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OBTAINING AND CHARACTERIZATION OF HIGH-YTTRIA CONTENT ZIRCONIA COATINGS

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Abstract
Ceramic coatings are regarded as suitable materials for high-temperature applications due to their excellent stability, both mechanical and chemical. Zirconia stabilized with yttria is a material that presents many advantages when used as a bulk or coating material in applications where exposure to high temperatures is a factor. The atmospheric plasma spraying technique was utilized in the fabrication of ceramic coatings, characterized by elevated yttria content and stabilized zirconia, on metallic substrates. The structural, chemical and mechanical properties of the coatings were investigated through a range of analytical methods, including scanning electron microscopy (SEM – SE detector), energy dispersive spectroscopy (EDS – automatic and mapping) and tribo-indenter equipment (indentation and scratch). The fabrication process was successful in producing homogenous ceramic layers with a thickness of 150 µm.

Keywords: *ceramic coating, microstructure, TBC, SEM, EDS.*

1. INTRODUCTION

Yttria-stabilized zirconia (YSZ) ceramic coatings have found widespread application as thermal barrier coatings (TBCs) in the domain of air turbines and internal combustion engines. This can be attributed to their remarkable high-temperature resistance and chemical stability. The material's notable advantages include low thermal conductivity, tetragonal phase stability at elevated temperatures, and favorable thermal compatibility with metal substrates [1,2]. The most common deposition methods include thermal sputtering (atmospheric plasma, APS, and vacuum plasma, VPS) and electron beam vacuum deposition (EB-PVD). Each method exerts a distinct influence on the microstructure and performance of the coating. As demonstrated in the studies by Levi et al. [3] and Padture et al. [4] YSZ-based TBC systems have been shown to exhibit superior performance. In addition, Clarke and Levi [5] have proposed criteria for assessing durability based on microstructure and degradation mechanisms [1-5].

In numerous instances, the scratch test has been recognized as a versatile instrument for evaluating the mechanical integrity of a surface. Its utility has been demonstrated in a wide range of materials research and industrial R&D applications. In the early stages of research on the interpretation of scratch marks, the defect's location along the scratch trace was determined through the use of optical microscopy [6] or scanning electron microscopy (SEM) [7].

2. EXPERIMENTAL

The plasma spray method was employed to produce ceramic layers, with the samples being prepared from YSZ ceramic powders, some of which contained an anchor layer of metal powders. The ceramic layers were deposited on structural steel by successive passes, and the thickness of the ceramic layer was measured to be 125-150 μm [8].

Microscratch testing is a mechanical characterization method used to evaluate the interfacial adhesion and cohesive strength of thin films and surface treatments [9,10]. Two experimental protocols were performed: the first involved constant normal load scratching, and the second involved scratching with a linear load increase. The experimental tests were conducted under a maximum vertical load of 15 N, over a distance of 1 cm, with a translation velocity of approximately 0.15 mm/s. The tests were conducted using a blade-type indenter (Nvidia model) with a tip radius of curvature of 0.4 mm.

The surface condition subsequent to the scratch test was examined through the use of scanning electron microscopy (SEM; VegaTescan LMH II, VegaTescan LMH II, SE detector, 30kV, 15.5 mm WD) and dispersive EDS spectroscopy (Bruker detector, 6I10, Automatic and Mapping modes).

