

An application of scanning thermal microscopy: Analysis of the thermal properties of plasma-sprayed yttria-stabilized zirconia thermal barrier coating

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Abstract

Yttria-stabilized zirconia coatings were deposited on Ti–6Al–4V substrate by plasma spraying. The thermal properties of the as-sprayed coating were characterized using a scanning thermal microscopy that allows thermal conductivity to be mapped down to the submicrometer scale. The analysis of the thermal properties shows the variations in thermal conductivity with the characteristics of the materials. The relation between microstructural features and thermal conductivity was discussed in correlation with the heat conduction mechanism in different layers. Based on the experiments, the thermal probe was calibrated and the thermal conductivities of the coating and the substrate were estimated. Experimental results and thermal conductivity estimation demonstrate that the SThM analyses can be used as a powerful tool for the thermal property and microstructure analysis of plasma-sprayed thermal barrier coating.

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1. Introduction

Although Ti–6Al–4V is one of the most widely used titanium alloys in aerospace engineering due to its unique high strength–weight ratio,¹ it shows a lack of mechanical and thermal properties at high temperature. Therefore, protective coatings are frequently required to insulate the alloy surfaces for the successful application and performance of this alloy in aerospace gas turbines.

Thermal barrier coatings (TBCs) have played an increasingly important role in enhancing gas turbine engine durability and performance.² For example, a TBC on Ti–6Al–4V alloy, in which the ceramic side contacting with high temperature offers heat resistance, and the metallic side contacting with low temperature provides mechanical strength and thermal conductivity. The TBC material almost universally used is yttria-stabilized zirconia (Y₂O₃-stabilized ZrO₂). This ce-

ramic has performed admirably as a TBC because of its favourable combination of properties, including low thermal conductivity, phase stability to 1400 °C, and good erosion resistance, etc.^{2,3}

Studies on thermophysical properties are of significance not only for fundamental research but also for applications of materials. It is known that the heat transport properties of polycrystalline materials are strongly affected by their characteristics and microstructural features.^{4,5} In general, TBC materials and substrate are different materials, different chemical compositions and microstructural features determine their thermal properties at high temperature.

Scanning probe microscopes (SPMs) provide one of the few methods of imaging structures, observing phenomena, and manipulating objects with nanometer scale spatial resolution,⁶ and the invention of the scanning thermal microscopy (SThM) provides a tool with which the thermal properties of materials can be evaluated on a very small scale. The SThM is based on an atomic force microscope (AFM), but uses a specialized thermal probe instead of con-

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ventional SiN_x tip of AFM. The SThM is developed to give simultaneously surface topography image and thermal property image of materials with micrometer or sub-micrometer spatial resolution. In SThM, the interaction between probe tip and sample is based on the heat flux.⁷ Therefore, differences in temperature or thermal conductivity constitute the imaging contrast. The SThM has many potential applications, one of them is to study local variations in surface thermal properties.

Some earlier works have studied the thermal properties of Y_2O_3 -stabilized ZrO_2 coatings,^{8–12} most of them dealt with the influence of temperatures, coating compositions, defect distribution, grain size, or plasma-spraying process variables on the thermal properties of coatings. Many interesting results have been obtained from these studies. However, no work dealt with the analysis of the thermal properties of TBC materials by means of SThM techniques. To assist in this effort, we attempt to use SThM to characterize the thermal properties of plasma-sprayed Y_2O_3 -stabilized ZrO_2 coatings on Ti–6Al–4V substrate.

In this report, Y_2O_3 -stabilized ZrO_2 coatings on Ti–6Al–4V alloy were performed by plasma-spraying method. A SThM was used to map the thermal conductivity images, the thermal probe was then calibrated and the thermal conductivities of the coating and the substrate were estimated. The influence of the material characteristics and microstructural features on heat conduction was discussed in correlation with the heat conduction mechanism in different materials.

2. Experimental details

2.1. Preparation of Y_2O_3 -stabilized ZrO_2 coating

Ti–6Al–4V wafers of dimensions \varnothing : 35 mm \times 10 mm were polished, degreased in benzene using ultrasonic cleaning, rinsed in deionized water, and dried. Films of ZrO_2 with 7 wt.% Y_2O_3 were deposited by plasma spraying onto two sides of the Ti–6Al–4V substrate at room temperature. A Metco 6M plasma torch (Sulzer Metco AG, Switzerland) was used to spray the Y_2O_3 -stabilized ZrO_2 coating. The final coatings with thickness of 70 μm on one side and 90 μm on the other side were sprayed. Table 1 summarizes the parameters for plasma spraying.

Table 1

Plasma spraying parameters for the investigated zirconia coatings

Powder composition	$\text{ZrO}_2 + 7 \text{ wt.}\% \text{ Y}_2\text{O}_3$
Particle size (nm)	80–140
Power input (kW)	41
Prim./sec. gas (slpm)	45 Ar/15 H_2
Carrier gas flow (slpm)	3.5 Ar
Powder feed rate (g/min)	20
Stand-off distance (mm)	120

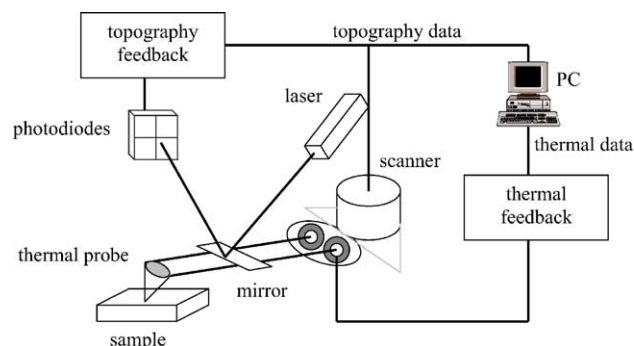


Fig. 1. Schematic diagram of the SThM set-up.

Microstructural examination was performed using a JSM 5800 scanning electronic microscopy (SEM) both on the cross-section and on the coating surface after plasma spraying and polishing according to a standard sample preparation routine.

2.2. SThM analysis

For the thermal conductivity analysis, a TopoMetrix SThM was used in which thermal imaging is achieved using a resistive thermal element incorporated at the end of a cantilever that makes it possible to achieve an AFM type feedback. The thermal element consists of a bent filament (5 μm diameter) of platinum/10% rhodium. Fig. 1 depicts the set-up of the SThM used in this work.

In SThM, normally two working modes are available: ‘temperature contrast mode’ and ‘thermal conductivity contrast mode’. In the present work, the ‘thermal conductivity contrast mode’ was used, in which the thermal probe functions as a resistive heater. The control circuit uses a feedback loop to adjust the voltage applied to the bridge in order to keep the thermal probe at a constant temperature. When the probe is brought in contact with the test specimen, the probe tip cools due to heat conduction from the probe tip into the specimen. This cooling will reduce the resistivity of the probe. The current through the probe will then be increased by the bridge feedback circuit, until the temperature of the probe, and hence its resistance, is again equal to the target operating value. The amount of power required to maintain the probe at a constant temperature is directly related to the thermal conductivity of the test specimen.

The specimens used for thermal conductivity analysis were cut from the plasma-sprayed sample. The specimens were embedded in a resin so that the scanning could be carried out at the edge of the specimen. The specimens were cautiously polished in order to avoid the surface influence on the heat conduction. The SThM scanning was carried out on the cross-section of the sample that includes the coating layer and the substrate. A series of scanning of different dimensions were carried out, conducted at a temperature of 116.9 °C in order to avoid the influence of water on heat conduction on the sample surface.¹³ The scan rate is 10 $\mu\text{m/s}$,

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