QUASI-STATIC AND DYNAMIC TORSION TESTING OF CERAMIC MICRO AND NANO-STRUCTURED COATINGS USING SPECKLE PHOTOGRAPHY

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1. Abstract

An experimental methodology for the quasi-static and dynamic testing of Al_2O_3/TiO_2 and WC/Co coatings on aluminum substrates is presented. A stored energy Kolsky bar apparatus was used for both quasi-static and dynamic loading. Acquisition of speckle images at different loading stages allowed the determination of the shear strains in the gage region of the specimen and the identification of deformation mechanisms as a function of coating grain size. The interaction between coating and substrate is investigated by correlating damage to measured stress-strain curves. It was identified that as cracks develop in the coating, they are bridged by the underlying ductile substrate. Optical and SEM interrogation of the coating fracture surface also show a low percentage of porosity and severe damage created by residual stresses. These residual stresses arise during the coating manufacturing process. The pre-existing damage is believed to be the cause of the low shear moduli and strengths exhibited by the coatings. A comparison between quasi-static and dynamic response is carried out, to assess strainrate effects, as well as micro versus nano grain size coatings of the same composition.

2. Introduction

Ceramic coatings are employed mainly as wear resistant materials in a variety of applications, including mining, grinding and metal cutting. Recent developments towards nano-structured cermets showed that reduction in grain size leads to enhanced mechanical properties [1],[2],[3],[4],[5].

The objective of this work was to determine mechanical properties for nano-structured and micro-structured Al_2O_3/TiO_2 and WC/Co coatings, and more specifically shear

modulus and strength from static and dynamic torsion tests. The coatings were sprayed on an aluminum 6061-T6 substrate. The experimental procedure combined different elements: The Kolsky bar [6], which was used for both static and dynamic tests, high speed photography and speckle correlation technique for the determination of strain fields as well as localized deformations. To get a full picture of the mechanisms of deformation and failure, optical microscopy and SEM were used to investigate the microstructures of the coatings in an attempt to relate them to the observed mechanical behavior.

3. Specimens

The specimens were composed of an aluminum 6061-T6 substrate on which a coating was deposited by spraying (Figure 1).



Figure 1: Schematic of the torsion specimens used in this work

The spraying procedure was performed by A&A (A&A Company, South Plainfield, NJ). The substrates were blasted with aluminum oxide grit before the deposition process to ensure better adhesion. Al_2O_3/TiO_2 coatings were made using a plasma deposition technique; WC/Co coatings were made by High Velocity Oxide Fuel (HVOF). For all materials, both nano and micro-structured specimens were obtained. At the end of the coating deposition, the specimens were cooled down in air without any post-treatment procedures.

4. Testing methodology

The torsion tests were performed on a Kolsky bar apparatus (Figure 2). The specimen was glued between input and output bars. A torque was applied on the bar and was transmitted to the specimen. For the quasi-static tests, a lever arm and a platen attached to it were installed on the output bars, and the input bar was clamped. Weights were added on the platen to increase the torque by increments. The transmission of the torque to the specimen was verified using the strain gage station #1 (Figure 2). For the dynamic test, the torque was stored between the torque actuator and the clamp, and suddenly released by fracture of an aluminum pin. Upon release of the stored torque, a torsional stress wave traveled down the input bar to the specimen. For both quasi-static

and dynamic tests, pictures of the surface were taken at different loading stages. For the quasi-static test these pictures were triggered manually (at each load increment), and for the dynamic case the strain gage station #1, upon arrival of the wave, triggered the high-speed camera. Adequate timing allowed taking eight high-speed pictures at different stages of the rising pulse of the wave. Dark and bright features on the specimen surface were used to correlate the pictures with each other, allowing the determination of the strain in the specimen.



Figure 2: The stored-energy Kolsky bar apparatus [6]

Speckle correlation technique

The principle of the speckle technique is to correlate an "undeformed" image with a "deformed" image, using features present in the two images [7],[8]. These features constitute the speckle, a random distribution of dark and light features. They can be naturally present on the surface of the specimen (roughness, etched surface, etc.) or they can be created artificially (toner powder on white paint or lithography). The correlation procedure uses a reference picture of the speckle, and a "deformed" picture where the speckle has been distorted by deformations. The following numerical scheme is used [7],[8]: consider a square subset in the undeformed, reference image, and a point P, of coordinates (x,y), located inside this subset. After deformation, the subset may have moved to a new location and P moved to P*, of coordinates (x,y*), viz.,

$$x^* = x + u + \frac{\partial u}{\partial x} \Delta x + \frac{\partial u}{\partial y} \Delta y$$
$$y^* = y + v + \frac{\partial v}{\partial x} \Delta x + \frac{\partial v}{\partial y} \Delta y$$
(1)

