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(54) **DETERMINATION OF ADHESION STRENGTH OF HVOF COATING BY SPHERICAL INDENTATION**

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(52) **U.S. Cl.** **73/81**

(58) **Field of Search** 73/774, 762, 775,
73/790, 78, 81

(56) **References Cited**

U.S. PATENT DOCUMENTS

4,232,559 A 11/1980 Favre et al.

4,263,811 A	4/1981	Shaw	
4,501,154 A	2/1985	Mori	
4,606,225 A	8/1986	Thomason et al.	
4,856,326 A *	8/1989	Tsukamoto	73/150 A
6,050,140 A	4/2000	Koch	
6,339,958 B1 *	1/2002	Tsui et al.	73/290 R
6,377,039 B1 *	4/2002	Goldfine et al.	324/232
6,414,506 B2 *	7/2002	Akram et al.	324/765

OTHER PUBLICATIONS

International Search Report for parent International Application, PCT/US00/09940, filed Apr. 13, 2000, earliest priority Apr. 13, 1999, entitled: Determination of Adhesion Strength of HVOF Coating By Spherical Indentation; Search Report Dated Jul. 31, 2000.

* cited by examiner

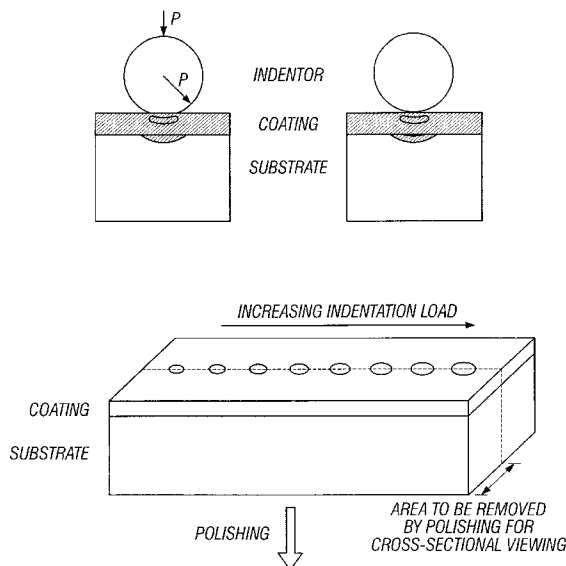
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(57) **ABSTRACT**

A method for determining the adhesion strength of any HVOF-coating/substrate system. This method applies a spherical indenter to the coated part in a loading-unloading cycle. During the loading phase, the substrate deforms plastically. During the unloading phase, the elastic-plastic mismatch between coating and substrate material properties gives rise to a tensile stress normal to the interface. This leads to delamination for a sufficient indentation load. A hybrid numerical-experimental approach calculates the interfacial adhesion. The threshold load for delamination is found experimentally. A finite element model calculates the corresponding adhesion stress for delamination. This method enables the quantitative evaluation of the adhesion for any coating/substrate interface, regardless of bond strength.

