

A Modelling Approach to Designing Microstructures in Thermal Barrier Coatings

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Abstract

Thermomechanical properties of Thermal Barrier Coatings (TBCs) are strongly influenced by coating defects, such as delaminations and pores, thus making it essential to have a fundamental understanding of microstructure-property relationships in TBCs to produce a desired coating. Object-Oriented Finite element analysis (OOF) has been shown previously as an effective tool for evaluating thermal and mechanical material behaviour, as this method is capable of incorporating the inherent material microstructure as input to the model. In this work, OOF was used to predict the thermal conductivity and effective Young's modulus of TBC topcoats. A Design of Experiments (DoE) was conducted by varying selected parameters for spraying Yttria-Stabilised Zirconia (YSZ) topcoat. The microstructure was assessed with SEM, and image analysis was used to characterize the porosity content. The relationships between microstructural features and properties predicted by modelling are discussed. The microstructural features having the most beneficial effect on properties were sprayed with a different spray gun so as to verify the results obtained from modelling. Characterisation of the coatings included microstructure evaluation, thermal conductivity and lifetime measurements. The modelling approach in combination with experiments undertaken in this study was shown to be an effective way to achieve coatings with optimised thermo-mechanical properties.

Keywords: Thermal barrier coatings, yttria-stabilised zirconia, OOF, microstructure, thermo-mechanical properties

1. Introduction

Development of Thermal Barrier Coatings (TBCs) with better performance to provide further improvements in engine efficiency is of great interest^{1–3}. This demands new coating designs and new coating morphologies, apart from research in the field of new spray materials. The usual approach to develop new coating designs is to experimentally spray and evaluate new coating morphologies by changing process parameters, i.e. to empirically derive relationships between process parameters, coating microstructures and coating properties. The drawbacks in this approach are that it is time consuming and expensive, specifically if new coating designs are to be developed^{4–5}. Deriving microstructure-property correlations by simulation would therefore be an advantage.

The material that is most commonly used as ceramic topcoat for thermal insulation is Yttria-Stabilised Zirconia (YSZ) because of its favourable properties, such as low thermal conductivity, phase stability up to 1200 °C, and good erosion resistance^{6–8}. The microstructures in YSZ coatings are highly heterogeneous, consisting of defects such as pores and cracks of different sizes. The thermal and mechanical properties of TBCs depend mainly on these microstructural features present in the topcoat^{9–12}. To achieve low thermal conductivity and high-strain-tolerant TBCs with a sufficiently long lifetime, optimization be-

tween the distribution of pores and cracks is required⁸.

Up until the late 1990s, most of the efforts to improve the performance of TBCs were made with an empirical approach, i.e. to find adequate spray parameter settings¹³. After that, more emphasis was put on understanding the fundamental relationships between coating microstructure and properties, based on both experimental and modelling methods. The over-simplification of porosity description in the analytical models limits the predictive capacity and the certainty for the complex interconnected porous structures¹³. Numerical schemes seem to be more promising in such cases as they involve a material structure that is close to the real structure.

The objective of this work was to describe a modelling approach to investigate these fundamental relationships between microstructure and thermo-mechanical properties, and obtain an optimised ceramic topcoat microstructure. The ultimate goal is to enhance the coating performance, that is reduced thermal conductivity and enhanced lifetime, and to reduce the research costs for the development of an efficient coating design, thus leading to the application of plasma-sprayed coatings in new fields. For this purpose, a Design of Experiments (DoE) was conducted by varying selected spray parameters for spraying YSZ topcoat and Object-Oriented Finite element analysis (OOF) was used to predict the thermal conductivity and Young's modulus of TBC topcoats. Thereafter, the

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Table 1: The L16 Taguchi test matrix.

