

Resonant-Based Identification of the Elastic Properties of Ceramic Coatings

T. Lauwagie*, W. Heylen

K.U.Leuven, Department of Mechanical Engineering,
Celestijnenlaan 300 B, B-3001, Heverlee, Belgium
e-mail: tom.tauwagie@imec.be

Abstract

The elastic properties of the individual layers of layered materials can be derived from the resonant frequencies of a set of test specimens comprising samples with different layer configurations [1]. Based on this, a mixed numerical-experimental framework for the identification of the elastic properties of layered materials has been formulated and a number of different identification routines were developed.

In this work, the elastic properties of an yttria-stabilised zirconia top coat of an air plasma sprayed thermal barrier coating are identified with the different mixed numerical-experimental routines that were developed. The obtained results are critically evaluated, and some recommendations on the use of the considered routines for the identification of coating properties are presented.

1 Introduction

Coated materials are becoming increasingly important for the production of high performance components and constructions. In the aerospace and automotive industries, ceramic coatings are commonly used to shield metallic components from high temperatures and corrosive environments.

The elastic properties of the coating materials are required to model the structural behaviour of the coated component, and are crucial to predict the residual stresses in the coating layer. Predictions of the residual stresses, which are induced by the thermal expansion coefficient mismatch between the substrate and coating, are essential for preventing a delamination of the coating.

There are several techniques to produce ceramic coatings and every technique results in a particular type of microstructure. The microstructure of an air plasma sprayed (APS) coating consists of a complex network of splats, pores and cracks. This microstructure has a severe impact on the mechanical properties of the coating; the elastic modulus of an APS coating can be up to twenty times lower than the elastic modulus of the corresponding bulk material [2]. A characterisation of the coating material can, therefore, only be performed on coated substrates and thus requires a identification routine for layered materials.

In this work, the elastic properties of an yttria-stabilised zirconia top coat (TC) of a air plasma sprayed thermal barrier coating are determined by means of a resonant-based mixed numerical-experimental technique, that was developed for the characterisation of layered materials

*Now at IMEC, Kapeldreef 75, B-3001, Heverlee, Belgium.

2 Resonant-Based Material Identification

2.1 Standardised Techniques

Resonant-based material identification methods are founded on the fundamental relation that exists between the vibratory behaviour of a structure and the elastic material properties. These methods derive the elastic properties of the material from the resonant frequencies of a test sample. The main advantage of the resonant-based approach is that the material can be tested with free boundary conditions. This results in a higher reproducibility of the obtained results [3] and makes it possible to perform the tests in a high temperature furnace to determine the temperature dependence of the material properties [4]. The resonant-based techniques are standardised by an ASTM [5] and a CEN [6] standard, which provide a set of analytical equations relating the elastic properties of the material to the resonant frequencies of the test sample.

Unfortunately, the standardised resonant-based techniques are restricted to homogeneous isotropic materials. The limiting factor in extending the resonant-based techniques to more complex materials is the use of analytical models to describe the vibratory behaviour of the test specimens. The limitations of the standardised methods can be overcome by replacing the analytical vibration models with finite element models. Due to the flexibility of the finite element method, this approach opens a whole new range of possibilities such as the identification of orthotropic [7–9] and/or layered materials [10, 11].

2.2 Mixed Numerical-Experimental Techniques

Replacing the analytical formulas by finite element models complicates the implementation of the resonant-based identification procedures. Finite element models allow the computation of the resonant frequencies of a test specimen made out of a particular material. Unfortunately, the finite element formulation cannot be reversed into a formulation that provides the material properties from the resonant frequencies of the test samples. The material identification problem has to be solved in an inverse way: starting from a set of trial values, the unknown material parameters are fine tuned in such a way that the finite element model reproduces the measured resonant frequencies. Techniques based on this approach are commonly referred to as Mixed Numerical-Experimental Techniques or MNETs [12]. Figure 1 provides the general flowchart of the resonant-based MNET for the identification of the elastic properties of materials.

