



# Microstructural evaluation of laser remelted gadolinium zirconate thermal barrier coatings

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## ABSTRACT

Thermal barrier coatings having NiCoCrAlY bond coat and Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> top coat were produced utilizing HVOF and APS processes. Then the coatings were subjected to CO<sub>2</sub> continuous wave laser remelting by using different laser parameters. The effect of laser remelting process on the surface roughness, microstructure, grain size, hardness and phase transformation was investigated. The microstructural characterizations showed that a smooth, flat and dense surface having a network of segmented cracks was observed and open porosities were sealed through-out with a thin (~35 μm) remelted layer. The surface roughness value decreased from 8.3 μm to 2.9 μm for as-sprayed and laser remelted specimens respectively. Furthermore, equiaxed and columnar grains that are perpendicular to the surface were formed and the grain sizes decreased from 7.03 μm to 3.69 μm due to laser process parameters. The hardness value increased from 10.66 GPa to 12.73 GPa with decreasing grain size. XRD patterns of as-sprayed and laser remelted top coats indicated that the structure of the coating has been changed due to preferred orientation after surface modification by laser remelting.

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## 1. Introduction

Thermal barrier coatings (TBCs) are used on some critical parts such as blades, vanes and combustor chambers of gas turbines to protect metallic substrate, to provide thermal insulation and to improve the efficiency of gas turbines [1,2]. In commercially used coating, generally an atmospheric plasma sprayed (APS) TBC's system is composed of different layers including (1) yttria-stabilized zirconia (YSZ) based ceramic top layer that is a mixture of tetragonal, cubic and small amount of monoclinic phases, (2) an oxidation resistant bond layer (typically MCrAlY) and (3) a thermally grown oxide (TGO, predominately alpha-alumina) layer [3,4]. This type of coatings has significant amount of microstructural defects such as cracks and porosities in the ceramic top coat. Microstructural defects which are phonon scattering centres and strain tolerant zones reduce both thermal conductivity and stress build up due to the thermal expansion mismatch between metal and ceramic [1–5].

Due to increasing demands for more efficient next generation gas turbines, it is necessary to increase the turbine inlet and operation temperature. The highest operation temperature reached is 1200 °C for YSZ. Above this temperature, severe damage takes place as a result of allotropic phase transformation [5,6]. Moreover, researchers indicate that limits for YSZ/MCrAlY system have been achieved [4–8]. Therefore, in order to push the limits, new strategies could be performed. The first strategy is to use alternative materials showing superior high

temperature performance by having high thermal properties against harsh environment. Second is to modify the surface of the coatings to have better structure and morphology.

In light of the first strategy, using rare earth oxides with pyrochlore or defect fluorite-type structure (A<sub>2</sub>B<sub>2</sub>O<sub>7</sub>) having high phase stability at elevated temperatures compared to YSZ could be a solution. Among the rare earth oxide materials, gadolinium zirconate (Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>) seems to be the most efficient one in terms of its comparable thermal expansion coefficient ( $10.4 \times 10^{-6} \text{ K}^{-1}$ ) to that of YSZ ( $11.6 \times 10^{-6} \text{ K}^{-1}$ ), low thermal conductivity ( $0.65 \text{ Wm}^{-1} \text{ K}^{-1}$ ), high phase stability (above 1200 °C), and a very good resistant to damage by molten Ca–Mg–Al–silicate glass (CMAS attack) and Na<sub>2</sub>SO<sub>4</sub> + V<sub>2</sub>O<sub>5</sub> (hot corrosion). Thus, both fluorite and pyrochlore forms of GZ are considered as a promising material for TBC applications due to their superior properties [1,5–13].

Surface modification by laser beam can be regarded as a second strategy. This technique has been used for approximately ten years to improve the properties of TBCs [14,15]. Studies have indicated that remelting and solidification during laser treatment lead to reduction in the surface roughness and specific surface area [14–22]. Furthermore, depending on laser process parameters, along with reduction in the surface roughness; a columnar structure having controlled (varied depending on the laser parameters) network of segmented cracks forms sealing porosities beneath the surface. Pidani et al. [15] reported that laser parameters (scanning speed, laser power and frequency) changed the structure of segmented crack network and they found the optimum parameters as 20 mm/s scanning speed and 80 W laser power to obtain regular (equiaxed) network of continuous segmented cracks for ceria–yttria stabilized zirconia (CYSZ) TBCs. Batista et al.

