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Fatigue and mechanical properties of nanostructured and conventional titania (TiO₂) thermal spray coatings

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Abstract

Nanostructured and conventional titania (TiO₂) coatings were thermally sprayed using air plasma spray (APS) and high velocity oxy-fuel (HVOF) processes. The fatigue and mechanical properties of these coatings were investigated. A previous study characterized these coatings by scanning electron microscopy (SEM) to investigate the microstructural features and Vickers indentation to determine the hardness and crack propagation resistance. This current complementary SEM work examined fracture surfaces of fatigue-tested samples to assess crack nucleation and to study mechanisms of deformations. The fatigue strength of coatings deposited onto low-carbon steel (AISI 1018) showed that the nanostructured titania coated specimens exhibited significantly higher fatigue strength compared to the conventionally sprayed titania. The strain–time curve of AISI 1018 coated with TiO₂ indicated that the strain amplitude of the HVOF-sprayed nanostructured TiO₂ coating was much lower than the corresponding data of APS conventional TiO₂. SEM analysis of fracture surfaces revealed valuable information regarding the influence of these coatings on the performance of the coated component. This investigation gives new understanding to the nature of fatigue and deformation of the nanostructured and conventional titania (TiO₂) coatings.

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Keywords: Fatigue; Nanostructured coatings; Mechanical properties; Thermal spraying; Titanium oxide (TiO₂); Strain–time-curve

1. Introduction

Nanostructured materials and coatings offer the potential for significant improvements in engineering properties based on the improvements in physical and mechanical properties resulting from the reduction of microstructural features by factors of 100 to 1000 times compared to present engineering materials. Nanostructured materials can have improved strength, hardness, ductility, toughness, specific heat, and enhanced diffusivity compared to conventional materials [1,2]. Nanostructured thermal spray ceramic oxide coatings, such as Al₂O₃–TiO₂ and TiO₂, have demonstrated superior wear performance compared to related coatings produced from conventional ceramic oxide powders [3,4].

Titania (or titanium dioxide, TiO₂) powder is used to produce hard, dense coatings that resist wear by abrasion or motion. Titanium dioxide can be used for advanced applications such as solar cells, fuel cells, and catalysts [2]. Titania coatings such as these have been applied via air plasma spray (APS), the traditional method of depositing ceramic thermal spray coatings.

The air plasma spray (APS) process has been used for many years to form TiO₂ and other ceramic oxide coatings. This APS process is preferred due to the high temperature of the plasma jet [5], which is necessary to thermal spray high melting point materials in an effective way. During the thermal spraying of ceramic oxides it is necessary to totally or partially melt the powder particles to make coatings. However, TiO₂ is a ceramic material that has a relatively low melting point (1855 °C) [6] and it can be thermally sprayed via the high velocity oxy-fuel (HVOF) process [1,7], which is a

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technique that exhibits relatively low jet temperatures ($<3000\text{ }^{\circ}\text{C}$) but high velocities [5]. Therefore, HVOF spraying is an alternative method to thermal spray TiO_2 to engineer new types of microstructures.

This paper is a continuation of a previous work [1] demonstrating that HVOF-sprayed nanostructured titania coatings exhibited superior mechanical behavior compared to APS and HVOF-sprayed conventional titania coatings. It was observed that coatings exhibited similar values of Vickers hardness (about 800 Vickers at a 300 g load), however, the crack propagation resistance (or relative toughness) of the HVOF-sprayed nanostructured titania coating was almost 2 times higher than that of the APS conventional coating and approximately 1.6 times higher than that of the HVOF-sprayed conventional coating. It was proposed that the semi-molten nanostructured particles embedded in the coating microstructure were acting as crack arresters [1], thereby increasing the toughness of the material. Abrasion wear tests (ASTM G65) [8] indicated that the volume loss of the APS conventional titania coating was 2.4 times higher than that of the HVOF-sprayed nanostructured titania under the same wear conditions. When HVOF-sprayed nano TiO_2 and APS nano TiO_2 coatings were compared in abrasion testing, the volume loss of the APS coating was 1.7 times higher than that of the HVOF-sprayed coating, i.e., the HVOF coating was clearly superior [9], therefore APS nano TiO_2 coatings were not fatigue-tested. It was also found that the bond strength (ASTM C633) [10] of the HVOF-sprayed nanostructured titania on low-carbon steel substrates was 65% higher than that of the APS conventional titania and 2.4 times higher than that of the HVOF-sprayed conventional TiO_2 [1]. This data clearly show the mechanical superiority of the HVOF-sprayed nano TiO_2 coating when compared to those of other TiO_2 coatings.