7 Claims, 4 Drawing Sheets



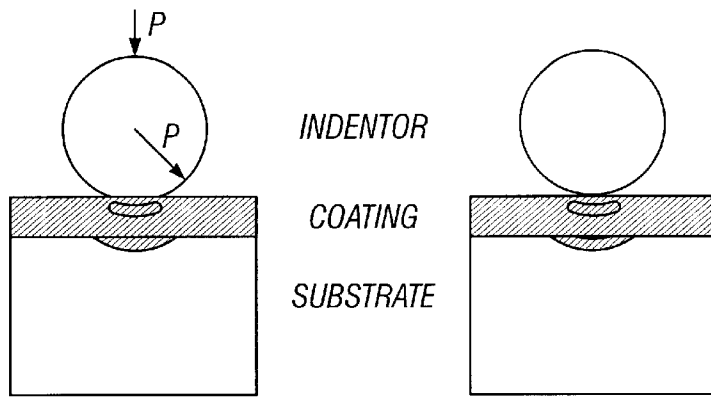


FIG. 1

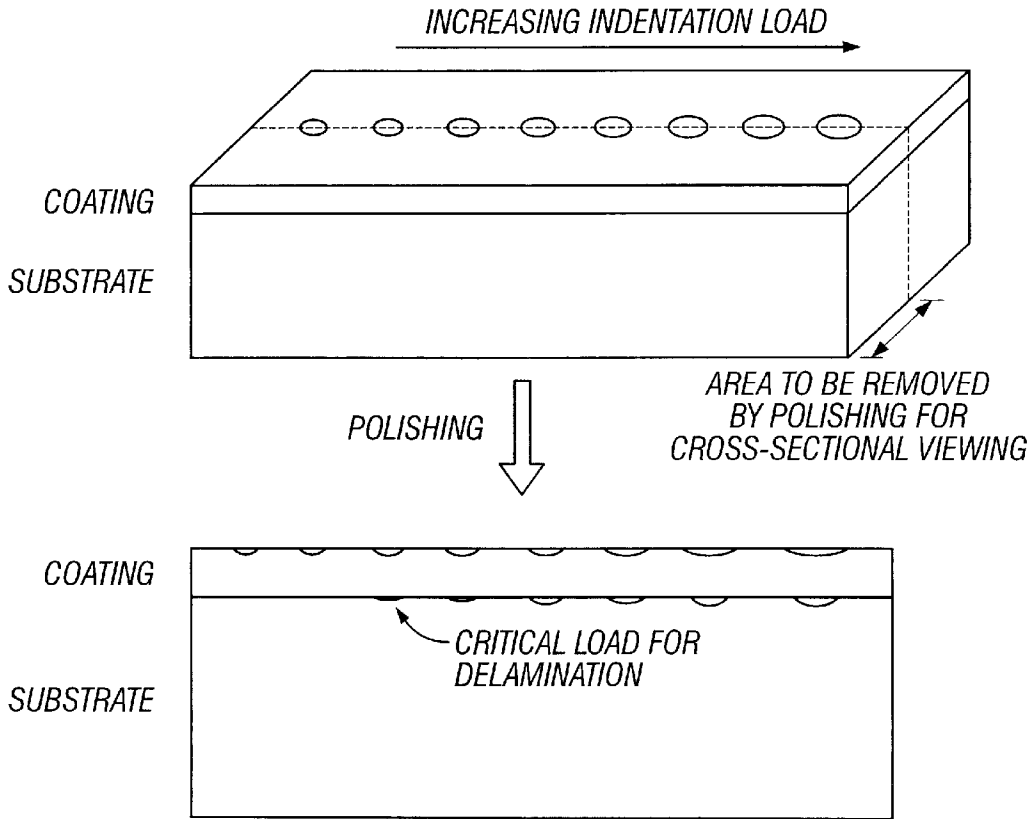


FIG. 2

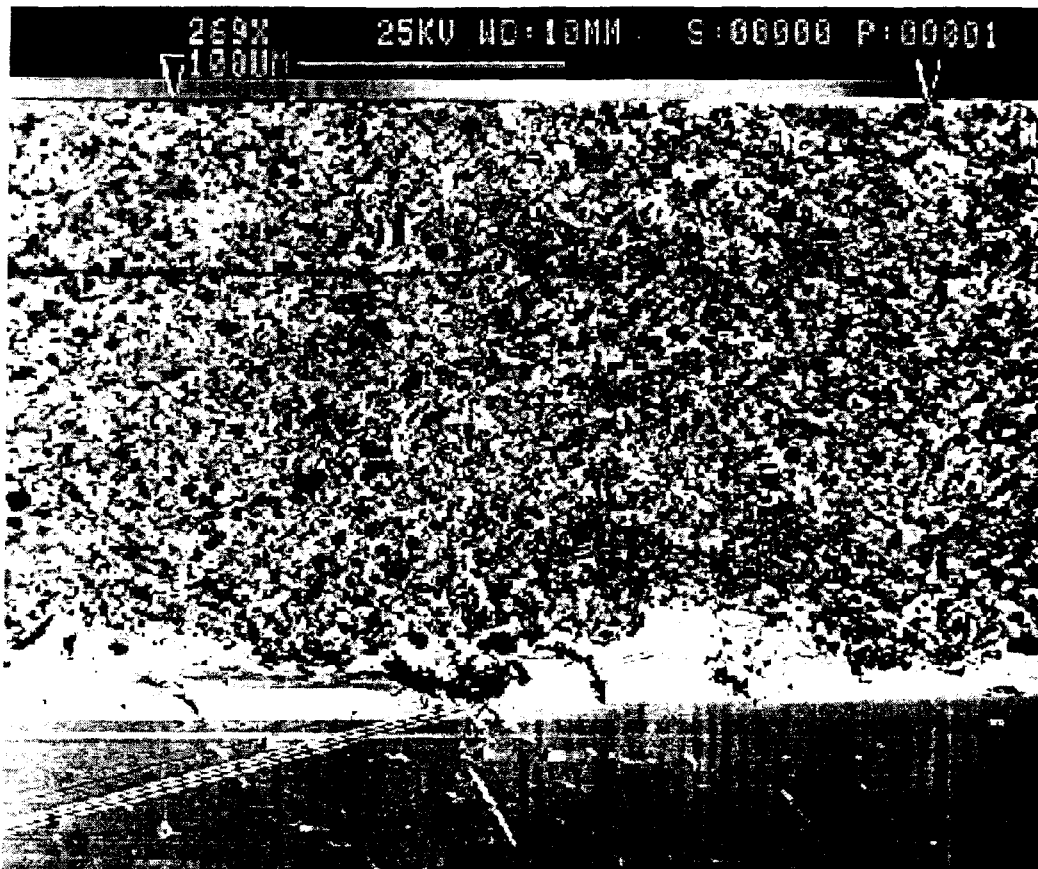


Figure 3: Interfacial delamination nucleated by the tensile residual stress. The two arrows-heads show the contact region