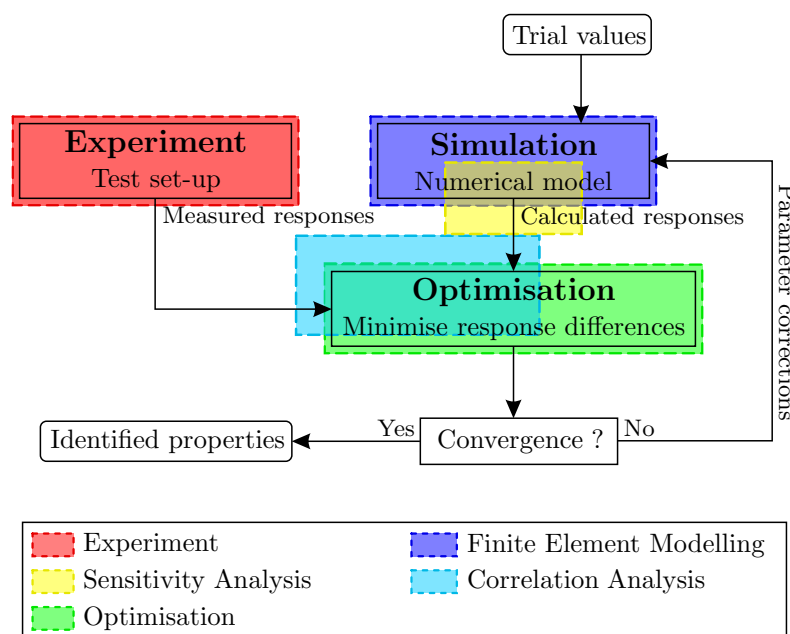


Figure 1: The general flowchart of the resonant-based MNETs.

The identification of the elastic properties starts with the computation of the numerical resonant frequencies using a set of trial values for the unknown elastic parameters. The numerical and experimental frequencies are compared, and the values of the unknown model parameters are corrected in order to minimise the differences between the two frequency sets. The improved material properties are inserted into the FE-model(s) and a new iteration cycle is started. Once the numerical and experimental frequencies match, the procedure is aborted, and the desired material properties can be found in the database of the FE-model. The MNET procedure consists of five main components: a set of experiments, a set of numerical models, a correlation analysis, a sensitivity analysis, and an optimisation problem.

2.2.1 Experimental Set-Up

The experimental part consists of measuring the length, width, thickness, mass and resonant frequencies of the freely suspended test samples. The resonant frequencies of the test specimens are preferably measured in a contactless way. This can be achieved by exciting the test specimens with a computer-controlled loudspeaker or a miniaturised hammer and by capturing the vibration response with a laser vibrometer or a microphone. The measured vibration signal is digitised with a data acquisition card and stored by a computer. Finally, the resonant frequencies are extracted from the time signals with a model parameter estimation routine. Figure 2 shows the test set-up using a loudspeaker and laser vibrometer.

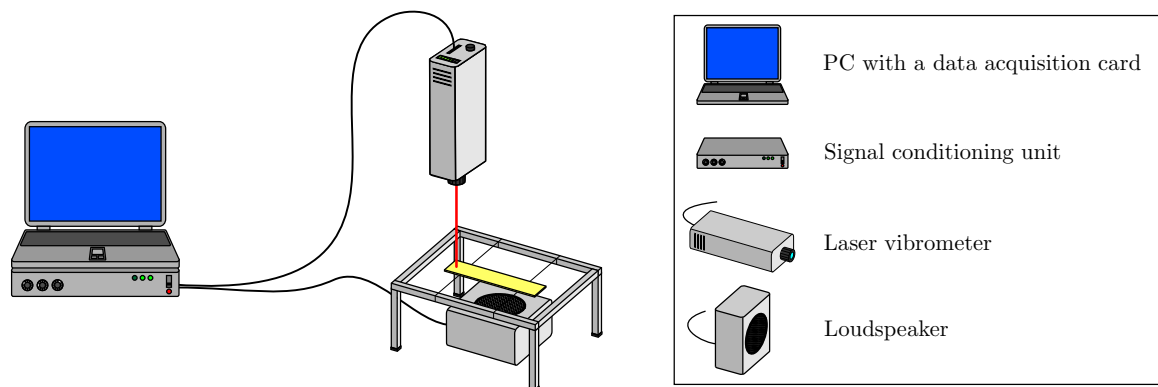


Figure 2: The experimental set-up of the resonant-based MNET procedure.

2.2.2 Numerical Modelling

The numerical part of the MNET computes the resonant frequencies of the test samples with a number of finite element models (FE-models). These FE-models have to be as accurate as possible. With MNETs, the elastic parameters are determined by updating the material parameters of the FE-models. Any inaccuracy of the FE-models will have a negative influence on the values of the identified properties since the MNET routine will compensate this inaccuracy by changing the values of the material parameters. Accurate FE-models can only be obtained if the geometry of the samples is relatively simple and if the mesh of the models is fine enough for the considered vibration modes. To limit the computation time, it is advisable to determine the optimal mesh density with a convergence study.