An important observation was made during the crack propagation resistance measurements carried out via Vickers indentation (5 kgf) [1], namely, that the HVOF-sprayed coating exhibited an “isotropic” behavior, i.e., four cracks approximately equal in length emanated from the four corners of the Vickers indentation impression. Therefore, the HVOF-sprayed coating behaved in a similar fashion to the bulk material. These results demonstrated that the HVOF spraying of nanostructured TiO_2 powders was an efficient alternative coating method to the APS processing of TiO_2 .

It is widely recognized that thermal spray coatings can significantly influence the fatigue strength of coated components [11–13]. This paper presents the results of a fatigue and deformation behavior investigation of thermally sprayed nanostructured and conventional titania coatings deposited by different spray processes, specifically HVOF and APS. As previously stated, the mechanical performance and wear behavior of HVOF-sprayed nano TiO_2 coatings were much superior to those of conventional TiO_2 coatings sprayed by HVOF and APS, and nanostructured TiO_2 coatings sprayed by APS. It is important to point out that the APS processing of conventional TiO_2 represents the traditional method currently used to make TiO_2 by the majority thermal spray research centers, universities and industries. This research has the

objective to identify potential advantages of HVOF spraying nanostructured TiO_2 powders in lieu of APS processing of conventional TiO_2 for structural applications, mainly the ones where a high performance coating is paramount. Therefore, to supplement the earlier work on wear behavior, fatigue studies were carried out to further compare the structural performance of HVOF-sprayed nano TiO_2 coatings to that of APS conventional TiO_2 . In this investigation the air plasma spraying of conventional titania represents the current and traditional method of ceramic coating application; whereas, the HVOF spraying of nanostructured titania discussed in this current work can be considered an alternative method for applying this material.

2. Experimental procedure

2.1. Feedstock powders

Nanostructured and conventional TiO_2 feedstock powders were employed in this study. The nanostructured feedstock (VHP-DCS (5–20 μm), Altair Nanomaterials, Reno, NV, USA) was agglomerated and sintered. It exhibited a nominal particle size range from 5 to 20 μm , Fig. 1. This feedstock was HVOF-

Fig. 1. (a) Microscopic titania feedstock particle observed at low magnification. (b) Surface of (a) observed at high magnification—individual nanostructured titania particles agglomerated via spray-drying.

sprayed (DJ2700-hybrid, Sulzer Metco Inc., Westbury, NY, USA). The conventional TiO_2 feedstock powder (Metco 102, Sulzer Metco, Westbury, NY, USA) was fused and crushed and exhibited a nominal particle size range from 7.8 to 88 μm . This feedstock was APS processed (F4-MB, Sulzer Metco Inc., Westbury, NY, USA).

2.2. Thermal spraying

The HVOF spray parameters were set to produce the highest average particle temperature for each of the two feedstocks selected for the coating. The air plasma sprayed coating was produced on the basis of spray parameters recommended by the manufacturer of the torch and feedstock.

A cooling system of air jets was employed during the spray process to reduce the coating temperature, which was monitored using a single color pyrometer (wave lengths: 8–12 μm ; temperature range of 0–500 $^\circ\text{C}$). The pyrometer was previously calibrated by using a reference titania coating. The maximum coating temperature for the air plasma sprayed coating was approximately 150 $^\circ\text{C}$; whereas the HVOF-sprayed coatings were prepared at ~ 270 $^\circ\text{C}$.

2.3. Crack propagation resistance

The crack propagation characteristics were determined by indenting the coating cross-section with a Vickers indenter under a 5 kg load for 15 s, with diagonals of the indenter so aligned that one of these was parallel to the substrate surface. The length of the major crack, designated as “2c,” parallel to the substrate surface that originated at or near the corners of the Vickers indentation impression was measured. Based on the indentation load (P) and 2c, the crack propagation resistance was measured according to the relation between load and crack length, $P/c^{3/2}$ [14], where P is in Newtons and c is in meters. All indentations were performed very near the centerline of the cross-section and the average of five measurements was taken to assess the resistance of the coating towards crack propagation.

2.4. Fatigue testing

Rotating-beam fatigue testing was conducted on AISI 1018, low-carbon steel. The fatigue-testing machine is an RBF-200, rotating-beam fatigue machine (Fatigue Dynamics Inc., Walled Lake, MI) is shown in Fig. 2. The test specimen used in the fatigue testing (Fig. 3) was a 12.7 mm (1/2 in.) hourglass bar prepared according to ASTM E466 [15]. The nominal coating thickness was 100 μm (0.004 in.). The fatigue experiments were conducted at room temperature under a rotating beam and stress ratio of $R=-1$ configuration at a load frequency of 50 Hz. The surface of the specimen was prepared for coating by grit blasting with # 24 alumina, and no grinding was performed so as to not alter the surface roughness of the coatings. All fractured surfaces were subsequently examined using an optical microscope and scanning electron microscopy (SEM).