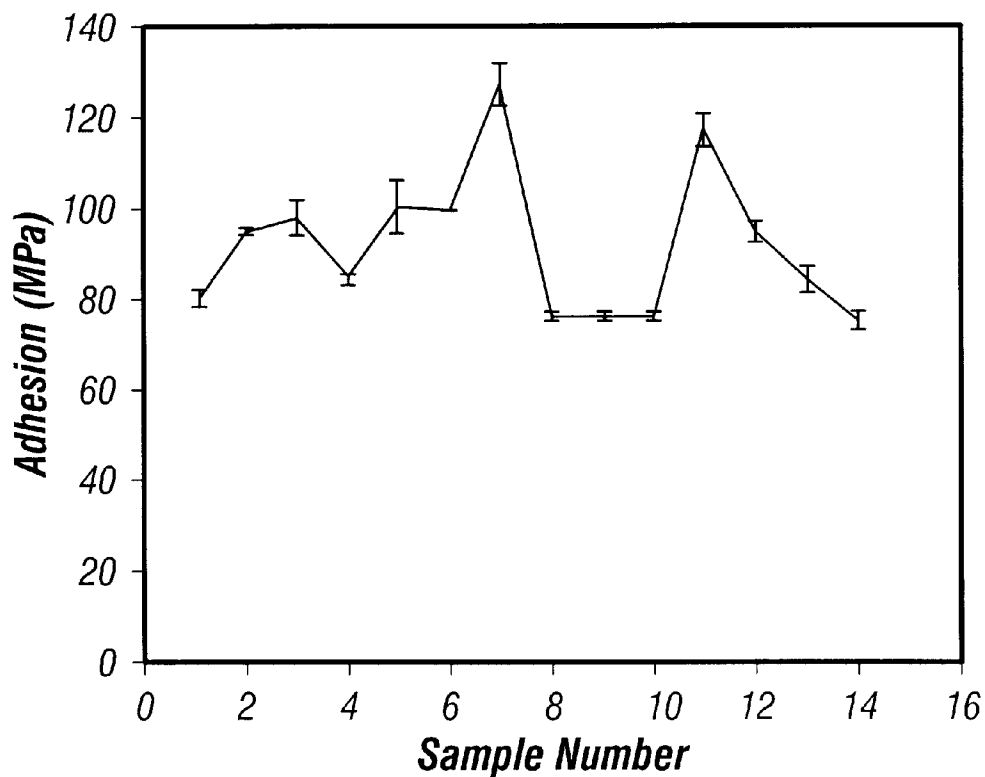


FIG. 4

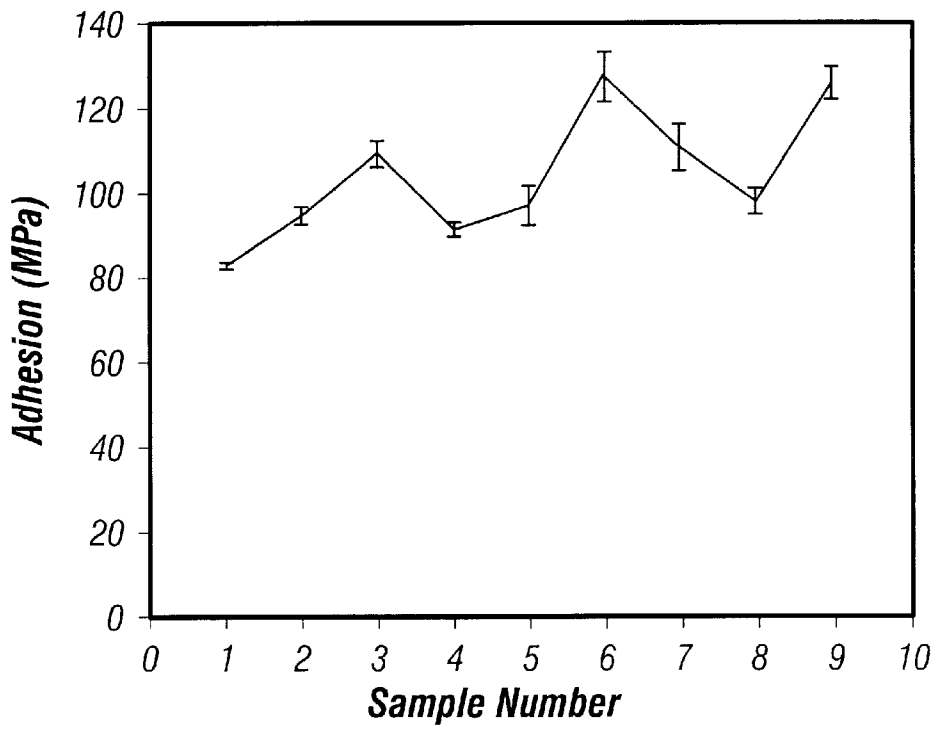


FIG. 5

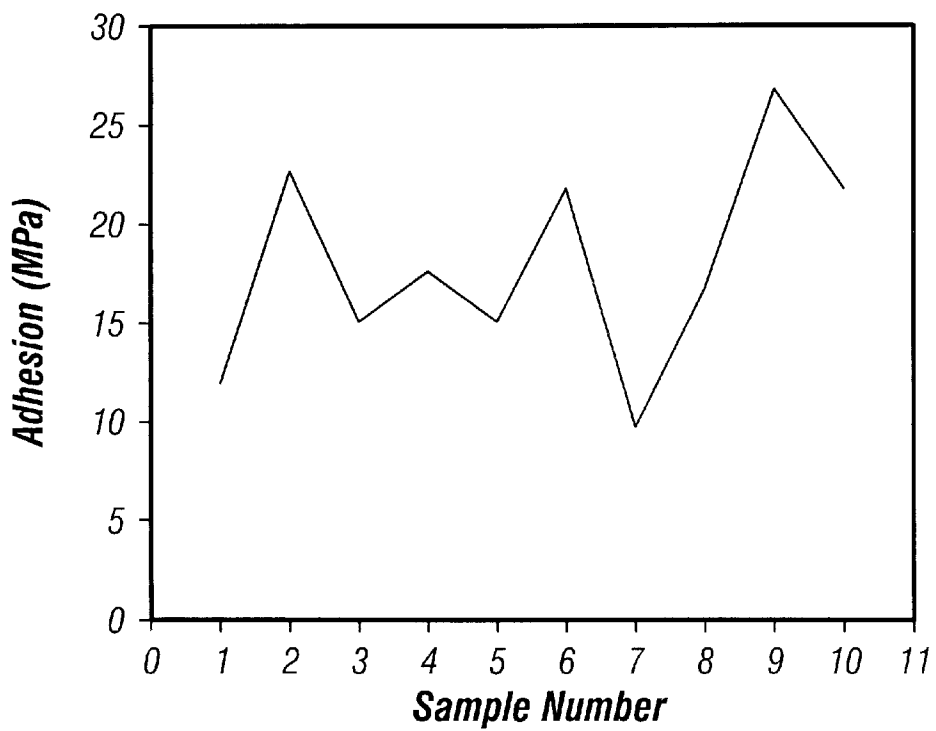


FIG. 6

DETERMINATION OF ADHESION STRENGTH OF HVOF COATING BY SPHERICAL INDENTATION

This application claims the benefit of Provisional appli- 5
cation Ser. No. 60/129,115, filed Apr. 13, 1999.

TECHNICAL FIELD

This invention relates to the field of methods to determine 10
adhesion properties of coatings, and, more particularly, to
high adhesion strength HVOF (High Velocity Oxygen-Fuel)
thermal sprayed coating.

