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OPPORTUNITIES AND NEEDS FOR STANDARDIZATION OF CERAMIC COATINGS

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Ceramic coatings have a large range of applications; for example to combat corrosive environments, to prevent high temperature oxidation, to minimize adverse wear, or to confer specific **electrical** insulation and conduction characteristics on a bulk material. All these properties are contingent on the coating adhering to the particular substrate and this property, in turn, is dependent on the methods which are available for its determination.

The coating integrity is also of importance since any coating failure will limit the overall **coating/substrate** usefulness. Coatings can be classified according to the coating process, the application and/or the coating thickness. The present work will emphasize the needs for appropriate standards of thick (>0.1 mm) ceramic coatings which may be applied by thermal spraying techniques.

1. INTRODUCTION

1.1 Ceramic coatings

Ceramic coatings have been available since antiquity; for example as glazes on pottery and, more recently, as enamels on steels. Such applications may be classified under a heading of "traditional uses". A set of traditional standards has evolved for these materials; and, for example, **colour**, coating adhesion, and toxicity are quite important design factors which must be controlled by the ceramics industry. The "material system"; that is, the combination of the ceramic coating and the substrate, needs an appropriate specification for the whole component to perform a particular task. The present paper refers to ceramic coatings which do not have the historical basis of traditional materials and this represents a challenge for materials technology.

Advanced applications for ceramic coatings have grown since the mid-1900's and these have evolved mainly due to aerospace and bioengineering challenges. The examples of manufacturing **zirconia-yttria** alloy coatings for thermal barrier applications¹⁻³, and of processing hydroxyapatite coatings⁴ for hip prostheses can be cited as recent challenges that the materials engineer has undertaken.

Coatings can be defined⁵ according to their thickness as either thin films (<1 micron), thick films (>1 micron) or as bulk coatings (>25 microns). The current work will

be related to examples of bulk coatings. The term 'standardization of coatings' implies that coatings which are produced under identical conditions will also exhibit the same material response; **whether** these be of a thermal, chemical or mechanical nature.

1.2. The formulation of standards

There are many types of standards which, for the same material, may be used for different purposes. The use of an engineering material often precedes any formal standard with respect to its intended application and thus it is only after the material has shown utility that a standard evolves. This standard may be formulated in response to several factors that are driven by either engineering or commercial enterprises. Another problem may be that a manufacturer or user finds it necessary to compare products and therefore a specification is written that will enable the relative merits of different products to be distinguished by a customer.

The three major types of standards which evolve are industrial, national and international; although it can be noted that major interest groups, such as the military, also specify their own acceptance criteria for many materials.

The above standards associations that have been formed may accept the standard from another group. However it is more common for a material to pass a regional standard in order to have acceptance in a particular economic community. Thus, for example, it may become necessary for a North American manufacturer to re-test their materials in order to "get acceptance" for a European market. Thus the passing of a material by a certain standard does not constitute a guarantee for the manufacturer that their product can have universal use. Nor does the attainment of some standard (which is issued by the appropriate body) for a **product** infer that the material will have acceptance by the consumer.

The upshot of the above comments is twofold; every standard must have a rigorous scientific basis to ensure international acceptance; and, the method which is detailed in the standard must be relevant with regard to the intended application of the material system. Therefore it would be useful for the standard to reproduce the service failure conditions of the material so that further development of the material system can be facilitated. The above idealized criteria will be expanded upon with particular reference to thermally sprayed coatings which are intended for high temperature applications.

2. STANDARDS FOR THE THERMAL SPRAY INDUSTRY

The present work will mainly refer to the standards of the American Society for Testing and Materials (**ASTM**)⁶ although a comparison between several standards societies will be presented with regard to measuring the adhesion strength of coatings. It is not intended to present a complete listing of the standards which apply to the thermal spraying industry; since such a list would only be of limited value.