Exp. No.	Current (A)	Argon flow rate (slpm)	Hydrogen flow rate (slpm)	Powder feed rate (g/min)	Torch velocity (mm/s)	Spraying angle (deg)	Substrate Temperature (°C)	Vertical speed (mm/rev)
1	-	-	-	-	+	+	+	+
2	-	-	-	+	-	-	-	-
3	+	-	-	-	-	-	+	+
4	+	-	-	+	+	+	-	-
5	-	+	-	-	-	+	-	+
6	-	+	-	+	+	-	+	-
7	+	+	-	-	+	-	-	+
8	+	+	-	+	-	+	+	-
9	-	-	+	-	-	+	+	-
10	-	-	+	+	+	-	-	+
11	+	-	+	-	+	-	+	-
12	+	-	+	+	-	+	-	+
13	-	+	+	-	+	+	-	-
14	-	+	+	+	-	-	+	+
15	+	+	+	-	-	-	-	-
16	+	+	+	+	+	+	+	+
17	0	0	0	0	0	0	0	0

best coating microstructure obtained from the experimental design and modelling analysis was fabricated again using a different spray gun, so as to verify the modelling results. Thermal conductivity and lifetime of this optimised coating was measured and compared to the reference coating sprayed with standard settings before optimisation.

II. Specimen Preparation and Analysis for DoE

(1) Sample production

The topcoat material used in this study was an agglomerated sintered $\text{ZrO}_2 - 7\% \text{Y}_2\text{O}_3$, H.C. Starck Amperit 827–864. The bondcoat material was NiCoCrAlY (Praxair Ni-171) powder, and it was sprayed on discs with the dimensions $\varnothing 25.4 \times 5$ mm made of the nickel-based superalloy Hastelloy X. The discs were grit-blasted with Al_2O_3 60 mesh before being sprayed.

Table 2: Upper and lower level values for the varied spray parameters shown in Table 1.

Parameters	Lower (-)	Upper (+)
Current (A)	300	420
Argon (slpm)	23	32
Hydrogen (slpm)	3	5
Powder feed rate (g/min)	40	70
Torch velocity (mm/s)	5	15
Spraying angle (deg)	45	90
Temperature (°C)	100	400
Vertical speed (mm/rev)	4	8

A SM-F 100 Connex gun (Sulzer Metco), which is capable of spraying on components with internal surfaces, was used for atmospheric plasma spraying. A Design of

Experiments (DoE) was constructed in the main study in this work^{5,14}. A Taguchi L16 matrix was sprayed with 17 spray settings using eight main process parameters, as described in Table 1. Other spray parameters were kept constant for all the experiments. The L16 designation refers to a two-level orthogonal matrix constituted by 17 tests, which is extrapolated from a 28 factorial design. In Table 1, the (+) and (-) signs stand for upper and lower levels, respectively, of the spraying parameter, while the 17th test represents the average level of design for each spraying parameter. The upper and lower level values of the varied spray parameters are given in Table 2. The coating thickness was kept uniform on all the sprayed samples.

(2) Image acquisition and analysis

The samples were analysed with Scanning Electron Microscopy (SEM) in the backscattered electron mode. The field of view was $180 \mu\text{m} \times 180 \mu\text{m}$. The size of the images acquired from topcoat was 1024×1024 square pixels. The acceleration voltage used for SEM was 15 kV. An automated procedure was used for the image analysis to avoid errors caused by operator variations. The images (originally in grey scale) were first converted into binary format by means of thresholding; with the matrix represented by the black and the porosity by the white. Ten images were taken across the cross-section of the coating to represent the coating microstructure. The microstructural features obtained from the image analysis were (in %) – total porosity, porosity of globular pores and porosity of cracks. The porosity of cracks was further divided in “cracks connected to pores” and “free cracks”. Connected cracks are defined as those cracks that are linked to the globular pores while free cracks are those that are not in contact with any of the globular pores, as illustrated in Fig. 1.

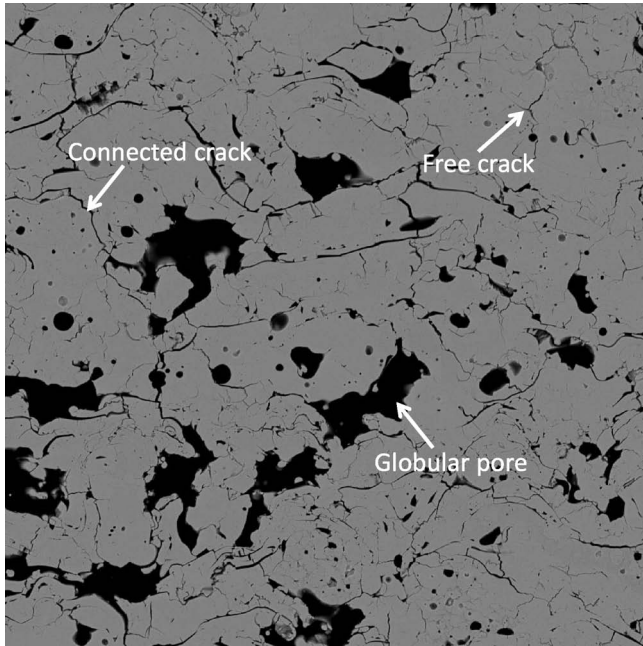


Fig. 1: SEM microstructure image illustrating globular pores, connected cracks and free cracks (the image represents an area of $180\ \mu\text{m} \times 180\ \mu\text{m}$).