Fig. 2. Rotating-beam fatigue-testing machine, RBF 200.

2.5. Measurement of elastic properties of the coated specimen

For coated beams under applied bending moments or dynamic vibrations, the stiffness of the coated beam is an important factor in determining the strength of the component [16]. An interesting characteristic of the RBF-200 fatigue machine is that it can be used as a simple apparatus for carrying out static tensile bending test. A static cantilever beam bending experiments were performed on a round bars using the RBF-200 to determine the stress–strain curves. In these experiments, coated specimen (100 μm thickness) and uncoated specimens were used. Both specimens have a circular cross-section and having 12.7 mm (1/2 in.) diameter. One rosette strain gage mounted in 45 $^\circ$ orientations along the longitudinal axis of the beam was glued at the center of the specimen to measure the strain at this point.

The specimens were subjected to several bending moments through a poise weigh moving along the calibrated beam of the RBF-200. Starting from 10 in/lb and gradually increasing by 10 in/lb to the maximum load of 80 in/lb. The strains, ϵ , were measured by a digital strain indicator, P-3500 (Measurement Group, Inc., Raleigh, NC) The stress, σ was calculated using the flexural formula ($\sigma = MY/I$); here M is the applied bending moment, Y is the distance from the neutral axis to the surface of the specimen, and I is the moment of inertia. Young modulus E of the coated specimen as a system (substrate+coating) was determined by plotting the stress, σ vs. ϵ . Young modulus E of the substrate material (AISI 1018) obtained from this test was about 2.8% lower than the value published by the MatWeb network. The stress–strain curves obtained from this test are shown in Fig. 9.

2.6. Measurement of dynamic deformation of the coated specimen

An experimental method was designed and developed specifically to measure the dynamic deformation of the coated specimen. A slip ring device made by Michigan Scientific

Fig. 3. (a) Specimen configuration according to ASTM E466–96. The placement of the coating, (b), is indicated by the hatched lines.

Corporation (Charlevoix, MI, USA), which transfers electrical signals to strain gages, was coupled to both (i) the rotating bending machine and (ii) a stress analysis system. This arrangement permitted the dynamic strain (ϵ) to be monitored during the fatigue test. A constant stress of 355 MPa was maintained and the strain was measured as a function of time. The scanning rate was 10 points per second.

Fig. 4. (a) Cracking features emanating from a Vickers indent (5 kgf) on the cross-section of the APS coating produced from conventional feedstock. (b) Cracking features emanating from a Vickers indent (5 kgf) on the cross-section of the HVOF-sprayed nanostructured coating [1].

3. Results and discussion

3.1. Nanostructured morphology of the feedstock

Fig. 1a shows the SEM picture of a typical particle of the nanostructured TiO_2 feedstock. It has the typical donut shape morphology of spray-dried agglomerated particles. When the particle surface is examined at high magnification, Fig. 1b, each particle is observed to be formed from the agglomeration of individual nanostructured TiO_2 particles smaller than 100 nm. Therefore it was confirmed that this feedstock powder was nanostructured.

3.2. Crack propagation resistance

Fig. 4 [1] shows the typical crack indentation behavior of the coatings produced in this study. The cracking behavior of the HVOF-sprayed nanostructured coating (Fig. 4b) is different from that of the APS-sprayed conventional coating, Fig. 4a. The crack pattern induced by indentation of the nanostructured coating is characteristic behavior for an isotropic medium where four cracks with similar lengths propagate from the four corners of the Vickers indentation impression. The crack propagation resistance measures the relative toughness; therefore the HVOF-sprayed nanostructured coating exhibits higher toughness relative to that of the APS conventional coating. The results of crack propagation resistance were $28.4 \pm 1.4 \text{ MPam}^{1/2}$ and $14.8 \pm 1.6 \text{ MPam}^{1/2}$ for the HVOF-sprayed nanostructured and APS conventional coatings, respectively [1]. As additional information, the crack propagation resistance of the HVOF-sprayed conventional TiO_2 coating exhibited a value of $17.4 \pm 3.4 \text{ MPam}^{1/2}$. Therefore, the crack propagation resistance of the HVOF-sprayed nanostructured coating is almost 2 times higher than that of the APS conventional coating and approximately 1.6 times higher than that of the HVOF-sprayed conventional titania coating.