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**Table 1**  
APS and HVOF spraying parameters of ceramic top coat and bond coat.

Parameters	NiCoCrAlY (HVOF)	GZ (APS)
Hydrogen flow rate (l/min)	–	42
Argon flow rate (l/min)	–	18
Ampere (A)	–	600
Voltage (V)	–	65
Oxygen flow rate (SLPM)	140	–
Air flow rate (SLPM)	385	–
Propane flow rate (SLPM)	90	–
Spray distance (mm)	250	70
Spray angle to surface (°)	90	90
Powder feed (lb/h)	28	5.5

[20] showed that laser parameters such as scanning speed and track overlapping had a strong effect on the density of segmented crack networks of YSZ. It has been shown that, it is likely to control the network of segmented cracks by changing the laser parameters. This segmented cracks improve the strain accommodation capability thus, thermal cycle life time of the TBCs. Lee et al. [31] tested the thermal cycling behaviour of the laser surface modified YSZ TBCs by heating the coatings to 1100 °C for 1 h and then quenched. Test results showed that the thermal cycling performance of the laser surface modified coatings are about two times higher than that of non-treated coating. In another study, the surface of CYSZ TBCs was treated by laser beam and their thermal shock behaviour was examined by holding at 950 °C for 5 min, following water quenching. According to the results an improvement in life time took place on the laser surface modified coatings and this situation was attributed to the segmented cracks produced by laser surface modification process [16]. In addition to this, it can be concluded that beneficial effect of the segmented cracks achieved by laser surface modification would not be lost during service. Therefore, thermal cycle life time of GZ based TBCs can be further improved by laser surface modification process.

Another promising effect of laser modification is to increase the resistance of TBCs against molten salt and oxygen penetration [14–22,25–28]. As known, the microstructural defects of TBCs provide infiltration path for molten hot corrosion and CMAS products to attack the coating [13–15,33]. Moreover, the surface roughness of plasma sprayed TBCs decreases the hot corrosion resistance due to excessive reactive specific surface area between coating layer and molten salts [19,25,28]. Previous studies have shown that, after laser remelting process, molten salt and oxygen penetration into the coating are obstructed by sealing open porosities and decreasing surface roughness [14–22,25–28]. In a research that was conducted by our group, laser surface modified CYSZ TBCs were oxidized at 1100 °C for different duration (50–200 h). According to results, the thickness of the TGO layers of the laser surface modified TBCs was thinner than that of the as-sprayed TBC. This situation was attributed to reduced oxygen penetration due to sealing and elimination porosities by laser surface modification [14]. From these studies it is likely to say that, the benefits acquired by laser modification do not disappear by annealing. Laser remelted dense layer also has a potential to improve resistance of the gadolinium zirconate TBCs to CMAS, hot corrosion and oxidation penetration because of a smooth and dense thick layer having a crack network as well as improvement in thermal cycle life time.

There are many studies on the laser surface modification of the plasma sprayed YSZ and CYSZ TBCs [14–22,26–35]. In this study, for first time in literature laser surface modification of plasma sprayed Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> (GZ) thermal barrier coatings was investigated to determine the laser remelting parameters. Therefore, this study can be regarded as necessary and important. According to literature, desired properties from the remelted layer were 20–50 μm melting depth, non-separation from the as-sprayed layer, smooth surface (with surface roughness value (Ra) lower than 5 μm) and a distribution of the crack network that is equiaxed [15–22]. During the study, first, GZ thermal

barrier coatings were produced by using HVOF and APS techniques and second GZ TBCs were subjected to a CO<sub>2</sub> continuous wave laser surface modification process. The effect of laser remelting on the surface roughness, microstructure, grain size, hardness and phase transformation of the GZ coatings were investigated.