The relevant standards which would be of assistance to the general thermal spraying community are grouped under the headings which are listed in Table 1. The standards which do not have an **ASTM** designation but which would be useful are also listed in this table. This information has been compiled by performing a hand search through the index section of the **ASTM** standards. In this fashion relational topics between fields can be distinguished; ie, the classification "coating adhesion" can be related to "adhesion" and "**cohesion**"; which in **turn** can be connected to "**wear**" and other topics. It is clear that there are many ASTM standards which are concerned with the field of coating technology. The summary in Table 1 can be used as a basis for further developments.

Some of the topics do not have an ASTM reference because the specific standard that was originally referenced is not pertinent to thermal spray coatings. In an alike fashion, other standards or specifications which would be of value but which are not published by the ASM are included in the table for completeness.

Table 1 indicates that the complete specification of a ceramic-substrate material system encompasses powder manufacture; thermal spray processing and testing of the coating; and reporting. It should be emphasized that all of these factors are interrelated. For example, a poor adhesion value for a particular ceramic coating may be traced back to, among other factors, incorrect powder size distribution, poor substrate preparation due to over-used grit media, incorrect thermal spraying parameters or poor testing procedures. It can be noted that any of these shortcomings in the coating may, in fact, still be acceptable for the intended application of the customer since the test method was inappropriate. A certain degree of interpretation of the measurement technique is fundamental in understanding the test result. The results of tests which are performed according to the standard test are rarely completely unambiguous.

The "need" for establishing reproducible material property measurements is therefore two fold. First, from the practical viewpoint, the availability of **self-consistent** properties will enable a reliable data base to be established and hence engineers will have a valuable resource at the design stage of a product. A second point is more fundamental since the adoption of a particular standard implies that an effort has been made at a basic understanding of physical phenomena occurring within the coating and at the **coating/substrate** interface. It is evident, from the above arguments, that appropriate standardization methodology for coatings presents opportunities to improve their performance by ascertaining basic **structure/property** relationships. It must also be emphasized that the adoption of any standard should not limit the potential uses of the material.

Several tests in the area of high temperature and mechanical properties of thermally sprayed materials will be discussed in detail.

TABLE 1. ASTM STANDARDS FOR THERMALLY SPRAYED COATINGS⁶

TECHNOLOGICAL DESCRIPTION	ASTM DESIGNATION
1. Powder specifications	
1.1 powder manufacture	
1.2 particle size	8214, E11
1.3 powder quality control	8212, 8213, 8214
2. Thermal spraying specifications	
2.1 spray tables	
2.2 substrate specification	
2.3 substrate preparation	
2.4 safety	
3. Testing of coatings	
3.1 coating continuity	C743
3.2 coating adhesion	C633
3.3 bond strength	C633, D3359
3.4 cohesion	
3.5 deformation	E6, E7, E8, E9
3.6 abrasion and abrasion resistance	B611, C158, C448
3.7 hardness (indentation)	E10, E18, E92
3.8 wear	B611
3.9 wear resistance	
3.10 wear life	
3.11 corrosion and corrosion resistance	G31, G32, G40, G52, G61
3.12 erosion	G32
3.13 thermal degradation and endurance	C385
3.14 thermal expansion	C824
3.15 thermal flux resistance	B571, C351
3.16 thermal cycling life	
3.17 high temperature oxidation resistance	G54
4. Coating characterization	
4.1 microstructure of coating	
4.2 porosity	C493, C699
5. Reporting recommendations	
5.1 statistical significance of test	G16
5.2 number of tests	
5.3 relevance of test to service conditions	
5.4 complete description of the coating system	

3. THERMAL PROPERTIES

The major thermal properties of interest are those of thermal expansion, resistance to thermal fluxes, hot corrosion behaviour and thermal diffusivity. These material **properties** are required by design engineers in order to compare the relative merits of thermal barrier coating systems. Much of this experimental work has not been formally

standardized. However it can be noted that many major laboratories do use quite similar experimental techniques.