Fig. 5. Fatigue life distributions of AISI 1018 steel uncoated and coated with HVOF-sprayed nanostructured titania and APS conventional titania coatings.

Fig. 6. Fracture surface of AISI 1018 steel specimen (failed at 128,500 cycles) coated with HVOF nanostructured titania coating. Features indicated as “A” points to ductile tearing in the coating; while “B” features are indicative of the origin of cracks.

It was observed that semi-molten nanostructured TiO_2 particles embedded in the coating microstructure tended to arrest cracks formed during Vickers indentations [1]. Therefore, the superior crack propagation resistance of the HVOF-sprayed nanostructured coating was attributed to the presence of these semi-molten nanostructured TiO_2 particles. The amount of these semi-molten nanostructured particles embedded in the coating microstructure was estimated via X-ray diffraction (based on the percentage of the anatase phase) to be approximately 25% [17].

3.3. Results of fatigue tests

Fatigue life data generated in the fatigue tests were analyzed to determine the relationship between stress level, S , number of cycles to failure, N and probability of failure, P_f , for the samples tested. The failure probability [18], P_f , corresponding to the order number i is given by:

$$P_f = \frac{i}{n+1} \quad (1)$$

Fig. 5 shows the probability of failure (P_f) as a function of the number of cycles to failure (N) for the coated and uncoated specimens. The results indicate that the HVOF-sprayed nanostructured titania coating exhibited higher fatigue lives compared to the APS conventional titania coating. This result agrees with those obtained from the crack propagation resistance test, Fig. 4.

Fig. 5 also shows that the 3 sets of samples belong to different data populations. For example, if the slope of the uncoated sample can be taken as the base level of fatigue response, then the coated samples belong to more broad data distributions. The conventionally APS prepared ceramic appears to have reasonably consistent mono-modal character where the sample population is distributed about a mean value that is similar to a Gaussian distribution. On the other hand, the nanostructured material exhibits different behavior where (i) the sample population is broader, and (ii) the sample

population is bi-modal. The groups of samples that exhibit the lowest number of cycles to failure follow a gradient, and hence a population distribution, that is quite similar to the conventional ceramic. At the high end of the number of cycles to failure data, there is an indication of a quite broad distribution.

The inferences concerning the shape of the data sets presented in Fig. 5 are (i) the fatigue response can be attributed to different types of splat morphologies and (ii) a nanostructured ceramic coating has many morphological features that influence the overall fatigue lifetime of the coated component.

3.4. Characterization of the fracture surfaces

The fatigue-testing results were complemented with SEM characterization of the fracture surfaces. Fig. 6 shows the fracture surface of the nanostructured titania coating specimen that failed at $\sigma=355$ MPa, $N=128,500$ cycles. The nanostructured titania coating exhibits ductile tearing, feature A, a characteristic of bulk materials [19]. It is not common for thermal spray ceramic coatings to exhibit such characteristics. Ductile tearing consumes a significant amount of energy making the material “tougher”. It is also a clear indication of a coating with a relatively high elastic modulus (E).

Fig. 7 shows the fracture surface of the nanostructured titania coating specimen that failed at $\sigma=355$ MPa, $N=325,900$ cycles. This figure clearly shows that the HVOF-sprayed titania coating produced a uniform thin layer of compressive residual stress on the AISI 1810 steel substrate. Therefore, cracks initiated below this layer [11,20,21]. Consequently, the rate of fatigue damage decreased and the fatigue life increased. Hence, cracks started near the surface at inclusions and/or highly stressed locations. It is well known that thermal spray coating process imparts a residual stress in the surface layer of the coated component [22–24]. Compressive residual stresses are often deliberately induced in the surfaces of components, using a variety of surface enhancement methods, to improve fatigue life [25].

Fig. 7. SEM fractograph of fracture surface of steel specimen coated with HVOF nanostructured titania showing a layer of compressive stress indicated by an arrow, which was formed directly under the coating.

Fig. 8. (a) Fracture surface of AISI 1018 steel specimen coated with APS conventional titania coating. (b) High magnification image of (a). Small particles that are indicative of crushing of the conventional titania coating at the interface.

Fig. 8a shows the fracture surface of the APS conventional titania coated specimen that failed at $\sigma=355$ MPa, $N=73,800$ cycles. The fracture surface indicates poor adhesion between the coating and the substrate. When the hard coating

layer has weak adhesion strength, then it may detach from the substrate and any residual debris would promote cracking by a wedging action of particles at the interface. It was also reported that when the coating adherence strength is weak a small external stress or a variation in temperature will provoke coating detachment [26].