2.2.3 Correlation Analysis

To identify the material properties, every numerical frequency has to be compared with the experimental resonant frequency of the same vibration mode. For example, the frequency of the numerical torsional mode has to be compared with the experimental torsional frequency. Unfortunately, the order of the numerical modes depends on the elastic properties of the numerical model, e.g. an over- or underestimation of the shear

modulus of a beam-shaped sample can change the position of the fundamental torsional mode with respect to the flexural modes. The order of the modes of the finite element model is thus not necessarily the same as the order of the experimental modes, therefore the frequencies cannot be compared sequentially. The numerical and experimental modes can be correctly paired by means of a correlation analysis based on the computation of the MAC matrix between the numerical modes and a set of reference mode shapes describing the considered experimental modes [10].

2.2.4 Sensitivity Analysis

The aim of sensitivity analysis is to quantify the influence of the elastic material parameters on the resonant frequencies of the specimens. Local sensitivity analysis techniques result in a number of sensitivity coefficients (s_{ij}). Each sensitivity coefficient represents the rate of change of a particular resonant frequency (f_i) caused by a change of a specific elastic parameter (p_j), and can thus be used to estimate the parameter changes that are required to match the numerical resonant frequencies with the experimental frequencies. In the case of finite element models, the sensitivity coefficients can easily be computed with the semi-analytical approach [13, 14] which provides the following expression for the sensitivity coefficients.

$$\frac{\partial f_i}{\partial p_j} \frac{p_j}{f_i} = \frac{p_j}{8\pi^2 f_i^2} \left(\{\Psi\}_i^T \frac{\Delta[K]}{\Delta p} \{\Psi\}_i \right) \quad (1)$$

in which $\{\Psi\}_i$ is the mode shape vector of the considered mode, $[K]$ is the stiffness matrix of the finite element model and $\Delta \diamond$ represents a finite difference of the considered quantity. All the sensitivity coefficients of the resonant frequencies of one particular sample can be grouped into a sensitivity matrix $[S]$ as

$$[S] = \begin{bmatrix} \overbrace{\frac{\partial f_1}{\partial E_{1,1}} \frac{E_{1,1}}{f_1} \dots \frac{\partial f_1}{\partial \nu_{12,1}} \frac{\nu_{12,1}}{f_1}}^{\text{Material 1}} & \overbrace{\frac{\partial f_1}{\partial E_{1,n_m}} \frac{E_{1,n_m}}{f_1} \dots \frac{\partial f_1}{\partial \nu_{12,n_m}} \frac{\nu_{12,n_m}}{f_1}}^{\text{Material } n_m} \\ \vdots & \vdots \\ \frac{\partial f_{n_f}}{\partial E_{1,1}} \frac{E_{1,1}}{f_{n_f}} \dots \frac{\partial f_{n_f}}{\partial \nu_{12,1}} \frac{\nu_{12,1}}{f_{n_f}} & \frac{\partial f_{n_f}}{\partial E_{1,n_m}} \frac{E_{1,n_m}}{f_{n_f}} \dots \frac{\partial f_{n_f}}{\partial \nu_{12,n_m}} \frac{\nu_{12,n_m}}{f_{n_f}} \end{bmatrix} \quad (2)$$

where n_f is the number of resonant frequencies that were measured on the considered sample, and n_m is the number of materials that have to be identified.

2.2.5 Optimisation

Using the sensitivity matrix, the effect of a change of the elastic parameters on the resonant frequencies of the k^{th} sample can be expressed as

$$\{\Delta f\}_k = [S]_k \{\Delta p\}, \quad \forall k = 1, \dots, n_s \quad (3)$$

where n_s represents the number of tested samples. These n_s sets of equations can be combined into one global set of equations as

$$\underbrace{\begin{Bmatrix} \{\Delta f\}_1 \\ \{\Delta f\}_2 \\ \vdots \\ \{\Delta f\}_{n_s} \end{Bmatrix}}_{\{\Delta f\}} = \underbrace{\begin{bmatrix} [S]_1 \\ [S]_2 \\ \vdots \\ [S]_{n_s} \end{bmatrix}}_{[S]} \{\Delta p\} \quad (4)$$

where $\{\Delta f\}$ is the global frequency difference vector and $[S]$ is the global sensitivity matrix. The parameter corrections $\{\Delta p\}$ are found by solving the system of equations (4) in a least-squares sense. To ensure a stable convergence of the material parameters, it is advisable to limit the relative changes of the parameters during each iteration. The optimal parameter corrections are thus obtained by solving the following optimisation problem