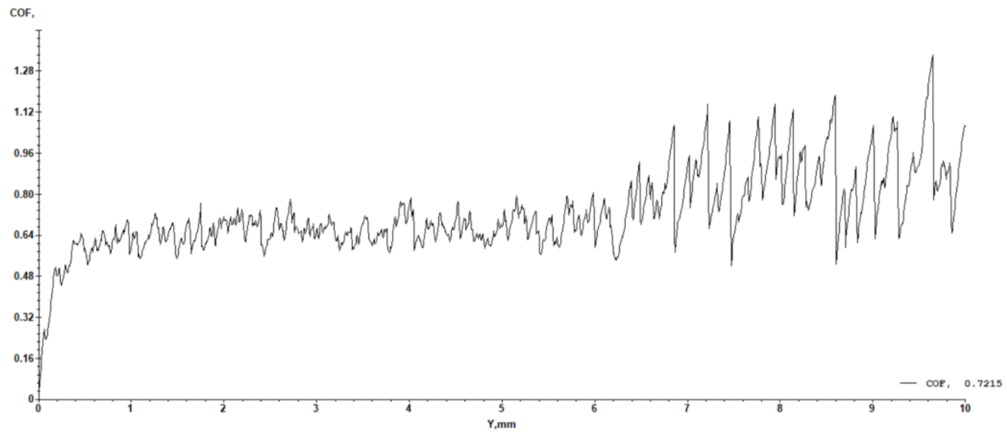
3. RESULTS AND DISCUSSION

The driving forces for the failure of the coating-substrate system in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses, and residual internal stresses present in the coating (usually as a result of the deposition process) [11,12]. When a progressive load scratch test is carried out, a series of consecutive coating failure events can be observed. As the load increases, the final event usually corresponds to total delamination. The critical load is contingent upon several parameters; some are intrinsic to the test itself, while others are extrinsic to the coating-substrate combination.

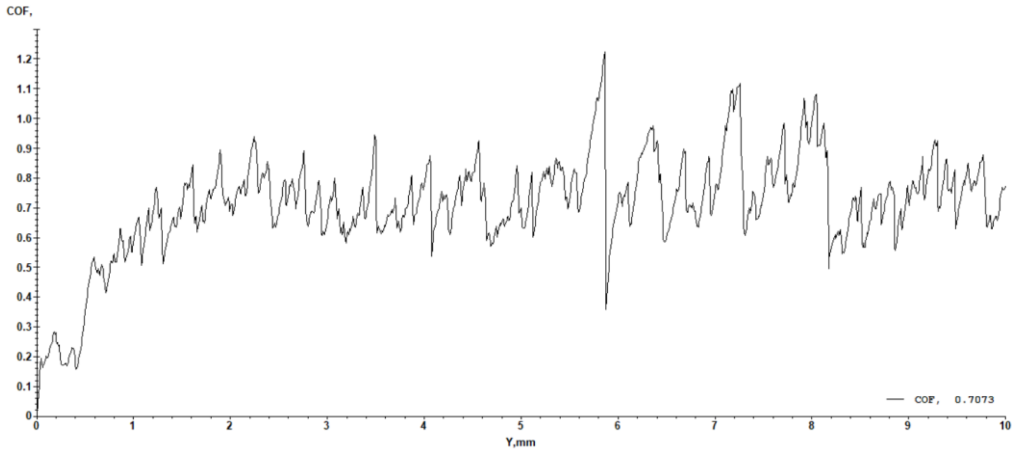
As illustrated in Figure 1(a), a substantial and unstable variation in the coefficient of friction was observed along a 10-mm displacement under a load of 15 N. This oscillation can be attributed to the inadequate adhesion of the ceramic layer to the metal substrate, resulting from the absence of an anchor layer. The sudden fluctuations in COF (friction coefficient) are indicative of micro-decohesivity processes, partial exfoliation of the layer, and potential local exposure of the substrate. Furthermore, intermittent rubbing has the potential to generate uneven wear, thereby reducing the reliability of the protective coating.

The tangential force is the most frequently recorded force signal. It can be correlated with the scratch deterioration points and used to recalculate the measured coefficient of friction (COF). The COF is defined as the ratio of the tangential force (Ft) to the normal force (Fn).

In the afferent plot of the sample lacking an anchor layer, a sudden and irregular increase in COF is observed after a specific distance. The perforation of the ceramic layer occurs in the region where the COF exhibits a substantial increase or abrupt fluctuation, indicating the exposure of the steel substrate and a modification in tribological behavior. The estimated piercing occurs within the range of 5-6 mm, with the point estimated to be around 5.5 mm, where a discernible transition in the COF curve is observed. The distribution of elements on the coated and scratched surface (Figure 2(a)) has also been shown to confirm this behavior. In this figure, the Fe substrate is observed to appear in the middle of the scratch.



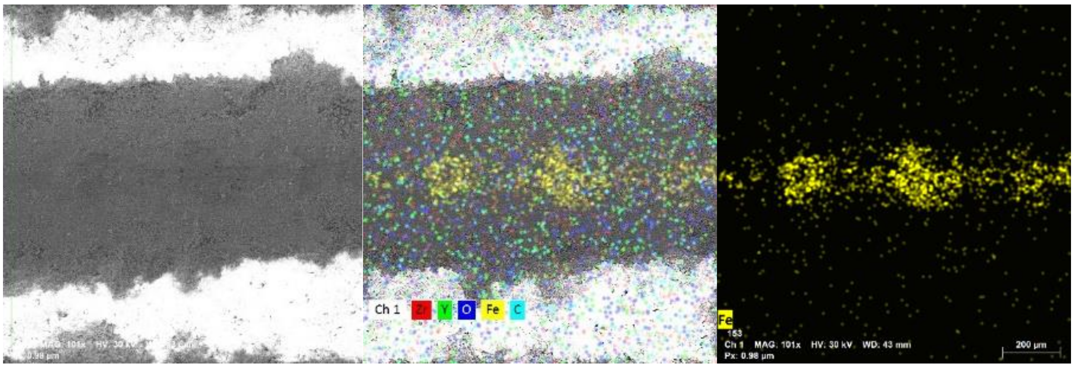
(a)