(3) Modelling using OOF2

OOF is a microstructure-based finite element modelling approach that can be used to determine the macroscopic properties from two-dimensional images of real microstructures^{15–16}. The current version of the code, OOF2¹⁷, is able to convert digitized images of material microstructure into a finite element mesh, thus incorporating all the microstructural features into the model. OOF has been successfully implemented in several applications in the past, apart from computing the thermal and mechanical properties of TBCs^{18–22}. In this work, OOF2 was used to predict the thermal conductivity and Young's modulus of the topcoats of the sprayed TBCs at room temperature. The modelling procedure used in this study is described in the following sections.

(a) Pixel selection

The binary images generated during the image analysis procedure were used as input to OOF2 for finite element analysis. In the first step, two pixel groups were generated in OOF2 on the basis of the colour, to distinguish between different representative materials. The two constituents were then assigned material properties. The material inside the pores and cracks was assumed to be air.

(b) Material model and properties

YSZ coatings have been seen earlier to show non-linear stress-strain behaviour^{23–26}. The non-linearity seems to arise owing to the complex microstructure of the coatings. When a load is applied to the coatings, the opening/closing of the cracks and sliding of sprayed lamellae over each other gives rise to a non-linear response. The apparent stiffness decreases with increasing tensile stress as the cracks' faces open apart, while it increases with increasing compressive stress as the cracks' faces are closed together²³. For small deformations, however, the coat-

ings should show linear behaviour. A linear elastic material model was chosen for both YSZ and air. The bulk material properties used in the model are given in Table 3. For simplicity, the Poisson's ratio for YSZ and air were assumed to be the same in the model.

Table 3: Bulk material properties used in the model.

Material property	YSZ	Pores & Cracks (Air)
Young's modulus	200 GPa ¹⁸	142 kPa
Poisson's ratio	0.221 ²⁷	0.221 (assumption)
Thermal conductivity	2.5 W m ⁻¹ K ⁻¹ ¹⁸	0.025 W m ⁻¹ K ⁻¹

(c) Mesh generation

After the material properties had been assigned, a finite element mesh was generated over the image on the basis of features and colours present in the image by using an adaptive meshing routine. During the meshing procedure, the elements near the material interfaces were refined and the nodes were moved to the material boundaries so that the material interfaces were well defined. Finer elements were generated near the interfaces to account for the higher thermal and stress gradients.

(d) Boundary conditions and solving equations

The thermal conductivity of the coating was determined by solving the steady-state heat equation in the two-dimensional domain. A dummy temperature difference of $10\ ^\circ\text{C}$ ($= \Delta T$) was set up between the top and bottom boundaries, while the left and right boundaries were assumed to be completely insulated. As the heat flux across any cross-sectional area (A_y) parallel to top and bottom boundaries would be a constant in steady state, the following heat equation can be used to determine the thermal conductivity (K) of the coating:

$$dQ_y/dt = K \cdot A_y \cdot \Delta T / \Delta y$$

where dQ_y/dt is the heat energy flow/unit time across the cross-section and Δy is the thickness of the model domain.

For the determination of Young's modulus, a prescribed deformation of $\Delta x = 25$ pixels was applied on both left and right boundaries, while the bottom-left and bottom-right corners were fixed to move only along the x-direction. The average stress (σ_x) and strain (ϵ_x) values were calculated across all the elements in the mesh, and were used to solve Young's modulus (E) for the coating from Hooke's law given as:

$$\sigma_x = E \epsilon_x$$

A linear solver was used for solving the resulting matrix equation for the finite element problem.

It should be noted that the area representing the image is only a small fraction of the total cross-sectional area of the coating and the microstructure features might vary from one part of the coating to the other. Therefore five

SEM images were randomly selected for each experiment set from the available binary images and the average of the predicted values for those images was chosen as the final value.