$$\begin{aligned} & \underset{\Delta p}{\text{minimise}} \{ \Delta p \}^T [S]^T [W] [S] \{ \Delta p \} - 2 \{ \Delta f \}^T [W] [S] \{ \Delta p \} \\ & \text{subject to } \Delta p_i \leq \overline{\Delta p} \quad \forall i = 1, \dots, 4 \times n_m \\ & \quad \quad \Delta p_i \geq \underline{\Delta p} \quad \forall i = 1, \dots, 4 \times n_m \end{aligned} \tag{5}$$

where $[W]$ is a diagonal weighting matrix for the responses, $\overline{\Delta p}$ and $\underline{\Delta p}$ are the upper and lower bounds of the parameter changes, respectively. Experience has shown that limiting the parameter corrections during every iteration step to 25% is a good compromise between convergence speed and stability [10].

2.3 Layered Material Identification

2.3.1 Identifiability of the Layer Properties

Using the classical lamination theory [15], it is possible to prove that the out-of-plane vibration modes of a sample with orthotropic layers are predominantly controlled by four integrated stiffness coefficients, i.e. the four independent bending stiffness coefficients D_{11} , D_{22} , D_{12} , and D_{66} , where

$$D_{ij} = \frac{1}{3} \sum_{k=1}^{n_l} (\overline{Q}_{ij})_k (z_k^3 - z_{k-1}^3) \tag{6}$$

in which \overline{Q}_{ij} represents the reduced stiffness coefficients which are a function of E_1 , E_2 , G_{12} , ν_{12} , and the material orientation, k indicates the index of the considered layer, n_l represents the number of layers, and the z values represent the positions of the layer interfaces in the thickness direction. From these four integrated stiffness coefficients, only four independent material parameters can be derived. The resonant frequencies of a single layered sample thus allows the identification of the elastic properties of one of the layers of the test sample.

The identification of the n_m materials that are used in the various layers of the laminate requires n_m sets of four integrated stiffness coefficients. In order to provide a unique solution, the relation between the elastic layer properties and the integrated stiffnesses must vary from set to set. Equation (6) shows that this can only be achieved by changing the thickness or the stacking of the layers, changing the size or shape of the samples will not have any effect on the integrated stiffnesses. Since every layer configuration provides four additional stiffness coefficients, the identification of the n_m layer materials requires the resonant frequencies of at least n_m layer configurations.

The vibratory behaviour of isotropic materials is controlled by only two independent stiffness coefficients. These two coefficients can provide the values of two independent material parameters, i.e. the elastic parameters of one material. This means that for isotropic materials the number of required layer configurations also equals the number of materials that have to be identified. A detailed study of the identifiability of the elastic properties of the materials of the individual layers of laminated materials can be found in [10].

2.3.2 The Identification Routines

Using the MNET approach in combination with the concept of layer configurations, two identification routines to determine the elastic properties of layered materials were developed: the single- and multi-orientation routine.

The single-orientation (SO) routine derives the elastic properties of the layers from the fundamental flexural and torsional frequencies from a set of beam-shaped samples that all represent the same material orientation. The routine is capable of identifying the apparent elastic (E_x) and shear (G_{xy}) modulus in the direction parallel to the long-axis of the samples, and this for all the layer materials.

The multi-orientation (MO) routine derives the elastic properties of the layers from the fundamental flexural and torsional frequencies of a set of beam-shaped samples that all represent a different material orientation. The routine is capable of identifying the four principal elastic parameters (E_1 , E_2 , G_{12} and ν_{12}) of all the layer materials. Note that the four principal elastic parameters allow the computation of the apparent elastic properties in an arbitrary orientation.

3 Identification of the Coating Properties

3.1 The Test Samples

To identify the coating properties, three plates with nominal dimensions of 150 mm × 145 mm were machined out of a 2 mm thick stainless steel sheet. The first plate was left uncoated, the other two plates were coated with a 250 μm and 500 μm thick layer of top coat. These three test plates provided the following four test configurations. The first test configuration gave a non-layered identification problem that provided a set of reference values of the substrate properties. The last three test configurations gave layered identification problems that provided the properties of both the substrate and the coating.

