI measurements is based on the standard procedure developed by Oliver and Pharr, in which the area of the indent is calculated based on depth of the indentation and an area function [3,4]. As the so obtained mechanical properties may depend on the size of the indent, multistep nanoindentation tests were carried out in the present work. In particular, the indentation load was varied in the range between 100 and 500 mN, by step of 100 mN. The results obtained are reported in **Figure 2** below.

Both hardness and elastic modulus decreased with increasing the maximum load. It is possible to conclude that the hardness and Young's modulus are dependent on the applied load at relatively low loads. However, for increasing load this effect becomes limited and it is usually accepted to consider the corresponding material properties as representative of the mechanical behaviour.

This behaviour may be related to the accuracy achieved in the determination of the contact area at low loads. Indeed, at lower load the contact areas is usually underestimated.

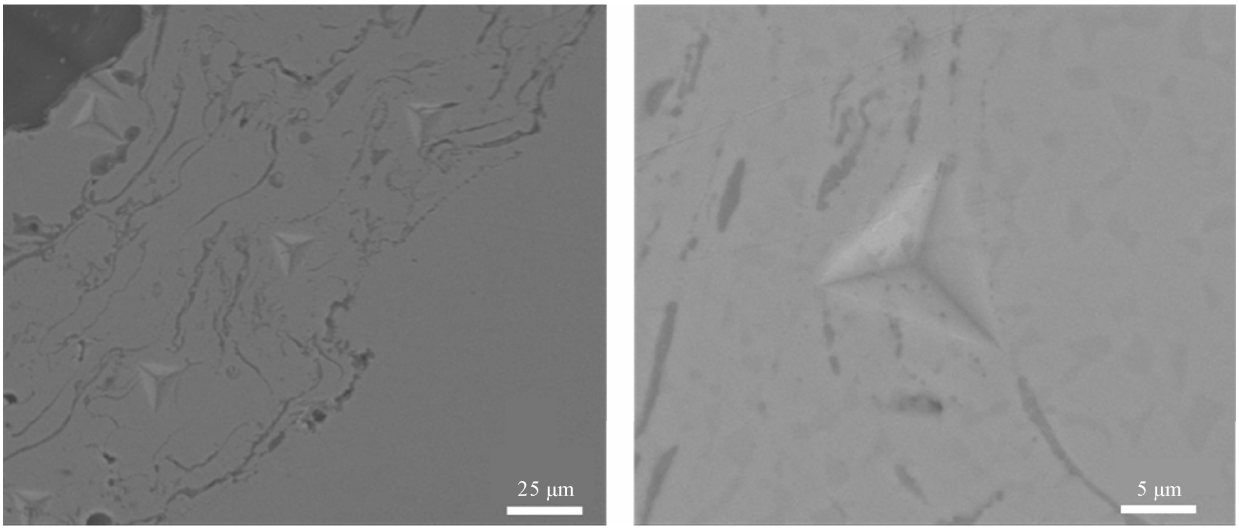
Moreover, problems associated with the "pile-up" or "sink-in" of the material on the edges of the indent during the indentation process could affect the measured values. To this purpose, SEM analyses of the indents were made (**Figure 3**) and significant pile-up or sink-in was not observed. However, it should be recognized that AFM images of the indents would be needed in order to drawn a quantitative conclusion on the above mentioned issues. The analysis of the results reported in **Figure 2** for higher loads, demonstrate that the nanohardness and the elastic modulus increase with increasing the aging time. This effect can be addressed to the partial densification of the microstructure. In particular, a sharp increase of mechanical properties occurs during the first hours of heat treatment and then tends to stabilize. The values measured are higher than those obtained by Kwon *et al.* for plasma sprayed CoNiCrAlY coatings by using NI and similar to those obtained for coatings sprayed by HVOF[5]. This result can be explained in terms of lower porosity and lower degree of oxidation of the coatings herein analyzed.



(a)

(b)

Figure 2. Evolution of (a) nanohardness and (b) Young's modulus as a function of the maximum indentation load.



(a) (b)

Figure 3. SEM image showing indents within coating microstructure.

4. Conclusions

Air Plasma Spraying was used to fabricate CoNiCrAlY coatings with relatively low porosity and oxide content. High-temperature exposure produced a partial densification of the microstructure. Nanohardness and elastic modulus decreased with increasing the maximum load during NI tests. The average hardness, measured at 500 mN, was 3.53 GPa for as-sprayed coating and increaseed up to 5.34 GPa after 24 h at 1110˚C. In turn, the mean value of the reduced Young's modulus was 131.4 GPa for as-sprayed coating and increased with increasing the aging time up to 216.3 GPa after 24 h at 1110˚C.

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