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Date 5 April 2004

Dear Bill

Re: Milestone Completion – LPM 2.2/T6/M14 – d1 – Steve Osgerby

Please find attached the deliverable necessary for the completion of this milestone, namely:

<table>
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<th>Identifier</th>
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<tbody>
<tr>
<td>LPM 2.2-T6-M14 d1</td>
<td>Measurement Note on new test procedure, distributed to DTI and IAG, promoted to coatings manufacturers, gas turbine operators and COST522 group via LPM 0.5</td>
<td>Measurement Note: MATC(MN)54 Young’s Modulus Measurement for Coatings used in Gas Turbines by S Osgerby, J W Nunn, J P Banks, A S Maxwell and G Aldrich-Smith December 2003</td>
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I have reviewed the attached deliverable and compared it against the contractual requirement. In my view this milestone is now complete and in accordance with our agreement of 16 May 2001 shall be invoiced at 100%.

I trust this is acceptable, please contact me if you have any further queries.

Yours sincerely

Clive Scoggins  
MATC Proposals Manager
Measurement Note

Young's Modulus Measurement for Coatings used in Gas Turbines

Young's Modulus is a fundamental property of metallic and ceramic materials. As such it is an important input parameter to many predictive models of material behaviour. However, despite a widespread appreciation of its importance, measurement of Young's Modulus still presents significant challenges in some areas. In particular methods are not optimised for measuring modulus of coatings. This Measurement Note compares four possible methods for the measurement of the Young's modulus of coatings, viz impact excitation, nanoindentation, laser surface acoustic wave and uniaxial straining of coated testpieces and thick free-standing coatings.

S Osgerby, J W Nunn, J P Banks, A S Maxwell and G Aldrich-Smith

December 2003
INTRODUCTION

Many components in gas turbines are coated, either to protect them from degradation due to the atmosphere or as a thermal barrier system. Young's modulus is an important input parameter to models of lifetime prediction, but methods to measure this property in coatings have not been thoroughly validated. This Measurement Note compares several existing methods for modulus measurements and highlights the main features and limitations of each method.

IMPACT EXCITATION

This method is well established [1] and utilises the natural resonant frequency of a material, which is a function of its Young’s Modulus. A beam specimen is supported on two stands, which are spaced approximately 25 and 75% along its length, and vibrations are introduced by dropping a small sapphire ball onto the centre of the beam. The resultant acoustic vibrations are directed to a microphone through a hollow ceramic tube and analysed to identify the fundamental frequency of the beam. The experimental setup is shown schematically in Figure 1.

The Young’s modulus for a simple material can be calculated from the fundamental frequency using the expression:

\[ E = \frac{0.9465l^3mf^2}{wh^3} \]

where
E is the Young’s modulus
f is the fundamental frequency of vibration
m is the mass of the beam
l, w and h are the beam length, width and thickness respectively

For beams coated on both sides, the modulus of the coating can be calculated from the frequency of vibration of the composite beam and the known Young’s modulus of the substrate using the expression:

\[ E_c = E_s \frac{h_i^3}{h_i^3 - h_s^3} \left( \frac{0.9465mf_i^2l_i}{E_swh_i^3} \right) - 1 \]

where the subscripts c, s and t refer to the coating, substrate and total specimen respectively.

One major advantage of this technique is that it can be used at elevated temperatures. An example of data obtained on a corrosion resistant coating using this technique is shown in Figure 2.

Figure 2 Modulus as a function of temperature for CMSX-4 and Amdry 995 coating on a CMSX-4 substrate

LASER SURFACE ACOUSTIC WAVE (LSAW)

During this test [2] the specimen surface is energised using a short impulse from a laser. This results in a localised thermo-elastic expansion that generates high bandwidth surface acoustic waves (SAWs). The SAWs propagate along the sample and are detected by a piezo-foil transducer (Figure 3) at a set distance from the incident pulse. The surface waves are restricted to a depth of ~50 μm in metallic specimens, thus for typical coatings used in gas turbines the waves travel exclusively through the coating.

Figure 1 Schematic Representation of Impact Excitation Apparatus
The Young’s modulus is calculated from the simple expression:

\[ E = 2\rho(1 + \nu) \left[ \frac{(1 - \nu)c}{0.87 + 1.12\nu} \right]^2 \]

Where \( \rho \) is the density, \( c \) is the velocity of the acoustic wave in the specimen, \( \nu \) is Poisson’s ratio.

Using this technique the Young’s modulus of Amdry 995 on CMSX-4 substrate was measured as 197 GPa at room temperature.

At present the technique is limited to ambient temperatures and certain materials, however within these limitations it provides a simple and rapid measurement method for modulus.

**INDENTATION**

Analysis of unloading curves during depth-sensing indentation allows calculation of indentation modulus. The technique is well established and characterised [3]. Coatings may be indented either from the surface or in cross section. Indenting the coating surface is the most straightforward method as it does not require any specimen preparation, however surface roughness and/or cracking may cause problems. This is illustrated in Figure 4 where the load/displacement curve for a valid indent (Figure 4a) is shown alongside the curve produced when the loading cycles is disturbed by cracking of the coating (Figure 4b).

Previously published work has used indentation in cross-section to measure coating hardness and modulus [4]. This method requires metallographic preparation of specimens but produces more consistent measurements.

**UNIAXIAL STRAINING**

The method that appeals most strongly to the traditionalist is the simple tensile test. This may be done in two ways: 1) by producing a ‘free-standing’ coating of sufficient thickness that tensile specimens may be produced directly, or 2) by using a substrate that is thin enough that the stress/strain response of the substrate and coating can be de-convoluted from the behaviour of the composite. This latter approach has been used successfully to measure the creep properties of coatings (5) and work is in progress to apply a similar analysis to tensile behaviour.

This technique can be used at elevated temperatures but there are potential problems (eg buckling of the specimens) in producing coatings on thin substrates. There is also the question of whether a thick, free-standing coating is representative of the structure produced on coated components.
CONCLUSIONS

Two techniques, impact excitation and uniaxial straining, have been identified that allow measurement of the Young’s modulus of coatings at elevated temperature. Both of these methods require accurate data for the substrate when composite specimens are used. The alternative of using specimens produced from thick, free-standing coating raises the question of whether the structure of the coating in that form is representative of coated components.

Two other techniques, indentation and LSAW, are currently limited to ambient temperature although high temperature stages for both these techniques are becoming available. These techniques require care in operation but are capable of producing accurate data.

ACKNOWLEDGEMENTS

The work reported in this document was performed as part of the Lifetime Performance of Materials programme, a programme of underpinning research financed by the UK Department of Trade and Industry.

REFERENCES

[1] R Morrell, Traceability calibrating the 'Grindosonic', for impact excitation modulus measurement, NPL (A)36 August 1996

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