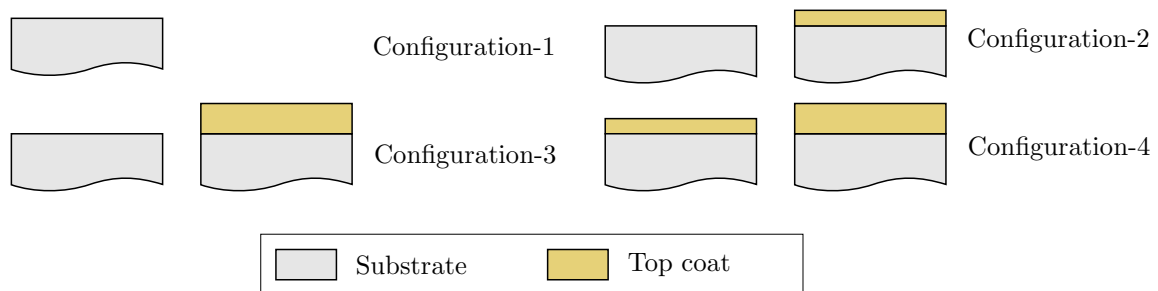


Figure 3: The four considered test configurations.

Out of every plate, nine test beams were cut using a computer controlled laser cutting machine. The beams had a nominal dimension of 40 mm × 12 mm and were cut in three different directions: three beams parallel to the rolling direction, three beams with an orientation of 45°, and three beams perpendicular to the rolling direction. Figure 4 gives an overview of the nine test beams that were cut out of every plate.

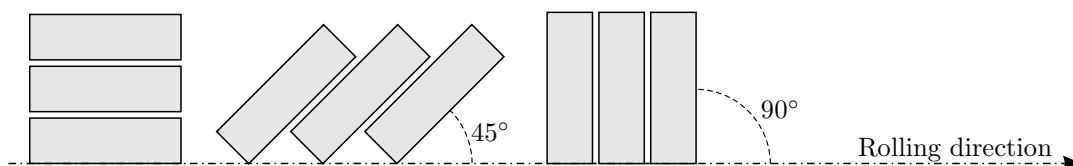


Figure 4: The orientation of the test specimens.

3.2 The Substrate

The reference values of the substrate properties were identified from the fundamental flexural and torsional frequencies of the uncoated beams (configuration-1) with both the single- and multi-orientation routine. The substrate material exhibited an elastically anisotropic behaviour. The elastic modulus varied about 4.5 % in

function of the material orientation, and was maximal for the direction perpendicular to the rolling direction. The difference between the minimal and maximal shear modulus was about 5 %, and Poisson's ratio varied about 12 % in function of the material orientation. The plots of figure 5 present the identified elastic parameters in function of the material orientation.

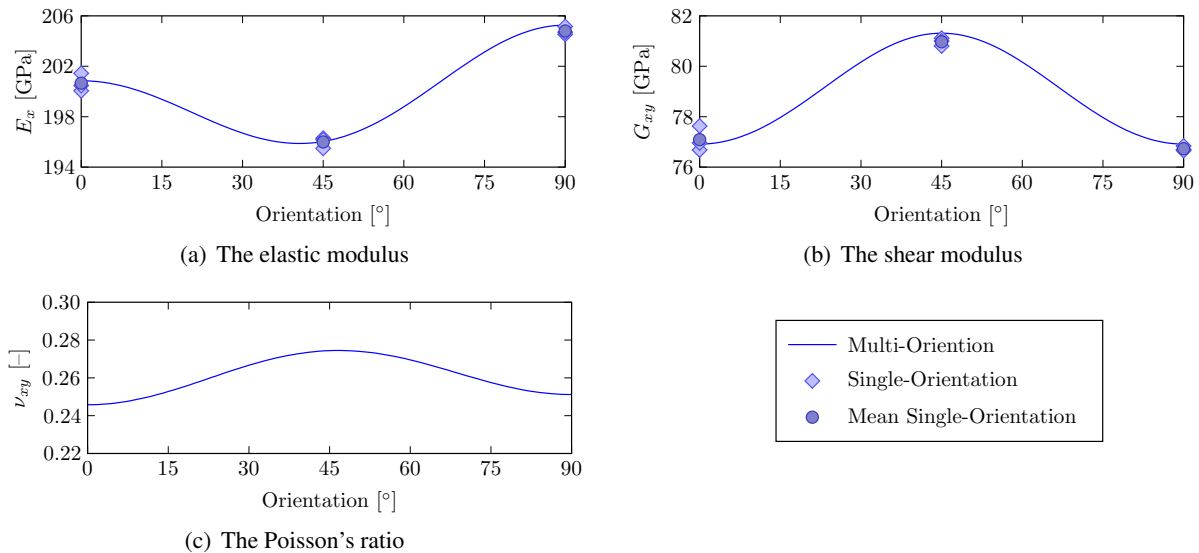


Figure 5: The elastic properties of the steel substrate.

