Functionally Graded Thermal Sprayed Coatings

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The manufactured industrial pieces have often the external surfaces being in contact with harsh environment. The turbine blades are submitted to hot gas, the implanted prostheses to body liquids, etc. The protection of these surfaces can be realized using films and coatings. The latters have an important function of rendering the life in service of industrial piece longer, belong however, generally, to another group of materials with very different properties than the piece itself. For example, ceramic coatings are applied frequently on metal and alloys and some intermediate layers should be added between substrate and top coating. This is the concept of "functionally graded coatings" reviewed for the technology of thermal spraying in present entry basing onto paper *Appl. Sci. 2020, 10, 5153; doi:10.3390/app10155153*. The excerpt of this paper shows the chapters related to the applications of functionally graded coatings and their perspectives of development together with selected cited references.

Keywords: thermal spray coatings ; functional graded coatings ; application of thermal spray coatings

1. Major Applications of Functionally Graded Coatings

The functionally graded coatings (FGCs) are used as e.g. thermal barrier, biomedical and photocatalytic coatings, and coatings in printing industry. These applications are described in the present section.

1.1 Thermal Barrier Coatings

Thermal barrier coatings (TBCs) are the coatings designed to protect the gas turbine parts against intensive heat flux and various eroding and corroding particles contained in air and fuel [1][2]. The frequent TBC's configuration includes ceramic top coat (TC) and metallic bond coat (BC). Each layer, shown in Figure 1, has different role to play in protecting the gas turbine components. Two goals to reach by TBC are: (i) extension of the turbine lifetime in service; and (ii) increasing the operating temperature and the thermodynamic efficiency.

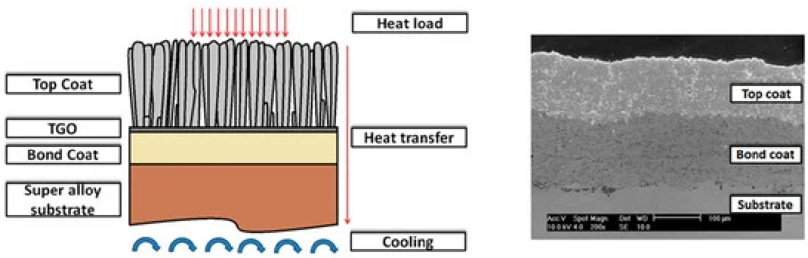

Figure 1. The schematic representation of TBC structure (**left**) and as-sprayed one (**right**). Reproduced with permission from [Advances in Materials Science Research], Nova, 2016.

The TC is exposed directly to the thermal, thermomechanical, mechanical, and chemical loads. The role of this coating is to protect the internal ones. The coating is ceramic, being usually partly stabilized zirconia. The BC is composed mainly of MCrAlY alloy (M is metal as e.g., Ni, Co, Fe). Its role is the reduction of the coefficient of thermal expansions (CTE) mismatch between TC and substrate. Another role is to protect the super alloy substrate against oxidation at high temperature. The BC contains aluminum, which diffuses forming a protective α-Al_2O_3 which is called thermally grown oxide layer (TGO). The TGO growth results in a generation of stresses at the BC/TC interface. These stresses reduce the time of TBC service [3][4].TBC is a very well-known FGC system. The initial studies on the functionally graded thermally sprayed TBCs were performed more in the 1970s [5][6]. During the last years, because of the progress in spray technology and feedstock materials, two advanced functionally graded TBCs concepts were developed:

- ***First concept*** consists of gradual change from fracture-resistant BC layer to heat-resistant TC layer by initial mixing the feedstock powders or by injecting feedstock by different injectors. It resulted in a significant decrease of residual stresses and in improvement of adhesion between subsequent layers leading to the long-term performance of TBC.