BACKGROUND OF THE INVENTION

HVOF (High Velocity Oxygen Fuel) thermal sprayed 15
coatings are often used to extend the service life of metal
parts in harsh environment. In order to determine the viabil-
ity of the coatings, it is necessary to determine the adhesive
strength of the coating.

Therefore, a standard ASTM test (C633-79) has been 20
developed for this purpose. In this standard test, termed
Tensile Adhesion Test, a coated specimen is attached to a
support fixture of a tensile testing machine by epoxy. A
tensile force is then applied by the machine until the coating 25
fails. The stress at failure is called the tensile adhesion
strength or bond strength.

However, some of the HVOF coatings exhibit higher 30
adhesion strength than epoxy glue such that the standard test
leads to delamination of the epoxy instead of the coating.

Therefore, there is a need for a testing method that will 35
determine the adhesion strength of coatings that exceed the
capability of the ASTM C633-79 standard test.

SUMMARY OF THE INVENTION

The present invention is a technique that determines the 40
adhesion strength of any HVOF-coating/substrate system by
spherical indentation. We emphasize that while we have
characterized HVOF coating/substrate system, and demon-
strated the test for this case, the test method is generally 45
applicable to all coatings, regardless of the method used to
obtain the coating. The principle of the method is illustrated
in FIG. 1. The coated part is subjected to a loading-
unloading cycle using a spherical indenter. During the
loading phase, the substrate deforms plastically and during 50
the unloading phase, the elastic-plastic mismatch between
coating and substrate material properties gives rise to a
tensile stress normal to the interface that will lead to
delamination for a sufficient indentation load. A hybrid
numerical-experimental approach is used to calculate the
interfacial adhesion; the threshold load for delamination
is found experimentally and a finite element model is used
to calculate the corresponding adhesion stress for delamina-
tion.

The present invention enables the quantitative evaluation 55
of the adhesion for any coating/substrate interface, regard-
less of bond strength, while the prior art can only evaluate
interface strengths of a bond strength less than that of the
adhesive used for bonding the two ASTM samples.

LIST OF FIGURES

FIG. 1 is a schematic of the principle of the method;
FIG. 2 is a schematic of the experimental procedure for 60
the method;
FIG. 3 is a micrograph of the sample at the critical load
value;

FIG. 4 is a graph showing the adhesion strength of
RAM31 on 17-4 PH;substrate1;

FIG. 5 is a graph showing the adhesion strength of
RAM31 on 1 7-4PH;substrate2;

FIG. 6 is a graph showing the adhesion strength of
RAM49 on 17-4PH;substrate2.

DETAILED DESCRIPTION OF THE PROCEDURE

The method that has been developed is a hybrid 10
numerical-experimental method, as detailed here:

Experimental Determination of Critical Load (See Schem-
atic of FIG. 2)

- a) A row of indents are mechanically placed on the 15
deposited coating, each with an increasing normal load.
- b) The specimen is sectioned and polished carefully up to
the fictive line corresponding to the centers of the
indents.
- c) Optical microscopy methods are used to identify the 20
first appearance of coating delamination associated
with one of the indents.
- d) The corresponding peak indentation load is taken as the
critical load. The critical load is the lowest indentation
load at which an interfacial delamination is seen in the
polished specimen.

Numerical Determination of Interfacial Adhesion Stress

- a) The spherical indentation experiment is modeled using 30
finite element software. The geometry is represented
through appropriate FEM meshing, including the
spherical indenter in contact with a coated specimen.
- b) The coating is represented as an upper layer of thick-
ness corresponding to that of the real coating. The
material properties of the coating and the substrate
must be incorporated into the model.
- c) The critical load found experimentally (see above) is 35
now input to the finite element model. Displacement
rates in this model faithfully simulate those from the
experiment, as determined from the universal load
frame.
- d) The adhesion strength at the interface is taken as the
normal tensile residual stress at the interface given by
the model. We have determined that the contribution of
shear stresses to delamination is negligible due to the
topography typical of the grit-blasted substrate surface.
It must be assured by microscopic observation that
delamination occurred under tension and not by shear.
This can be done by determining that the delamination
was under the center of the indenter and not below the
edge of the contact zone

EXAMPLES

The inventors used three different systems for the present 55
invention. All the substrates were grit-blasted with alumi-
num oxide particles. The achieved roughness on the sub-
strate after grit blasting was 300 Ra. Thermal sprayed
coatings were then deposited using the HVOF technique.
Alloy Carbide Co., Houston, Tex., performed all coating
processes.