3.1. Thermal expansion

It is believed that thermal expansion mismatch stresses between the coating and substrate lead to the progressive cracking and ultimate failure of coatings⁷. The thermal expansion phenomenon is, however, quite difficult to measure since the coating geometry is usually in the form of a thin (<1 mm) rectangular plate. The ceramic plate may, or may not, be attached to the substrate and the particular investigation may require the expansion coefficient of the coating or the **coating/substrate** system to be ascertained.

The thermal expansion behaviour of zirconia - **8wt%yttria** ceramics (Figure 1) is approximately linear with a regression coefficient greater than 0.9. The thermal expansion coefficient is about $10 \times 10^{-6} \text{C}^{-1}$ and does not change significantly upon repeated thermal cycling⁸. It has been shown that an absolute variance of less than 6%, and usually less than 2%, can be obtained for **zirconia-yttria** alloy coatings when measurements are performed in the in-plane orientation. The results for the **NiCrAlY** alloy bond coat material⁹ which is normally used in conjunction with this ceramic overlay are also presented. The implication from comparing these results is that there will be significant stress build-up at the interface between the bond coat and ceramic due to the differences in thermal expansion behaviour. Similar studies¹⁰ have been performed in

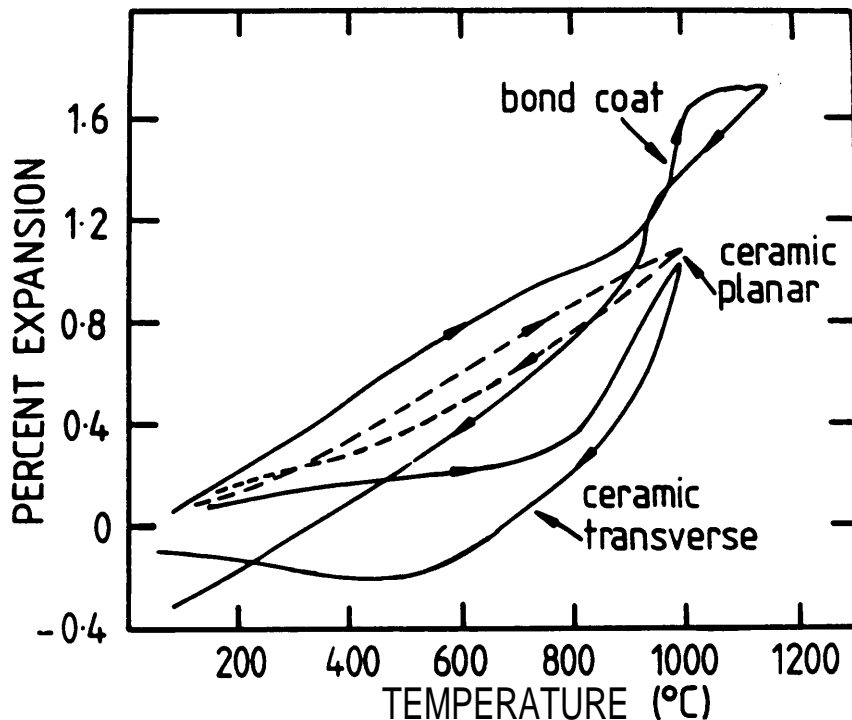


FIGURE 1

Thermal expansion data for **zirconia-8wt%yttria** coatings and a **NiCrAlY** alloy bond coat. Measurements have been performed in the in-plane and through-thickness directions. The data comes from references 8-10

the thickness direction of coatings and values of from about 50% to 200% of the in-plane measurement have been determined. The variance of these measurements is significantly greater, from 8 to 20%, since the dilatometric sample size is much smaller than the in-plane geometry. The major error arises from the absolute detection limit of the dilatometer. The value changes with respect to the nature of the heat treatment on the coating; although there is no clear correlation between the thermal expansion of the coating and the heat treatment parameters.

3.2. Thermal flux testing

The thermal expansion data presented above may be of limited value since the thermal flux environment causes large temperature gradients across the coating. These gradients have been estimated to be of the order of 200°C^{11} . Therefore tests have been developed whereby the response of coated materials under non-equilibrium conditions can be examined. These tests are not standard throughout the industry.