3.3 The Coating

The elastic properties of the top coat were identified using two different approaches.

With the first approach, the coating properties were identified while the properties of the substrate were kept fixed at the values identified on the uncoated specimens. This approach represents the case where a coating with unknown properties has been applied to a substrate of which the elastic properties are known. The main advantage of this approach is the stability of the identification procedure.

With the second approach, the coating properties were identified by simultaneously updating the properties of both the substrate and coating material. This approach represents the situation where both the coating and substrate properties are unknown. The main advantage of this approach is obviously the fact that it does not require any a priori knowledge on the substrate properties.

3.3.1 The Test Specimens

The technician who cut the test beams from the initial plates made a mistake while cutting the specimens with the 250 μm thick top coat layer. Instead of cutting three samples in every direction, he cut four samples in the 0° direction and only two specimens in the 90° direction. The initial test plates also had a number of small holes along the edges. Unfortunately, the technician did not notice these holes and cut a sample (0° with 500 μm TC) from an area that comprised one of those suspension holes. Furthermore, during the cutting process the coating of one of the samples (90° with 500 μm TC) spalled off.

3.3.2 The First Identification Approach

Using the first identification approach, the properties of the top coat were identified from the layered samples by only updating the coating properties. With the single-orientation routine, the coating properties were

identified from the fundamental flexural and torsional frequencies of a single beam-shaped specimen. The substrate properties were fixed to the values that were identified on the uncoated specimens in the considered material orientation. With the multi-orientation routine, the coating properties were identified using all the beam-shaped samples with the same nominal coating thickness, i.e. 250 μm or 500 μm . During the identification, the substrate properties were fixed to the values that were obtained with the multi-orientation routine on the uncoated specimens. Note that the MNET identification routine uses a weighted least-squares cost-function (5). Since the number of samples (n_s) varied from one direction to another, the least-squares weighting coefficients were chosen in such a way that every material direction had the same importance. Table 1 gives an overview of the weighting coefficients (w_i) that were used in the multi-orientation routines.

Table 1: The weighting coefficients of the multi-orientation routines.

Dir. [$^\circ$]	250 μm coating			500 μm coating		
	n_s	w_i	$\sum n_s w_i$	n_s	w_i	$\sum n_s w_i$
0	4	0.75	3.00	2	1.50	3.00
45	3	1.00	3.00	3	1.00	3.00
90	2	1.50	3.00	2	1.50	3.00

Figure 6 presents the material parameters that were obtained with the first identification approach (test configurations 2 and 3). The plots indicate that there was no significant variation of the properties as a function of the material orientation, which indicates that the top coat was isotropic. The average difference between the properties obtained with single- and multi-orientation routine is 6.2% for the elastic modulus, and 1.5% for the shear modulus.