The concept could be realized by mixing the zirconia and MCrAlY alloy powders and then spraying the mixtures [7][8]. The authors found out that some interlamellar discontinuities were formed in the final structure because of different properties of the mixed feedstock. Other authors used the mechanically pre-alloyed MCrAlY and 8YSZ powders with different ratios. The atmospheric plasma spraying (APS) of powders prepared in that way enabled high deposition rate, uniform coating density, and chemical homogeneity to be achieved [9][10]. The use of two injectors for 8YSZ and MCrAlY powders separately was another idea [11][12]. The coatings sprayed in this way were almost crack-free and their chemical composition changed gradually across the thickness without a distinct interface between layers. A new idea is the deposition of functionally graded TBCs using spraying of hybrid powder-suspension feedstock. The authors report about gradual change of TBC microstructure from dense bond coat to columnar top coat. The TBC showed high resistance against oxidation at high-temperature test and high resistance against thermal shocks.

- ***Second concept*** consists of gradual change of chemical composition in TC. At least two different ceramics are used as layers. The outer one made of ceramics with high phase stability and low thermal conductivity, like e.g., alumina. The bottom ceramic layer should have high toughness, high fatigue performance, and low thermal conductivity. This specification corresponds to e.g., yttria stabilized zirconia such as 8YSZ [12][13].

There are several works focused on multi-ceramic TC in which YSZ is used as the bottom layer. The studies concerned:

- APS and SPS deposited 8YSZ/$La_2Zr_2O_7$ coatings [14];
- APS deposited 8YSZ/$Gd_2Zr_2O_7$ coatings ;
- APS deposited $La_2Ce_2O_7$/8YSZ [15];
- APS deposited $LaMgAl_{11}O_{19}$/8YSZ bilayers [16].

The studies aimed at prolonging thermal cycling lifetime comparing to the conventional two-layer TBC. The goal was reached owing to the increase of phase stability and of resistance against sintering. A small modification was proposed by applying yttria- and ceria-stabilized zirconia (CYSZ) as bottom layer instead of yttria-stabilized zirconia (YSZ) [17][18]. The obtained FGC showed low thermal conductivity and satisfactory thermal shock resistance.

Finally, an interesting idea of depositing the YSZ layers having various microstructures was presented [19]. The bottom layer was dense to improve bonding and the external one was porous to decrease thermal conductivity.

1.2 Biomedical

Millions of different types of prostheses (of hip for example) are produced annually in the world; different biomaterials and various methods of manufacturing in production.The major group of biomaterials include: (i) bioinert as titanium and its alloys; and (ii) bioactive as hydroxyapatite (HA) and bioglasses. The bioactive materials dissolve in human body, which accelerates the processes of prosthesis implantation in a bone [20]. The production of hip's and knee's prostheses, dental implants, and repairing of the bones are the major fields of activity [21]. Initial studies dedicated to application of FGC in biomedical applications were made by group of Khor [22][23][24]. The investigations aimed in obtaining well adhering and bioresorbable coatings. The use of TiO_2 as intermediate coating enabled to reach the tensile adhesion strength of the HA-TiO_2 system as high as 50 MPa [25]. Moreover, the use to titania bond coat increased the coating durability and improved fatigue resistance. Moreover, TiO_2 was a barrier for metallic ions migrating from metallic substrate to human body [26][27]. Another idea was APS deposition of titanium coatings with gradient of porosity [28]. The coating being in contact with substrate was dense; the middle one included micro- and macro-porosity and outer one was very porous to promote tissue growing. The authors [29] proposed the multi-coatings including HA-ZrO_2-Ti coatings by atmospheric plasma spraying (APS). The coatings with internal Ti coatings had reportedly satisfactory mechanical properties .An important drawback of HA is its thermal decomposition at temperatures slightly below its melting point (depending of partial pressure of steam). This was the motivation to develop functionally graded structures, multi-coatings, hybrid organic-inorganic composites by using the oxides $CaO–P_2O_5–TiO_2–ZrO_2$ [30][31][32]. An important issue in FGC technology is optimizing the process parameters. The researchers often use the *response surface methodology*. This approach was proposed to design the graded HA coatings which should be [33]: (i) stable for long time; and (ii) bioactive. The intensive studies on FGC composed with HA, Ti, and TiO_2 used statistical methods to improve coating properties [34][35].The frequently used thermal spray technique to manufacture biomedical coatings is APS. Other techniques were also tested. For example, Henao et al. used high velocity oxy- fuel (HVOF) to spray HA/TiO_2-graded coatings onto the Ti6Al4V substrate reaching satisfactory behavior at body simulated fluid (SBF) testing. The use of FGC of HA and bioglasses was favorably compared with the conventional plasma-sprayed HA [36]. The improvement of mechanical properties of coatings was achieved by the use of the composites of HA or bioglasses by nano-diamond or reduced graphene oxide [37][38].The suspension plasma spraying (SPS) technology to spray FGC of HA and TiO_2 was used by the authors of studies [39]. They compared two configurations of coating: duplex and gradient one. These oxides were SPS deposited with the use of peristaltic pumps to inject the suspension resulting in relatively thin coatings [40].The functionally graded coatings composed of bioglass and HA were produced by SPS. The studies aimed at combining rapid osseointegration of bioglass with long term stability of HA. The obtained coatings were immersed in SBF and exhibited strong reactivity with the medium. The authors produced all coatings by exclusively SPS technology or, by using APS technology to spray HA and SPS one to spray bioglass.The suspension was also used as a feedstock in high velocity oxy-fuel spraying. The authors manufactured multilayer HA/TiO_2 coatings and improved the mechanical properties of deposits such as adhesion and wear resistance with regard to pure HA coating.The high quality FGC biomedical coatings have an important potential in medical applications. The group of biomaterials for coatings, namely hydroxyapatite and bioglasses exhibit good in vitro as well as in vivo biocompatibility. The FGCs in biomedical applications allow improving osseointegration and reducing the shear stresses occurring at the bone–implant interface. The long-term stability of the FGCs and the stability of their biocompatibility still remain to be improved in future. The statistical methods may help to understand better the phenomena occurring in contact of coatings with the human body.