Several experimental techniques are available. The most simple method is to place the coated material within a rapid heating furnace which is thermally cycled between approximately 50 and 1150°C and note is made of the cycle at which failure is initially observed. These experiments have been performed for a number of zirconia-yttria alloys and some results are shown in Figure 2.

The results from high heat flux sources, such as natural gas - oxygen torches and burner rig tests are superimposed on the high temperature (1100°C) and low temperature (990°C) cyclic furnace data¹². This data from many investigators corroborates that the optimum zirconia alloy composition for thermal barrier applications is of about 6 to 8wt% yttria¹³.

3.3. Discussion of thermal property data

A major aim of this paper is to present an argument for the rationalisation of thermal property measurements on coatings. An additional facet of the work is to present the requirements for the thermal specifications of coatings. However it is crucial to first examine whether such standards are necessary.

The thermal property data finds application in the two areas that are described below. The thermal expansion data is necessary in order for the modelling studies^{14,15} that are currently in progress. The aim of such work is to predict the stress distribution across the coating profile and thus, if the failure criteria of the coating is known, then the fatigue life of the coating system can be predicted.

The workers in the above area are currently restricted to using data which is either derived from that of the bulk material; or from experiments that are founded on the assumption that the material behaves in an isotropic fashion. Neither approach is truly satisfactory; although there is general consensus that the current measurements on coatings enable comparative measurements. The future challenge will be to consider the case of absolute measurements so that corresponding absolute lifetimes of practical

systems can be ascertained. This development will proceed with adoption of the principle testing features which are detailed in Table 2.

The adoption of a standard specification, especially with regard to specimen dimensions and operating parameters of the dilatometer, has the additional benefit of comparisons and data sharing between workers. A system of specifications could also be drawn up for the thermal flux testing of **substrate-coating** systems and this list would closely parallel the requirements that are indicated in Table 2.

4. THE ADHESION AND BOND STRENGTH OF COATINGS.

4.1. The limitations of strength measurements

The adhesion standards are quite rigorous in the specification of the method whereby the bond strength of a coating is determined, Table 3. A major difference between the standards which influences the bond strength reproducibility is the specimen