(e) Limitations

There could be several reasons for the differences in the values predicted by the model and the experimental values, the major factor being the limitation of the model to the two-dimensional domain. Another factor that could affect the predicted value is the limitation of the resolution of the image, as all small cracks and pores (and specifically SEM “invisible” cracks) could not be included in the analysis. A more accurate value for material properties used for bulk YSZ would also make a difference.

III. Results from the DoE

The results from the image analysis procedure are given in Table 4. The centre-point coating in the DoE (E17) shows a typical porosity level of atmospheric plasma-sprayed TBCs of around 15 %. The lowest and highest porosity level was shown by coatings E9 and E10 respectively.

Table 4: Image analysis results for the DoE (all microstructural features are given in %).

Exp. No.	Total porosity	Globular pores	Free cracks	Cracks connected to pores
E1	15.3	10.8	2	2.5
E2	20.7	15.2	1.7	3.8
E3	12.5	8.2	2.3	2
E4	15.1	9.8	1.9	3.4
E5	19.2	13.8	2	3.4
E6	20.5	14.8	2.4	3.3
E7	18.2	11	2.7	4.5
E8	15.8	10.9	2.3	2.6
E9	11.3	5.9	2.3	3.1
E10	23.2	17.4	1.6	4.2
E11	15.9	9.4	2.7	3.8
E12	14.1	9.8	1.8	2.5
E13	20.5	14.8	1.8	3.9
E14	20.2	14.1	2.3	3.8
E15	18.1	11.2	2.5	4.4
E16	16.1	10.6	2.3	3.2
E17	16.5	11.1	2.2	3.2

Fig. 2a shows the plot between the predicted average thermal conductivity and the percentage of globular pores for all the experiments. It can be seen that predicted thermal conductivity decreases with increasing percentage of globular pores. A similar trend is seen in the plot for pre-

dicted average Young's modulus values versus percentage of globular pores, as shown in Fig. 2b. No statistically significant correlations between other microstructural features and properties were observed for these experiments. It must be kept in mind that although these plots were drawn for coating property versus one of the microstructural feature, other microstructural features were also changing between different experiments. So, in this case, the microstructural feature needs to have a very significant effect in order to establish a clear relationship.

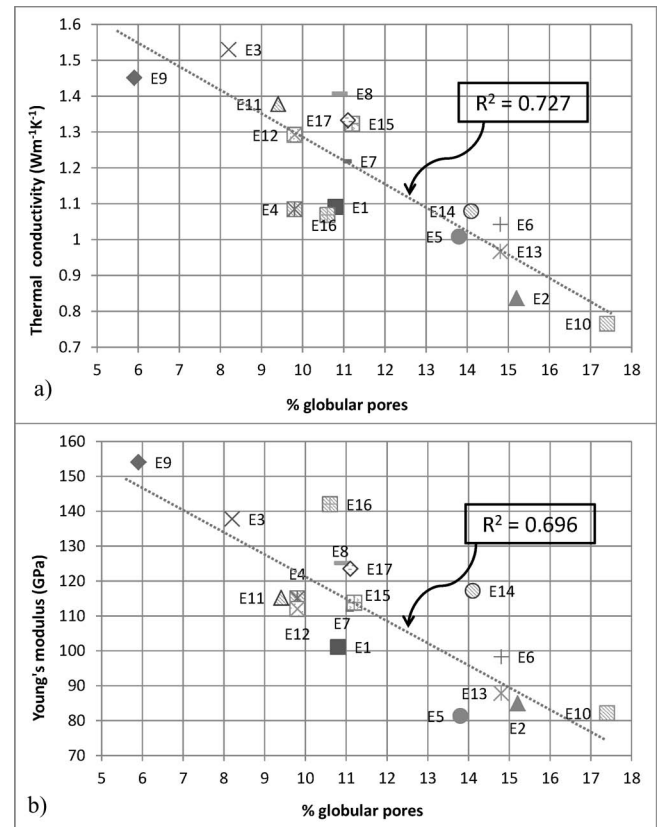


Fig. 2: Plot of predicted average (a) thermal conductivity versus % of globular pores and (b) Young's modulus versus % of globular pores.

The results indicate that OOF2 is able to distinguish between different microstructures. It can be clearly seen that E10 coating exhibits the lowest thermal conductivity and Young's modulus values, and also exhibits the highest porosity level.