Two types of RAM (Rocket Applied Metallics) coatings 60
were tested:

- RAM31 whose composition is 80% Cr₃C₂/20% Ni-Cr
with an average thickness of 250 microns;
- RAM49 (Stellite®) whose composition is 67% Co, 28%
Cr, 4% W and 1% C with an average thickness of 350
microns.

Both coatings were applied over a 17-4 PH stainless steel substrate. For RAM31, the coating was applied on two different 17-4PH stainless steel samples of different yield stresses. Mechanical properties of materials used for all combinations are summarized in the table1 below.

TABLE 1

Material	Elastic modulus, E (GPa)	Poisson's ratio, ν	Yield stress, Y (GPa)	Hardening coefficient, n
17-4 PH; Sustrate 1	206.8	0.27	0.8	0
17-4 PH; Sustrate 2	206.8	0.27	1.2	0
RAM31 coating	96	0.3	1.4	0.4
RAM49 coating	60	0.3	1	0.4
WC-indentor	614	0.22	—	—

Except for the Young modulus of the coatings, all elastic moduli and Poisson's ratio values were taken from the literature.

The elastic modulus of the coatings; yield stresses and hardening coefficients for all materials were determined by using an indentation technique. Using a universal test machine, indentations at different loads were performed with a tungsten carbide spherical indenter. The loading rate is low, followed by a rapid unloading rate. The residual impressions were measured optically using Nomarski illumination techniques.

The Young's modulus of the coatings were deduced from a plot of the indentation stress versus indentation strain.

For the plastic properties, the numerical model developed for the determination of the adhesion was used. A standard incremental J2 plasticity theory, with the Von Mises yield and the Prandtl-Reuss flow rule as implemented in ABAQUS was used to model plasticity. The tungsten carbide indenter was considered as remaining elastic during the indentation experiment and therefore described as perfectly elastic. Coating and substrate were considered as elastic-plastic material exhibiting a strain-hardening behavior.

Plastic properties of both coating and substrate were obtained by comparing experimental and numerically determined results, where different values for yield stress and hardening coefficient were input. When comparing experimental and numerical results at several loads, the combination of yield stress and hardening coefficient matching the experimental data was considered as being the material properties.

Once the model is totally defined in terms of mesh geometry and material properties, several rows of indents with increasing normal loads were performed on the different combinations of materials. Along each row, indentations

were separated of 1 mm and the increment in load was 5 N. The rows were then polished carefully up to the row using a finishing abrasive paper grade of 800 in order not to damage the interface.

Once the material was sectioned through the center of the residual impressions, interface damage was observed with an optical microscope. Delamination is defined by the appearance of a crack of 25 microns or larger.

For each experiment, the finite element model was run with the indentation load corresponding to experimental delamination load. Adhesion strength of the coating/substrate interface was considered as being the simulated tensile residual stress normal to the interface along the centerline of the indentation.

Adhesion results for the different combinations are in FIGS. 4, 5 and 6.

What is claimed is:

1. A method for determining a coating bond strength between a coating and a substrate, comprising the steps of:

- a. determining experimentally a critical load for delamination due to unloading of a spherical indentation;
- b. performing an FEM simulation of an indentation cycle at an experimental critical load; and
- c. deducing an adhesion stress at the interface from the FEM simulation.

2. The method of claim 1, further comprising the step of determining Young's modulus by spherical indentation using the Hertz theory for elastic contact.

3. The method of claim 2, further comprising the step of modeling the plastic properties of both the substrate and the coating by matching experimental and numerical results.

4. A method for determining interfacial adhesion between a first and a second thick film coating, comprising the steps of:

- a. determining experimentally a critical load for delamination due to unloading of a spherical indentation;
- b. performing an FEM simulation of an indentation cycle at an experimental critical load; and
- c. deducing an adhesion stress at the interface from the FEM simulation.

5. The method of claim 4, further comprising the step of determining Young's modulus by spherical indentation using the Hertz theory for elastic contact.

6. The method of claim 5, further comprising the step of modeling the plastic properties of both the first and the second thick film coatings by matching experimental and numerical results.

7. The method of claim 6, wherein the thick film coatings are substrates.FIG. 1: principle of the method.

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