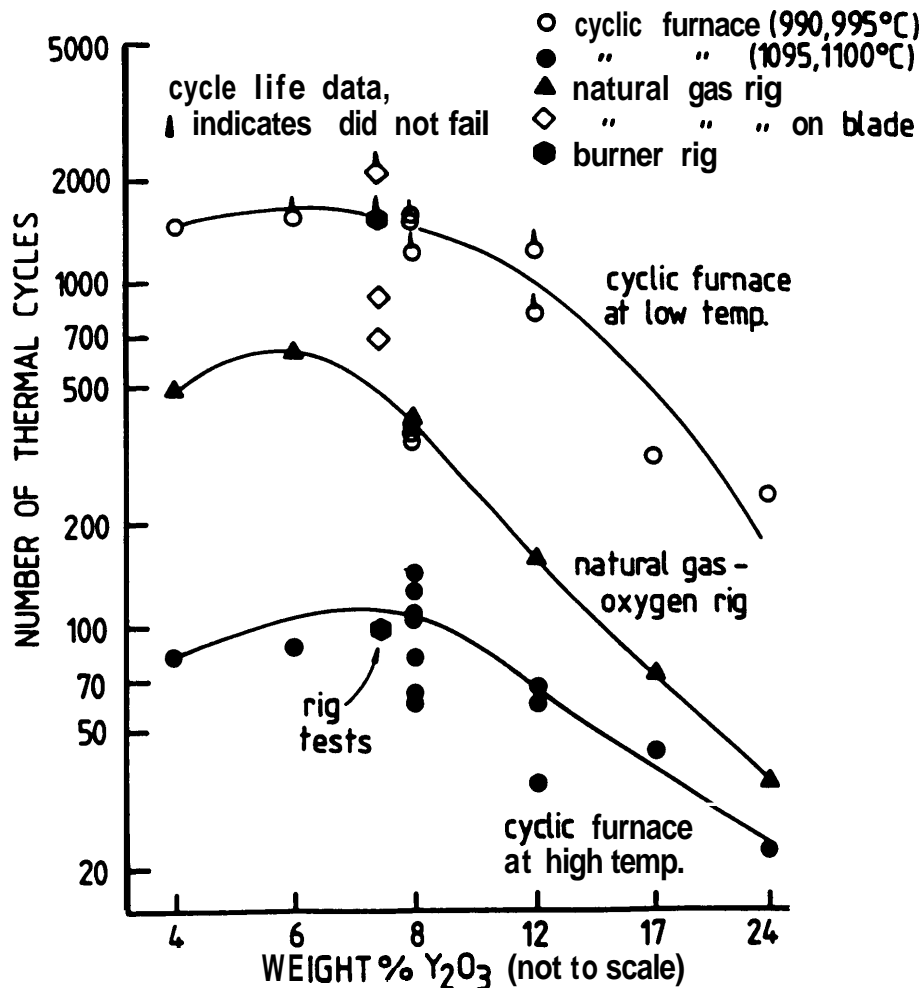


FIGURE 2
Thermal life-cycling data for zirconia-yttria alloys

TABLE 2. REQUIREMENTS FOR THERMAL EXPANSION MEASUREMENTS ON THERMALLY SPRAYED COATINGS

1. Specify whether the coating is free standing or attached to the substrate. All specimens should be planar to the dilatometer measuring rod(s) axis.
2. Specification of the specimen dimensions (length, width and thickness). **Include** the substrate dimensions for systems which incorporate a substrate.
3. Specification **of the** linear heating and cooling rates for the dilatometer.
4. Specification of the maximum temperature and the minimum temperature for any cycling studies.
5. Detail any calibration procedures which account for residual off-sets in strain and specimen alignment.
6. Analysis and reporting of data to include comments on the linear nature, or otherwise, of the thermal strain versus temperature data. Detail the number of replicate tests.

cross sectional area. The DIN and AFNOR standards are much more reliable from a fundamental viewpoint because any stress concentrations are much less. Several features of these standard tests limit their utility and these are listed **below**²⁰.

1. The test is destructive and is therefore performed on coupons of planar material which are not necessarily typical of the coated engineering component which will be used in service.
2. The tensile mode which is measured during the test may not reproduce the appropriate deformation mechanisms and failure mode of the coated component.
3. The standards do not specify the reproducibility of the test and therefore it is difficult to assess the reliability of a test. The reporting of such data should be formalized.
4. The criteria for failure is often linked to the failure morphology and this specification is ambiguous between the standards. The failure mode and its distribution, if of a mixed character, should be clearly defined.
5. The material property of tensile strength may not be the mechanical property that is required for design considerations. For example the elastic modulus or strain to failure may be a more desirable measurement.

The tensile adhesion methodology has much application despite the above shortcomings. The results that are presented below indicate the methods whereby these testing methods can be used to advantage. For example if approximately 30 to 35 tensile adhesion tests are performed then a distribution such as that represented in Figure 3²¹ is obtained.

TABLE 3. COMPARISON OF BOND STRENGTH STANDARDS' 6-19

Standard Specification	Test Specimen		Coating Thickness (mm)	Testing Conditions		Minimum Number of Tests	Failure Description
	Diameter (mm)	Length (mm)		Load Rate (N min ⁻¹)	Crosshead Displacement (mm s ⁻¹)		
ASTM C633-69	25.15 to 25.4	25.4	>0.38	-	0.013 to 0.021	5	● adhesive ● cohesive ● epoxy ● see note 1
DIN 50 160-A	40 ± 0.1	50	>0.15 and <0.4 (note 2) or <0.8(note 3)	1000 ± 100	-	5	● adhesive ● cohesive
AFNOR NF A91-202-79	39.9	45	>0.5	1000 ± 100	-	6	● adhesive ● cohesive ● anything special
JIS H8666-90	25 to 40	40	>0.3 and <0.5	9806.6	0.017	-	● adhesive ● cohesive ● coating/ epoxy failure