The evaluation of Young's modulus provides an indication of the coating integrity, porosity and bonding quality between splats. It also provides an idea of the thermal stress developed in the coating during operation since the modulus is roughly proportional to the induced thermal stresses²⁸. It is believed that a coating with a low Young's modulus will perform better in lifetime tests owing to its higher strain tolerance. Thus, based on the modelling results, it can be concluded that E10 is the best coating produced in this study since it has low thermal conductivity and a low Young's modulus, which would imply long lifetime in operating conditions.

In a previously published statistical study done by the same research group, it was seen that the combination of globular pores and connected cracks has the most signifi-

cant influence on reducing thermal conductivity²⁹. Fig. 3a shows the microstructure for the E10 coating. The presence of large globular pores and several cracks connected to these pores can be clearly seen. These globular pores are in the size range of approximately 30–50 μm . This effect was also noted in another recent work done by the authors in which polymer porosity former was used in the topcoat material to increase the porosity content in the topcoat which can be obtained by burning off the polymer after spraying³⁰. Similar microstructures were seen in that study which resulted in a coating with low thermal conductivity compared to the reference coating in that study. Fig. 3b shows the microstructure for the E17 coating, which represents the average of the parameter levels. A typical microstructure for plasma-sprayed TBCs with a few pores and cracks can be observed.

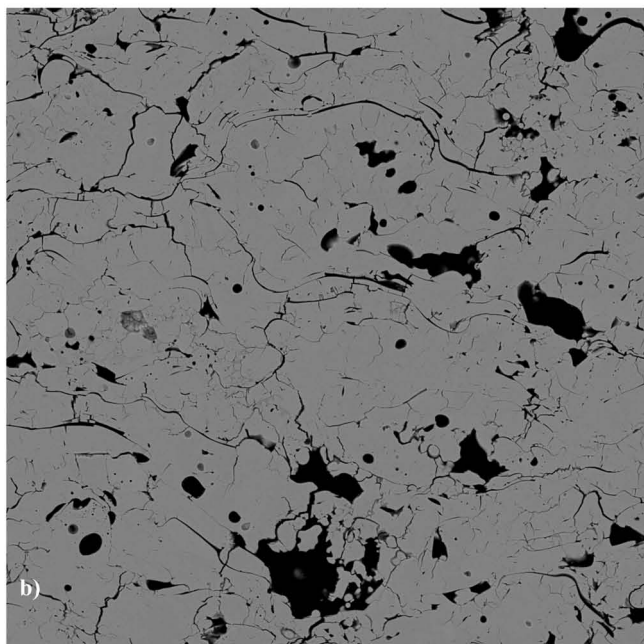
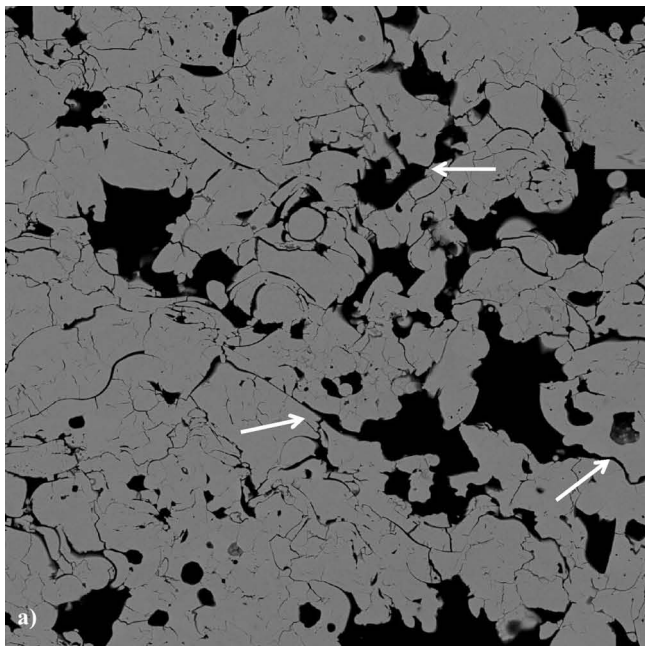


Fig. 3: SEM microstructure image for (a) E10 and (b) E17 coatings (the image represents an area of 180 μm x 180 μm).