(1)

Where *u* and *v* are the horizontal and vertical components of the displacement of the center of the subset, and δx and δy are the distances from the point (x,y) to the subset center. Using the dark and light features of the subset, in other words the gray level values F(x,y) in the undeformed subset and $G(x^*,y^*)$ in the deformed subset, the goal is to find the values of *u*, *v*, $\delta u/\delta x$, $\delta u/\delta y$, $\delta v/\delta x$ and $\delta v/\delta y$ that satisfies the best match between undeformed and deformed subsets. A way to do so is to minimize the correlation function *S*:

$$S\left(u,v,\frac{\partial u}{\partial x},\frac{\partial u}{\partial y},\frac{\partial v}{\partial x},\frac{\partial v}{\partial y}\right) = 1 - \frac{\sum \left[F(x,y) \cdot G(x^*,y^*)\right]}{\left[\sum F(x,y)^2 \cdot \sum G(x^*,y^*)^2\right]^{1/2}}$$
(2)

A Newton-Raphson scheme is then used to optimize *S*. Because of the discrete nature of a digital image, the resolution is limited by the size of the pixel. In order to increase this resolution, the gray values of the subset are fitted with a function, typically a cubic B-spline. Grey values between pixels can therefore be interpolated, and the search is performed using this function. This procedure allows the determination of sub-pixel displacements between two images. Once the displacements and deformations of the first subset are found, another subset is taken in the undeformed image and the same procedure is repeated. This allows the determination of a full field displacement between the undeformed and deformed images. The correlations were performed using the software VIC 2D (Video Image Correlation Package for 2-dimensional problem. © Correlated Solutions, 1998).

Validation tests on aluminum substrates

Aluminum substrates (without coatings) were tested in the first place using this method. Because of the smooth, shiny aspect of the surface of the specimen, the speckle pattern was created artificially, spraying toner power on a thin layer of white acrylic paint applied on the surface of the gage. Both quasi static and dynamic tests were performed. An example of displacement field given by the correlation method is shown in Figure 3. The pictures were taken using the high-speed camera during a dynamic torsion test on aluminum 6061-T6. The subset size used was 25x25 pixels, and correlations were performed every 5 pixels. The observed displacements are due to deformations of the specimen, but also to mechanical drift in the acquisition system (rigid body motion). Only the relative displacements (strains) are of interest in this test, and the results show an almost perfect state of pure shear in the specimen. From the displacements the shear strains were determined. Figure 4 shows a plot of the vertical displacement V as a function of the horizontal position x, along different horizontal lines. From this plot, the shear strain γ_{xy} is simply given by the slope of these curves.

The shear stress in the gage was computed from the torque using a thin wall approximation (constant stress throughout the thickness of the gage), namely,

$$\tau_{xy} = \frac{2T}{\pi D^2 t}$$

where T is the applied torque, D is the diameter of the specimen through the centerline, and t is the wall thickness. For the quasi-static tests the torque is well known and related to the weights on the platen of the lever arm.



Figure 3: displacement profiles (U: left, V: right) of the aluminum surface when loaded in torsion dynamically



Figure 4: displacement V along several horizontal lines

For the dynamic test, the torque is measured by gage station #1. Accurate synchronizations between load pulse and images allow correlation between torque and each high-speed picture, as shown on Figure 5. The resulting quasi-static and dynamic stress-strain curves are shown in Figure 6. They exhibit properties are consistent with literature values for aluminum 6061-T6; shear modulus G=25 GPa and yield shear stress τ_y =150 MPa. The yield strength for the dynamic case is slightly higher, which is also expected.

Figure 6 also shows the dynamic stress-strain curve obtained by the integration of the reflected pulse [6]. The shear stresses are computed in the same way as above, but the

(3)



shear strains are obtained by analysis of the incident, reflected and transmitted torques recorded during the dynamic test. Details of this approach can be found elsewhere [6].



Figure 5: Torque versus time plot obtained from dynamic torsion test performed on Al 6061 T6. Synchronization between the loading pulse and the high-speed pictures

Figure 6: Stress-strain curve from quasi-static and dynamic torsion tests performed on Aluminum 6061T6

These tests provided accurate stress-strain curves for the aluminum substrate. They also showed that the speckle correlation is very accurate, even when acquiring pictures through high-speed photography.

5. Results for Al₂O₃/TiO₂ and WC/Co coatings

The coated specimens were tested using the same procedure. The natural roughness of the surface of the coating provided the speckle pattern. Because the measured torque is due to both substrate and coating, it is necessary to subtract the torque in the substrate to obtain the torque in the coating. This approach relies on the assumption that the strain in the coating and substrate are identical. Figure 7 illustrates this operation, for a quasistatic test on a nano-Al₂O₃/TiO₂ coating. The strains were determined by the speckle correlation method. From the total torque, the torque produced by the aluminum substrate (obtained in the previous test) is subtracted to obtain the torque of the coating alone.



