



A new injection system for spraying liquid nitrates in a low power plasma reactor: Application to local repair of damaged thermal barrier coating

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ABSTRACT

In addition to the search for new Thermal Barrier Coating (TBCs) systems with increased reliability over very long time periods, the repair of current systems is a technological and economic issue for both civilian and military engine end-users. This paper describes the latest version of the deposition process known as the Low-Power Plasma Reactor (LPPR) process, specially developed to repair locally damaged TBCs. The LPPR process enables micro/nanostructured TBCs to be made from nitrate salts in aqueous solutions, which are sprayed in an Ar/O₂ plasma discharge at low power (240 W) and transformed into oxide coatings. A new injection device was designed to produce a fairly homogenous and reproducible spray to repair partially spalled APS and EB-PVD TBCs deposited on small flat coupons. The microstructure and the stability of the LPPR TBCs were assessed, in particular using SEM observations, during ageing tests under various time/temperature conditions. The Particle Image Velocimetry (PIV) technique and associated modeling have proved that the nitrates impact the substrate in a liquid state even in the presence of plasma and a vacuum. Due to the liquid state of the precursors, the new LPPR TBC seals the damaged areas and deeply infiltrates all porosities and failure cracks in the original coatings. This research has enabled the new version of the LPPR process to be validated as a simple, efficient, cheap and promising way to repair locally damaged TBCs.

1. Introduction

Thermal barrier coatings (TBCs) are multifunctional, multilayer systems used to protect the metallic parts in the hottest zones of gas turbine engines, in particular the combustor, blades and vanes [1,2]. These systems consist of a porous ceramic layer (mainly ZrO₂ + 8 wt% Y₂O₃ or YpSZ) deposited on an alumina-forming metallic bond coat in contact with the nickel-based superalloy substrate. Today, industrial TBCs are routinely manufactured by Air Plasma Spraying (APS) mainly for static components or Electron Beam Physical Vapor Deposition (EB-PVD) for rotating components [3]. Several promising new TBC deposition techniques are under development, such as Suspension Plasma Spraying (SPS) [4–6] or Plasma Sprayed Vapor Physical Deposition (PS-PVD) [4,7]. TBCs are designed to increase the gas inlet temperature and, therefore, the engine efficiency and/or to extend the lifetime of the components, leading to substantial economic benefits.

In operation, TBCs are exposed to severe thermal and mechanical

stress conditions [8–10]. The structure and composition of the various layers change over time due to sintering of the porous ceramic layer, oxidation of the bond coat and interdiffusion with the substrate. As a result, the properties of each layer are affected, as well as the interfacial toughness. These changes lead to the formation of delamination cracks in the bond-coat/ceramic interface zone, and to local spallation of the ceramic layer, which reduces the life of the engine. In addition to these intrinsic degradation patterns, other interactions with the environment (such as erosion, foreign object damage and CMAS - Calcium-Magnesium Alumino-Silicate-corrosion) accelerate TBC failure [11].

In addition to the development of new TBC systems with enhanced reliability and performance [1,2], the repair of current systems is a technological and economic issue for both civilian and military engine end-users [12]. While numerous patents have been filed which locally repair the TBC, most of these are based on Physical Vapor Deposition, Plasma Spray or tape casting techniques [13–17]. Several innovative papers related to this issue have also been published. They study the

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possibility of reinforcing and stabilizing the crack network of a TBC by using a sol-gel process [18] with a laser remelting process [19]. These techniques can heal the cracks and slow down the TGO growth rate but, because of the low yield of the sol-gel process, they may not easily repair the few square millimeters of spalled areas commonly reported around the oxidized components. For this reason, the complete removal of the ceramic coating with its bond coat, followed by a re-application of both bond coat and TBC, remains the routine procedure for most repairers. Therefore, the development of an innovative, fast and efficient solution for the local repair of oxides is as important today as it has ever been. This paper deals with the implementation of a previously designed Low-Power Plasma Reactor (LPPR) process [20–24] for the local repair of damaged TBC coated components.

Firstly, the new injection system of the LPPR is described as well as the principle of deposition and repair. The morphology and the microstructure of the deposited oxides, called YpSZ LPPR throughout the article and investigated using SEM analyses, are presented. These investigations were conducted on YpSZ LPPR deposited on both flat and infiltrated coupons of APS and EBPVD damaged samples. Finally the behavior of the precursor droplets in the reactor is examined through experimental measurements using a Particle Image Velocimeter (PIV) and theoretical simulation using a previously developed model. It demonstrates that the precursor droplets of the spray impact the substrate in a liquid form, which is the necessary condition for a good infiltration into the APS or EBPVD coating to be repaired.