The modelling results as well as previous studies indicate that large globular pores, i.e. pores in size range of 30–50 μm , with connected cracks are beneficial to achieve a high-performance coating^{29,30}. For verification of the results obtained from finite element modelling, a coating system with similar microstructure was sprayed and tested as explained in the next section.

IV. Verification of Modelling Results

The modelling approach to optimise microstructures is highly beneficial as it is less time-consuming and cheaper than the traditional experimental approach. However, an experimental verification of the modelling results is required. For this purpose, new samples were sprayed and analysed, and compared with the modelling results.

(1) Sample production

A different spray gun was used for these tests to analyse the effect of changing the spray gun on the results, and to assess the capability of knowledge transfer from one gun to another in terms of achieved microstructures. The atmospheric plasma spray F4 gun (Sulzer Metco) was used to spray bondcoats and topcoats on Hastelloy-X substrates using the same materials as used in the previous study. Several spray settings were examined with current, spray distance and powder feed rate being the varied spray parameters. The intention was to fabricate a coating exhibiting a microstructure with large globular pores, i.e. pores in size range of 30–50 μm , with connected cracks. An additional coating was sprayed with mean values of varied spray parameters for reference. The upper and lower level values of the varied spray parameters are given in Table 5. Coating thickness was kept uniform for all the sprayed samples. The microstructure was analysed with SEM.

Table 5: Upper and lower level values for the varied spray parameters during spray trials with F4 gun.

Parameters	Lower (-)	Upper (+)
Current (A)	600	750
Spray distance (mm)	70	140
Powder feed rate (g/min)	90	180

(2) Thermal conductivity measurements

The thermal conductivity of the samples was measured at room temperature with the well-accepted laser flash technique. A Netzsch Laser Flash Apparatus LFA 427 system (Netzsch Gerätebau GmbH, Germany) was used. First, samples of substrate and substrate sprayed with only bondcoat were analysed. The thermal conductivity of only the complete TBC system with both topcoat and bondcoat was then measured for the samples. Thermal conductivity of the topcoat was calculated later from the measured thermal conductivity values and thicknesses of individual layers. All samples were placed in a furnace with ambient atmosphere at 1100 $^{\circ}\text{C}$ for 25 h before measurement. An average of two samples sprayed for each set was considered to be the final value.

Thicknesses of individual layers were measured using optical microscopy from sample cross-sections. The specific heat capacity was measured using differential scanning calorimetry. The density of topcoats was calculated using their respective porosity content and the bulk value for YSZ from the simple rule of mixtures. The porosity content was calculated using the same image analysis procedure as described earlier in section II.(2).

(3) Thermo-cyclic fatigue lifetime

Assessing the lifetime of TBCs is a challenging task since testing them in real environments is very time-consuming. Accelerated and simplified tests are used to reduce testing times by exposing the TBCs to harsher environments than reality. For this work, thermo-cyclic fatigue (TCF) tests were used to assess the lifetime. In TCF tests, samples undergo a continuous cyclic process until failure. Each cycle involves the samples first being heated in a furnace at 1100 °C for 1 h and then cooled for 10 min using compressed air to around 100 °C. At the end of each cycle, an image of the sample surface is recorded using a webcam thus avoiding the need for an operator to be present at all times. The failure criterion is deemed to be 20 % visible spallation of the topcoat. An average of two samples sprayed for each set was considered to be the final value.

Table 6: Image analysis results for the samples sprayed with F4 gun (all microstructural features are given in %).

Coating	Total porosity	Globular pores	Free cracks	Cracks connected to pores
Optimised	22.8	19.0	1.6	2.2
Reference	10.7	5.9	2.9	1.8

(4) Results

The image analysis results for the two coatings are given in Table 6. SEM microstructure images for the optimised coating and the reference coating are presented in Fig. 4. It can be clearly seen in Fig. 4a that the microstructure has large globular pores with connected cracks, which is similar when compared to the E10 coating presented in Fig. 3a. The porosity level for this coating is also at a similar level of around 23 % as given in Table 6. The reference coating presented in Fig. 4b has a typical microstructure for a plasma-sprayed TBC with a few pores and cracks, which is again similar to the E17 coating presented in Fig. 3b. Thus, it can be concluded from Fig. 4 that microstructures similar to the coatings sprayed previously were successfully achieved, even when a different spray gun was used. The spray parameters that were observed to significantly influence the microstructural design were current and spray distance. Low current and long spray distance were used to fabricate the optimised coating microstructure.