Fig. 8b shows a high magnification image of Fig. 8a and provides evidence of the crushing of the conventional titania coating at the interface. Fig. 8b reveals valuable information about the nature of deformation of these coatings. This coating clearly exhibited relatively lower elastic modulus (E) than that of the HVOF nanostructured coating.

These results are in good agreement with the previous results obtained from the crack propagation test [1]. In addition to the weak adhesion (interface bond), the conventional titania coating exhibited very low resistance to bending (tension or compression) deformation during the fatigue test. This material response is attributed to the low fatigue resistance exhibited by this type of coating.

3.5. Elastic modulus of the coated specimen as a system

The stress–strain curves obtained from a cantilever bending experiments are shown in Fig. 9. The nanostructured titania coated specimen indicated an elastic modulus that was 23% greater than the uncoated specimen. The conventional titania coated specimen exhibited a corresponding 10% increase in the elastic modulus compared to the uncoated specimen. It has been reported that the coating spallation resistance and the stiffness of the coated component are greatly affected by the coating elastic modulus [27].

In this investigation, an attempt has been made to show the effect of the starting powder and the spray process of the titania coating on the fatigue strength and stiffness (modulus) of the coated specimen as a system (substrate+coating). This investigation did not attempt to predict or estimate the modulus of the coating separately. Instead, comparing the

Fig. 9. Stress–Strain curves of AISI 1018 steel uncoated and coated specimens.

Fig. 10. Stress–time curves of AISI 1018 steel coated with APS conventional and HVOF nanostructured titania coatings.

fatigue strength and stiffness of steel specimens HVOF-sprayed with nanostructured titania and APS sprayed with conventional titania.

3.6. Strain–time curves

Durability issues of thermal spray coatings under high cyclic conditions are of major concern. It has been recognized that the coating failure driving force increases, whereas the resistance decreases, with time [28]. Fig. 10 shows the strain–time data of AISI 1018 coated with TiO₂. An initial observation is that these coatings are in compression. Furthermore, the average strain amplitude of the HVOF-sprayed nanostructured TiO₂ coating is ~0.26 and significantly lower than the ~0.77 observed for the APS processed conventional TiO₂. The HVOF-sprayed nanostructured TiO₂ exhibited steady-state deformation during the test. The strain–time curve for the APS-sprayed conventional TiO₂ has a characteristic response. Initially, as the load was applied, the elastic strain for the APS conventional TiO₂ was relatively small. Then, after some time under constant stress, the rate of strain increased by a large degree.

The strain amplitude is an indirect measure of the stiffness and indicates the fatigue resistance of the coated specimen. Higher strain amplitude has a detrimental effect on the fatigue life of the specimen [28,29]. It is also important to note that the crack resistance is a function of the plastic behavior of the material and of its fracture characteristics.

The fatigue strength of thermally sprayed components can be considered as a combination of coating and substrate performance. A coating with a high modulus of elasticity (E) is stiffer and therefore will bear a greater load than one made from a coating with a lower E . A coating system with a compressive residual stress in the substrate will improve the fatigue strength of the substrate. These concepts are in agreement with the experimental results obtained in this paper.

4. Conclusions

The fatigue and mechanical properties of HVOF-sprayed nanostructured and APS conventional titania (TiO₂) coatings were investigated. The significant increase in the fatigue strength of the HVOF-sprayed nanostructured titania coated steel specimen was created by: a) the ability of the high elastic modulus coating to carry a relatively large portion of the load, b) the compressive residual stress imparted by the coating application process. The HVOF-sprayed coating produced a uniform layer of compressive residual stress on the AISI 1810 steel substrate. These characteristics were the result of the process of HVOF spraying. The fatigue strength results can also be correlated with the crack propagation resistance, which for the HVOF-sprayed nanostructured coating is almost 2 times higher than that of the APS conventional coating and approximately 1.6 times higher than that of the HVOF-sprayed conventional titania (according to previous findings). Therefore, the enhanced fatigue strength of the HVOF-sprayed nanostructured coating is caused by the combination of employing (i) a

nanostructured feedstock and (ii) the process of HVOF. The strain–time curve of AISI 1018 coated with TiO₂ showed lower strain amplitude for the HVOF-sprayed nanostructured TiO₂ coating compared to the APS-sprayed conventional TiO₂, i.e., the strain amplitude of the coated component can be used as an indirect measure of the stiffness and indicates the fatigue resistance of the coated specimen. Consequently, the results obtained during this work continue to demonstrate that in order to engineer titania coatings with enhanced mechanical performance, a very viable alternative to the traditional method of spraying conventional ceramic powders by APS is the HVOF spraying of nanostructured ceramic feedstock materials.

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