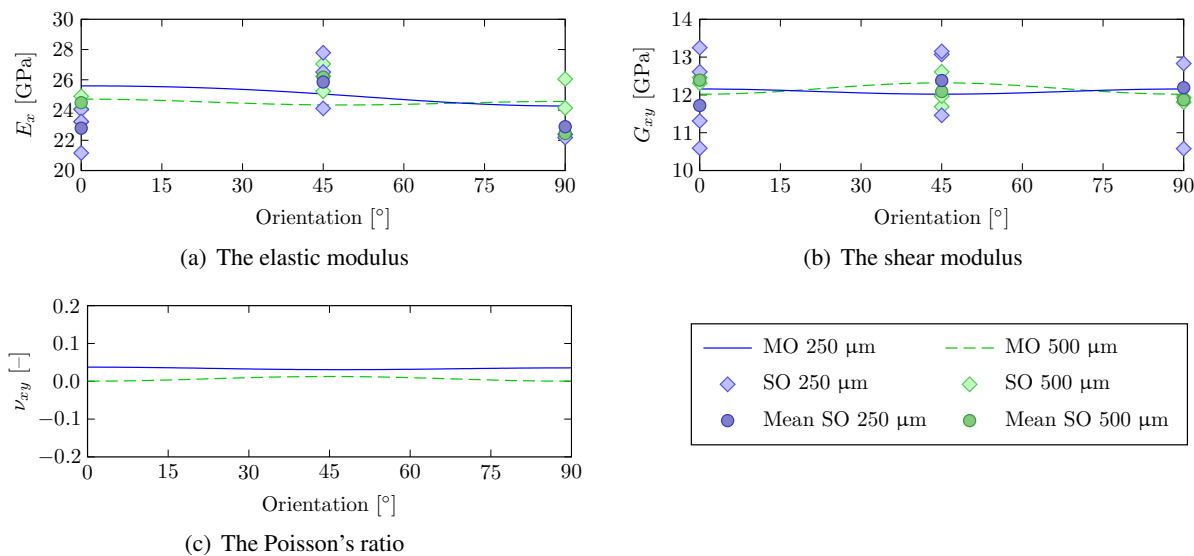


Figure 6: The elastic properties of the YSZ8 top coat.

3.3.3 The Second Identification Approach

With the second identification approach, the properties of the top coat were identified by simultaneously updating the substrate and the coating properties. This identification approach was applied on the samples of the fourth test configuration of figure 3. With the single-orientation routine, the substrate and coating properties were identified from the fundamental flexural and torsional frequencies of a sample set that comprised all the test beams with the same material orientation. With the multi-orientation routine, the substrate

and coating properties were identified using all the coated beam-shaped samples in a single identification routine. To ensure that every orientation of every layer configuration, i.e. 250 μm or 500 μm , had the same importance, the weighting coefficients of table 1 were used to define the least-squares cost-function, and this for both the single and multi-orientation routines.

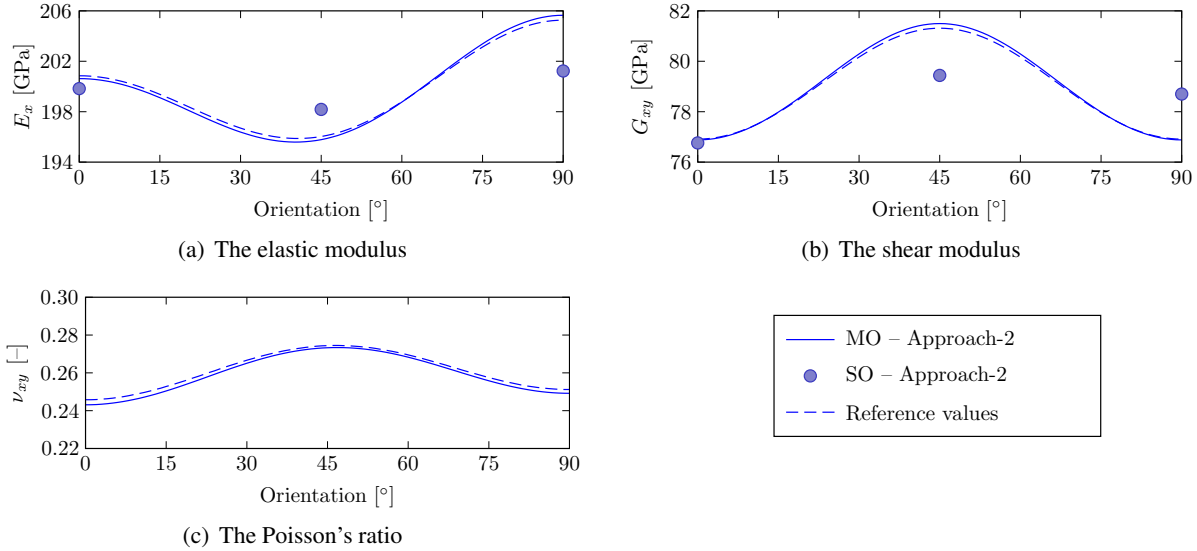


Figure 7: The elastic properties of the substrate.