Measured and predicted thermal conductivity values for the two coatings are presented in Fig. 5a. It can be noticed that the optimised coating exhibits lower thermal conductivity compared to the reference coating in a comparison of the laser flash results. The modelling results also show the same behaviour with significantly reduced thermal con-

ductivity for the optimised coating. The discrepancy between the measured and predicted values can be attributed to the limitations of the finite element model discussed in section II.(3)(e). It must be noted here that SEM images of as-sprayed samples were used for prediction while heat-treated samples were used for measurements. As the thermal conductivity usually increases after heat treatment owing to sintering, this effect would also contribute to the discrepancy between the measured and predicted values apart from the previously mentioned reasons.

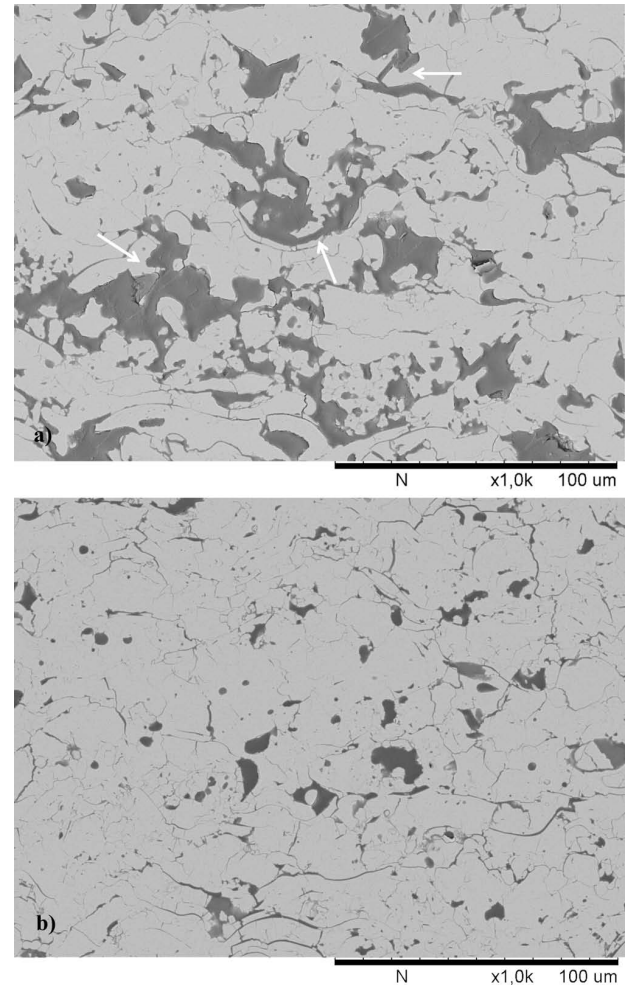


Fig. 4: SEM microstructure image for (a) optimised and (b) reference coatings.

Predicted Young's modulus and TCF lifetime values for the two coatings are presented in Fig. 5b. Higher TCF lifetime and lower Young's modulus were shown by the optimised coating, which strengthens the belief that a coating with a low Young's modulus will perform better in lifetime tests owing to its higher strain tolerance.

These results indicate that, as predicted by the model earlier, large globular pores with connected cracks inherited within the microstructure are beneficial for the coating performance.

V. Summary and Conclusions

In the first part of this work, thermal and mechanical properties of TBCs for a Taguchi L16 matrix sprayed with 17 spray settings using eight main process parameters were predicted using finite element analysis. The lowest thermal conductivity and Young's modulus were shown by

a coating exhibiting large globular pores with connected cracks inherited within the microstructure. In the second part, similar microstructures were sprayed with a different spray gun. Thermo-mechanical properties were predicted using the same finite element model while thermal conductivity and thermo-cyclic fatigue lifetime were measured experimentally. In this way, the modelling results obtained in the first part of this work were verified as the coating with large globular pores with connected cracks performed better compared to the reference coating sprayed with standard settings before optimisation. Low current and long spray distance can be used to fabricate the optimised coating microstructure.

This work shows that the modelling approach in combination with experiments can be used as a powerful tool to achieve optimised microstructures. This approach is both time-saving and cost-effective.

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