2. Materials and methods

2.1. Design of a new injection system

The first version of the Low-Power Plasma Reactor, described in [20], was developed to elaborate thermal barriers from aqueous solution sprayed in cold plasma. The plasma discharge is initiated under low pressure, by means of external inductive coils that enable energy transfer from a 40 MHz RF generator (power range: 50–550 W). The plasma gas consists of a mixture of argon and oxygen. The first version of the injection system was a homemade device consisting of a septum, a manual valve and a 1/4-inch steel tube through which gases and liquid precursors circulated. In the first step, the precursor solution was injected by the experimenter through the septum. In the second step, the manual opening of the valve enabled the introduction of both solution and gases through the steel tube, which sprayed the solution into the reactor. This injection system coupled with the plasma reactor allowed the introduction of droplets of an aqueous solution containing the selected precursors, e.g., nitrates, into the Ar/O₂ plasma discharge. Although this initial injection system permitted the successful deposition of thick and complex oxides onto a plane surface, it frequently became clogged and prevented the formation of reproducible sprays. As a result, as the experiment progressed the deposits became increasingly less homogeneous in terms of morphology (presence of large voids) and thickness. This injection system was modified to increase the quantities of the solution to be introduced and to improve the homogeneity and reproducibility of the spray [21–23]. The injection was still administered manually but using a 530 µm diameter capillary, instead of a 1/4-inch steel tube, to aspire and spray the solution in the plasma. Although this system was suitable for covering large flat surfaces, its spray was too dispersive to effectively and rapidly cover small localized areas. Moreover, problems of clogging were still observed and reproducibility was not completely ensured. For the local repair (0.01–5 cm²) of damaged components, a new configuration of the injection system was subsequently designed (Fig. 1). The objective was to significantly improve the homogeneity and reproducibility of the nitrate precursor spray for future industrial repairs. The new injection device consists of a mixing chamber connected to a pulsed valve (Fig. 1). Two capillaries (530 µm in diameter) are used to introduce argon and the aqueous solution into the chamber. These capillaries allow the introduction of

constant mass flows of argon and nitrates. The nitrate precursors (Aldrich, 99% purity) are dissolved in deionized water at the following concentrations: $[\text{ZrO}(\text{NO}_3)_2] = 3.4 \cdot 10^{-1} \text{ mol L}^{-1}$ and $[\text{Y}(\text{NO}_3)_3] = 2.96 \cdot 10^{-2} \text{ mol L}^{-1}$, corresponding to an expected composition of $\text{ZrO}_2 + 8 \text{ mol\% YO}_{1.5}$ or 7.4 wt% Y_2O_3 . The mixture of argon gas (typically 1000 mL min⁻¹) and aqueous solution (3.3 mL min⁻¹) is automatically injected for 1.5 min through the pulsed valve (opening and closing times 0.8 and 1 s respectively). As a result, the pressure in the reactor increases following the opening of the valve (typically up to 900 Pa) and decreases after its closing (typically down to 530 Pa).

The injection system works as an atomizer at low pressure, with a nozzle of 0.7 mm in diameter (manufactured by Spraying Systems Co), which enables small droplets to be produced by dual-flow atomization. A Particle Image Velocimeter (PIV) system developed by DANTEC Dynamics was placed between the injection system and the substrate in order to study the characteristics of the spray. This allows the velocity and diameter distributions of the droplets to be determined along the reactor axis, even in the presence of the plasma, by using a laser.

The chemical reactions occurring in the Ar/O₂ discharge lead to the formation of oxidant species, such as O and OH, reagents which have the ability to transform precursor nitrate salts into oxides [23]. After the injection of the liquid precursors, the plasma is maintained, in order to completely transform the deposited nitrates into an oxide layer (“post-treatment” step, typical duration about 8.5 min). It is worth noting that the temperatures of both the substrate and the reactor remain below 300 °C during post-treatment. The discharge is then stopped. After the reactor is cooled down, another cycle (injection followed by a post-treatment and cooling) can be carried out. This cycle is repeated until the desired thickness of the oxide layer is reached. After the deposition, the oxide ceramic coating can be annealed at $T = 1000$ or 1100 °C for a few hours, in order to enhance its crystallization. This annealing is optional and could be performed during the first use in a real turbine engine.

2.2. Coated and repaired specimens

Yttria partially stabilized zirconia (YpSZ) LPPR layers were deposited on flat coupons (25 mm in diameter) of dense alumina substrates. The process parameters were as following: power: 240 W; plasma gases: Ar (2000 mL min⁻¹) and O₂ (275 mL min⁻¹); Ar for the injection system: 1000 mL min⁻¹ and injection rate of the aqueous solution (containing the nitrate precursors): 3.3 mL min⁻¹. The distance between the injector and the substrate was 24 cm. 93 cycles of 1.5 min injection followed by 8.5 min post-treatment were carried out, in order to obtain a ~100 µm thick ceramic coating. Finally, an annealing treatment at 1100 °C was applied for 2 h.

Repair experiments have been performed on two types of industrial TBC coatings. The first one is a lamellar YpSZ TBC deposited by APS (Air Plasma Spraying) on Hastelloy X disks (25 mm in diameter), previously coated with an APS-deposited MCrAlY bond coat. Since the TBC lifetime of such systems is quite long, defects of different sizes and depths in the ceramic coating were created artificially by milling (Fig. 2a). The second type of coating to be repaired is a columnar YpSZ TBC deposited by EB-PVD (Electron Beam Physical Vapor Deposition) on a NiAl disk (25 mm in diameter, 2 mm in thickness). The specimen was heat-cycled at 1150 °C for 1500 h-cycles in order to create damaged areas. These cyclic oxidation tests were carried out in an open, horizontal furnace affixed to a motorized sample extraction system. Each thermal cycle consisted of 1 h in the furnace at 1150 °C followed by a 10 min cool down at 200 °C. After 1500 h-cycles, a delamination started at the edge of the coated disk and propagated mainly along the interface between the Thermally Grown Oxide (TGO) and the ceramic EB-PVD coating leading to some spallation of the TBC (Fig. 3a). In both cases, half of the sample was masked with an aluminum foil. After deposition of the repaired TBC, the mask was removed and an annealing treatment at 1100 °C was applied for 2 h. The repair process

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