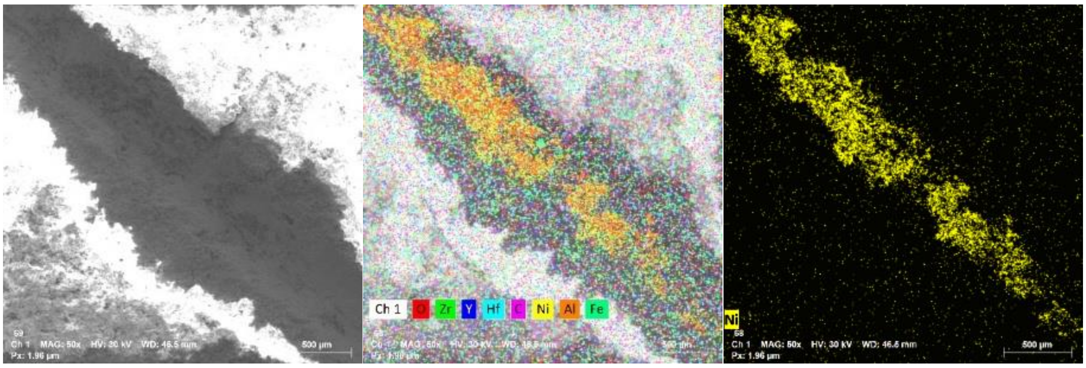
(b)

Figure 1. COF variation of steel coated sample (a) without and (b) with a bonding layer.

As illustrated in Figure 1(b), the graph demonstrates a significantly more stable and uniform evolution of the coefficient of friction, exhibiting relatively constant values throughout the entire test length. This stability is indicative of the robust adhesion between the ceramic layer and the steel substrate, thereby ensuring uniform load distribution and enhanced resistance to delamination. The metallic anchor layer functions as a mechanical and thermal transition, thereby reducing stress concentrations and enhancing overall tribological behavior. The curve is more uniform in comparison to sample (a), yet a slight change in the COF slope is observed in the ~7-8 mm region. This subtle, yet persistent, modification may signify the breakthrough of the ceramic layer and subsequent contact with the metallic anchor layer. Given the intermediate properties of the anchor layer, which exhibit a transition between those of ceramic and steel, the COF does not undergo a sudden variation but rather undergoes a smooth transition.



(a)



(b)

Figure 2. Scratch mark on the ceramic coating (a) SEM and elemental distribution for the sample without the bonding layer and (b) SEM and elemental distribution for the sample with the bonding layer.

The coefficient of friction signal can also function as an indicator of a critical deterioration point of the ceramic layer, even when the corresponding frictional force signal shows minimal change. However, the recalculated friction coefficient signal exhibits an observable transition corresponding to the cracking of the coating. This transition can be accurately evaluated by fitting straight lines to the two portions of the data, as illustrated in Figure 1(a) and (b). The cracking of a hard coating can originate from a surface crack or an internal (subsurface) crack. Both of these types of cracks can occur in some systems at very low loading values, as illustrated in Figure 1(b). This phenomenon elucidates the possibility of catastrophic coating failure during the second pass of a multi-pass scratch test, even when the constantly applied load is only a small fraction (< 40%) of the known critical failure load as defined by a progressive loading test.

4. CONCLUSION

A comparison of the two graphs revealed that the incorporation of an anchor layer resulted in a substantial enhancement in tribological performance. As demonstrated in sample (b), a more constant friction characteristic and stable behavior under identical load and displacement conditions are exhibited. This result indicates the effectiveness of the metallic interface layer in optimizing adhesion and mechanical strength. The presence of the anchor layer facilitated a seamless transition, thereby maintaining a more stable COF even after penetration of the ceramic layer. This discrepancy underscores the significance of the anchor layer in applications subject to recurrent or substantial mechanical stresses.

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