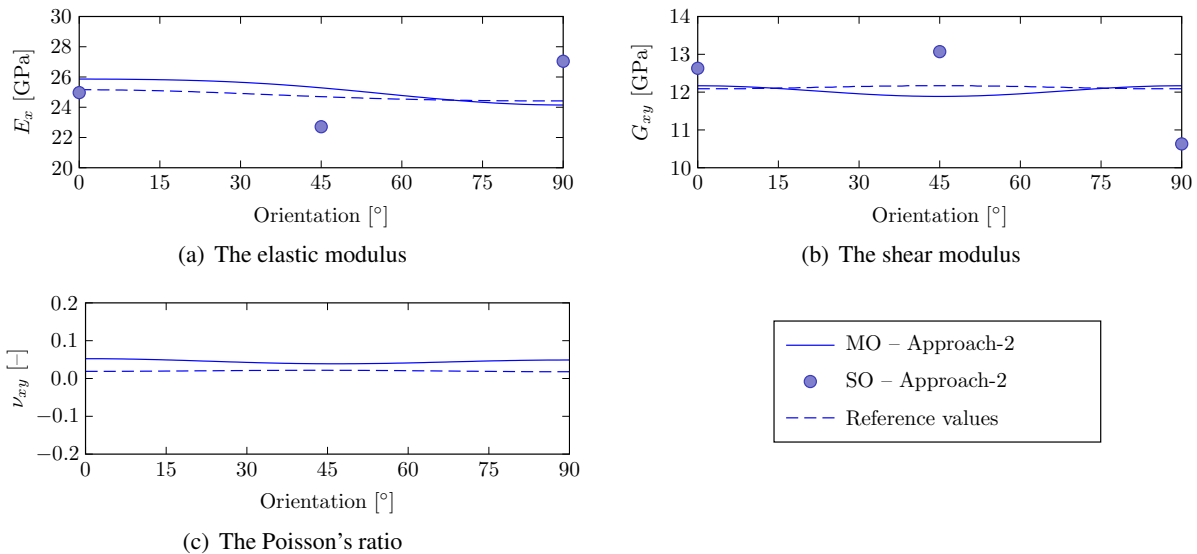


Figure 8: The elastic properties of the top coat.

Figures 7 and 8 compare the material properties of the second identification approach with the reference properties. As shown by the plots, the single-orientation routine provided acceptable results. The average difference between the identified and reference¹ substrate properties was about 1.4 %, while the average difference between the identified and reference² coating properties was estimated at about 10 %.

For the multi-orientation routine there was a better correlation with the reference properties. For the substrate

¹The properties obtained with the multi-orientation routine on the uncoated substrate samples are used as reference data for the substrate material.

²The average of the properties obtained with the first identification approach using the multi-orientation routine are used as reference data for the coating material.

properties, there was an average difference of only 0.4 % between the identified and reference properties. For the coating properties, there was an average difference of 1.5 % with the reference properties³.

3.4 Discussion

The material properties of the top coat were identified with both the single- and multi-orientation routine. Two different situations were considered, i.e. a coating on a substrate with known properties and a coating on a substrate with unknown properties.

Both routines are able to identify the elastic properties of the top coat. In the case where the substrate properties are known, the two routines are comparable, although the results of the multi-orientation routine appear to have a lower spread than the results of the single-orientation routine. In the case where the substrate properties are unknown, the multi-orientation routine performs clearly better than the single orientation routine.

The considered top coat is isotropic, this implies that samples with a different orientations provide the same information. So, in theory the single-orientation routine should be as powerful as the multi-orientation routine. However, in practice the multi-orientation routine is superior to the single-orientation routine. This is a direct result of the way the two procedures extract the elastic properties from the test data. The single-orientation routine does not consider any link between the test data of the samples with a different orientation; all the material directions are processed independently. The multi-orientation routine fits one material model on the whole data set. In this way, it links the information of the different material orientations, and is able to reduce the influence of the experimental errors by averaging them out over all the samples. The presented test case illustrates clearly that, even in the case of isotropic coatings, it is advisable to machine samples in a number of different material directions and identify the elastic properties with the multi-orientation routine, especially in the situation where the substrate properties are unknown.

Note that the identification routines provided Poisson's ratios that are close to zero. Although this result is rather surprising, it is confirmed by a microstructural characterisation of the considered top coat [10]. The coating microstructure revealed a dense network of cracks. Due to these microcracks, the material is segmented. Consequently, a deformation in one direction cannot cause a deformation in an orthogonal direction, which means that there is no Poisson effect.

4 Conclusions

The elastic properties of an yttria-stabilised zirconia top coat of an air plasma sprayed thermal barrier coating were identified using a two different mixed numerical-experimental routines. In the case the elastic properties of the substrate were known, the MNET procedures were able to identify the coating properties from one set of coated samples. In the case the elastic properties of the substrate were not know, the identification of the coating properties required two sets of coated samples, i.e. two sets with a different coating thickness. Although the considered top coat was isotropic, the best results were obtained with the multi-orientation routine.

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³The Poisson's ratio of the top coat is nearly zero, a small difference in absolute terms easily results in a large relative difference. Therefore, the difference on the Poisson's ratio values were not taken into account for the calculation of the average difference with the reference properties.

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