## 2. Experimental procedures

The feedstock powder for ceramic top coat was a commercial gadolinium zirconate (Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>, Treibacher Industrie AG, Althofen-Austria, d<sub>10</sub> = 53 μm, d<sub>50</sub> = 89 μm, d<sub>90</sub> = 137 μm) with an irregular morphology. The chemical composition of the Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> powder was determined to be “Gd<sub>2</sub>O<sub>3</sub> 59.97 wt.%, ZrO<sub>2</sub> 39.11 wt.%, HfO<sub>2</sub> 0.82 wt.%, Al<sub>2</sub>O<sub>3</sub> 0.07 wt.% and Y<sub>2</sub>O<sub>3</sub> 0.03 wt.%” by XRF (X-Ray Fluorescence, RIGAKU ZSX Primus II) analysis. NiCoCrAlY (Ni 23Co 20Cr 8.5Al 4Ta 0.6Y, Amdry 9951, SulzerMetco, –37 μm and spherical shaped) was used as a bond coat powder. Before the coating processes, stainless steel (C 0.08 wt.%, Cr 18 wt.%, Fe 68 wt.%, Mn 2 wt.%, Ni 11 wt.%, Si 1 wt.%) substrates with a diameter of 25.4 mm and 2 mm in thickness were subjected to cleaning and grit blasting process. After that, NiCoCrAlY powder was sprayed onto the prepared surface with a total thickness of 80 ± 10 μm by high-velocity oxy-fuel (HVOF) process (2700 DJHE DJ, SulzerMetco). GZ ceramic top coat powders were sprayed onto the bond coating layer by APS system (9MBM, SulzerMetco). The total thickness of the ceramic top layer was about 350 ± 20 μm. The spraying parameters of HVOF and APS processes were given in Table 1.

The coated sample surfaces were laser treated by using a continuous CO<sub>2</sub> laser system (PRC STS 3001 model, max 3.0 kW). Laser power density on the surface, the scanning speed and laser distance (distance to the surface from the laser torch) were selected as parameters (Table 2). The angle of laser beam incidence was vertical (90°) to the surface of the specimens. To determine the effect of laser parameters, single laser beam tracks were generated on the coatings. Laser beam tracks were evaluated considering melting depth, cross-sectional damage (state of separation from the as-sprayed layer), surface quality (smoothness and spallation) and the distribution of the crack network (state of being equiaxed) in the remelted layer. After that the whole surface area of the coatings was scanned with a laser beam, generating multiple parallel tracks with the CNC (Computer Numerical Control) X–Y table of the laser system.

Microstructural characterization of as-sprayed and laser remelted TBCs was carried out with an optical microscope (LEICA-DMRX) and field emission (JSM 7000F, JEOL) scanning electron microscope (FESEM). The surface roughness value of the specimens was measured by a Veeco WYKO NT1100 optical profilometer. The grain sizes were calculated by the intercept method, using high magnification FESEM micrographs. Hardness (HV) values were determined under a load of 1.96 N by using a micro-hardness tester (VHMOT, Leica Corp.) and Vickers indentation method. All indentations were performed on the laser remelted TBC's surfaces considering ASTM E384-11 standard test method. The average value of 15 measurements for each sample was taken for hardness evaluation. Phase characterization was performed using X-ray diffractometry (XRD; MiniFlex, Rigaku Corp.) within the range of 10 to 90° using Cu Kα radiation.

**Table 2**  
Laser remelting parameters for GZ coating.

Parameters	Name of laser tracks				
	LGZ-1	LGZ-2	LGZ-3	LGZ-4	LGZ-5
Laser power (W)	418	418	418	865	673
Laser power density (MW/m <sup>2</sup> )	110	110	110	90	70
Scanning speed (mm/s)	90	120	150	150	150
Laser distance (mm)	10	10	10	14	14
Laser spot size (mm)	2.2	2.2	2.2	3.5	3.5
Angle of laser beam incidence	90°	90°	90°	90°	90°

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