Figure 7: Torque-strain curves for aluminum + coating, aluminum alone and coating alone

Using Eq. (3) the shear stress in the coating is computed from the torque. Figure 8 shows the resulting stress-strain curve for the nano-Al₂O₃/TiO₂ coating. The curve exhibits a linear behavior up to 50 MPa, and then shows a strain hardening like behavior. Further investigation on the displacement fields reveals that this behavior cannot be attributed to the coating alone. Figure 9 shows a sequence of pictures and displacement fields at different loading stages. Figure 9a shows the displacement field in the linear elastic region. The displacements reveal inhomogeneities in the coating. Furthermore, in this article, it is shown that they are likely related to initial defects in the specimen. Figure 9b shows the displacement fields in the plateau region of the stress-strain curve. Although no crack or damage is visible in the picture, the displacement fields, measured by interrogating the speckle, reveal at least two cracks running at about 45° . Either these cracks are underneath the surface or the crack opening is so small that they cannot be seen in the pictures. The underlying aluminum substrate

bridges these cracks, which prevents the specimen from catastrophic failure. Figure 9c confirms the locations of the cracks. The figure shows the last frame taken before total failure of the specimen. The image clearly shows the two cracks with a significant opening. At this point the assumption of having the same strain in both coating and substrate does not hold and, therefore, the strain hardening like part of the stress-strain curve is computed under inhomogeneous deformations.



Figure 8: Quasi-static stress-strain curve for nano-Al₂O₃/TiO₂ coating. The 20 GPa shear modulus line is also shown.

All of the coatings, micro and nano grain size, exhibited the same type of behavior in both quasi-static and dynamic loading.

Figure 10 shows stress-strain curves obtained from the tested specimens, in both quasistatic and dynamic mode. The shear moduli were in the 20-25 GPa range for Al_2O_3/TiO_2 and 30 GPa for WC/Co. It should be noted that these moduli are well below the theoretical values as a result of the initial microcraking present in the coatings, which is later shown by means of microscopy. The shear strengths did not exceed 60 MPa. These values are below the reference values for these materials. This must be attributed to porosity in the coatings, as well as internal damage created by residual stresses during the coating process. Figure 11 shows micrographs of a micro- Al_2O_3/TiO_2 specimen, which was not tested. The specimen was saw cut in the middle of the gage and the surfaces were polished. These pictures reveal porosity in the coating, as well as severe internal damage resulting from residual stress build-up during the spray deposition process. The coating is debonded from the substrate and cracks are present in both circumferential and radial directions. The circumferential cracks, running parallel to the coating-aluminum interface, are much less detrimental to the coating torsional strength than the radial cracks.



a: linear elastic region (τ =20 Mpa)



b: displacements U and V in the plateau region (τ =55Mpa)



c: final picture before catastrophic failure of the specimen

 $\label{eq:Figure 9: Picture and displacement fields at different loading stages for a quasi-static torsion test performed on nano-Al_2O_3/TiO_2 coating$

Figure 12 shows an optical micrograph and an SEM picture of a nano-WC/Co specimen. The same kind of defects and damage is observed. The degree of initial damage varied somewhat from specimen to specimen. We tested those that exhibited the least amount of initial damage and no apparent radial cracks.



Figure 10: Stress-strain curves from torsion tests performed on micro and nanostructured coatings.



Figure 11: Optical micrographs of a cross section of a micro-Al₂O₃/TiO₂ coating (A) on aluminum substrate (B).



Figure 12: Optical micrograph and SEM of a cross section of a nano-WC/Co coating (A) on aluminum substrate (B).

6. Conclusions

A methodology for quasi-static and dynamic torsion testing of micro and nanostructured coatings was presented. The Kolsky bar apparatus was used, as well as highspeed photography and the speckle correlation method. Experiments conducted on aluminum 6061-T6 demonstrated the accuracy of the method. Results on nano and micro-structured Al_2O_3/TiO_2 and WC/Co coatings were obtained. The displacement field given by the speckle correlation technique allowed the identification of the deformation mechanism, i.e., early crack propagation and crack bridging by the ductile aluminum substrate. No significant difference in modulus and strength was found between the nano and micro-structured materials, but an eventual difference between them is probably masked by the initial damage in the coatings, as observed in optical and SEM pictures.

Manufacturing coatings free of initial microcracks remains a significant challenge. Research on optimization of the spray deposition parameters should be pursued to produce high quality nanostructured coatings that can fully exploit the benefits of nanosize grains.

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