Notes 1. Mixed mode failures are of no practical use.
 2. For oxides and carbides
 3. For metals

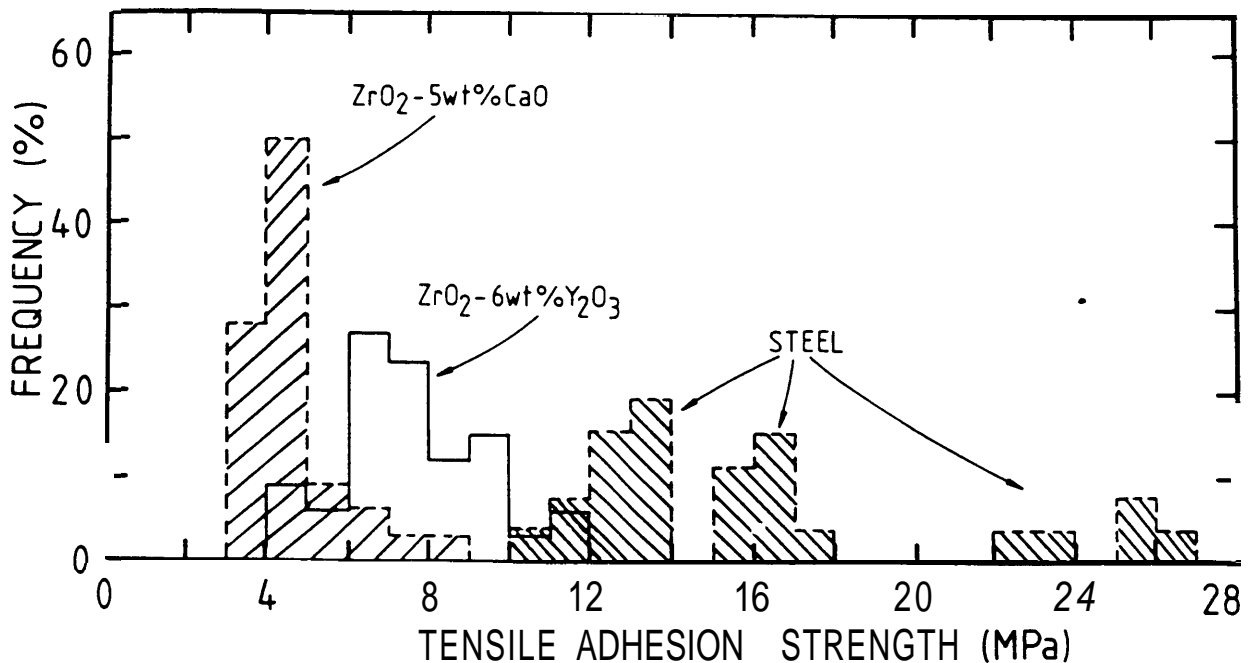


FIGURE 3
 Tensile adhesion test values for thermally sprayed coatings

It can be noted that the distribution in strength values is quite large and this would be expected for any material property which relies on control of any edge defects which are present in the specimen. An **alternative** analysis incorporates a measure for the nature of the statistical distribution of any defects and the Weibull analysis indicates that 'm' for thermally sprayed coatings is about **4²¹**.

4.2. Discussion on strength measurements

There is only a little chance that the tensile adhesion test will be superseded by more advanced techniques. The methods which employ fracture mechanics methodology^{20,22-24} rely on quite sophisticated experiments which are probably beyond the expertise of an industrial facility. However any methods which allow tensile adhesion tests to be interpreted in terms of a more appropriate design philosophy would be welcomed.

Thus it is possible to estimate the mode 1 fracture toughness (ie, under tensile conditions) of coatings from tensile adhesion test data. The estimation of **K_{IC}** values for alumina coatings range from 0.2 to 2.7 x 10⁶ Nm^{-3/2} when calculated by using this **approximation²⁵**. A comparison with other experimental work gives values of 1 to 1.35 x 10⁶ Nm^{-3/2} for **yttria** stabilized zirconia **coatings²⁶**; and 2.2 to 3.2 x 10⁶ Nm^{-3/2} for magnesia stabilized **coatings²⁷**.

Major features of the various tensile adhesion tests that need standardization are the description of the fracture morphology and a formal reporting procedure that accounts for the mixed mode type of failure that often occurs. It is also necessary to document the number of tests that were performed in order to determine the average. For example a statistical method that relies on the attainment of a specific variance (ie, the ratio of the standard deviation to the mean) of, say, 20% may be **appropriate²⁸**. In this fashion it is expected that comparison of the results between **laboratories** or industrial facilities will be possible.

There will, however, always be ambiguity in comparing results that have been obtained with the large (40 mm diameter) or the small (25.4 mm) test coupons that may be preferred in Europe or North America; since the stress concentration over the specimen surfaces are quite **different^{29,30}**. This question can be resolved by performing a controlled series of tests on the same coatings and then ascertaining whether the fracture stress or fracture morphology for this material differ significantly. Such an empirical correlation would be invaluable for the industry.

A final point concerns the limited use of the present strength standards. All of the present standards have been designed for simple implementation; but this criteria has also restricted their functionality so that they are not related to any real service conditions. This shortcoming is perceived as the greatest drawback of these tests since no fundamental knowledge of the failure mechanism is obtained. Thus the present standards are limiting any future development of thermal spray coatings.

5. CONCLUDING REMARKS

This paper has focussed on only two of the material properties that were presented in Table 1. It is proposed that one of these, the thermal testing of thermally sprayed coatings, should be formalized into a standard so that inter-laboratory comparisons are facilitated. The other standard, which is used to **determine** the tensile strength of coatings, is already well-established; but it is suggested that discrepancies between the various specifications need to be addressed.

The importance of the other material properties which are indicated in Table 1 is also regarded as an area where future activity is required. In particular much effort on the safety aspects of coating preparation and coating **processing**^{31,32} and microstructural preparation and **evaluation**³³⁻³⁶ needs to be performed. The question of safety is quite serious since there is the requirement to thermal process fine particles and **fibres**^{37,38}; as well as the necessity to prepare coatings of toxic materials such as those of **barium**³⁹ compounds which are used in superconducting materials.

The necessity of performing quantitative microstructural analysis is also important since these features distinguish coatings that have been prepared from identical starting materials. Thus an alternative view of the coating structure is an interpretation in terms of a material modification brought about by changing the processing (or thermal spraying) parameters. It can be pointed out that the preparation of the microstructure cross-sections is still an art and experience has demonstrated that different laboratories may measure quite different porosities on materials that have been prepared from identical coatings. It would be useful for the thermal spray industry to establish a metallographic standard which incorporates coatings of known porosity so that the above errors can be disregarded.

The last point to be covered concerns the relationship of the material standard to the application. Any of the existing standards or potential standards which are listed in Table 1 should be performed in the context of the intended use of the **coating/substrate** system. Thus it is important that any coating not only be subject to some specific standard but that the standard be relevant to the particular material. It can be foreseen that some ceramic coatings will be subject to either shear or tensile loading and the appropriate measurements must be **carried** out. If the tensile strength can be empirically correlated to the shear or any mixed mode loading then the standard tensile adhesion test may be quite justified as the sole test. There is also the potential of applying other methods such as the scratch **test**^{40,41} or an acoustic emission method^{42,43} which could be related to an intrinsic material property.

Finally; the thermal spraying practitioner should be cautioned that these coatings are not homogeneous. Future applications may require the degree of anisotropy to be ascertained and this will necessitate the formulation of new appropriate standards. The

purpose of the present paper has been to lay the foundations for any testing philosophy and practical methods which may be performed in the future.

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