1.3 Photo-Catalysis

Photo-catalysis is an important issue in the chemical industry being related to degradation and destruction of organic pollutants. Fujishima and Honda [41] were the first to describe this phenomenon. More details are presented in the studies [42][43]. Thermal spraying technology works on three semiconducting oxides, namely TiO_2, ZnO, and SnO_2. TiO_2 seems to be most frequently tested. An important point is the fact that at the photoreaction the surface of oxide remains unchanged [44]. The possible applications of the photocatalytic thermal spray coatings is the self-cleaning of surfaces (e.g., glass building). The photo-catalysis was also tested for water and air purification, for anti-fogging surfaces, for photo-catalytic lithography, and for many other applications [45][46]. An example of composite photo-catalytic coating used against air pollution is TiO_2 doped with Fe_3O_4 coating sprayed on mild steel substrates [47][48]. Because of the presence of intermediate phases, such as $FeTiO_3$, the band gap is narrower, than in pure TiO_2 which resulted in better photocatalytic activity with reported efficiency of more than 90%. Inversely, the nanostructured TiO_2/Fe_3O_4 plasma sprayed coatings did not exhibit satisfactory photocatalytic properties. Their best photocatalytic efficiency was as low as 23%. Chen et al. [49] sprayed the coatings of TiO_2 doped with ZnO or CeO_2 or SnO_2 on the foamed aluminum substrate and used them for benzene degradation. The coatings doped with CeO_2 and SnO_2 degraded better than that doped with ZnO. Nevertheless, their efficiency was greater than 90%. Ctibor et al. [50] sprayed titania doped with iron coatings. The coatings were used for butane decomposition under visible and UV radiation. The photocatalytic activity was also observed in plasma-sprayed TiO_2 + ZnO.Fe_2O_3 coatings [51]. The photocatalytic efficiency was promoted by $FeTiO_3$ phase being present in the coatings. On the other hand, the large amount of $ZnFe_3O_3$ phase was not favorable for the photoactivity. The innovative plasma-sprayed composite of TiO_2 with carbon nanotubes had photo-catalytic activity greater than pure TiO_2 deposits [52]. Robotti et al. [53] deposited TiO_2 with ECTFE polymer composite coating by low pressure cold spraying (LPCS) method. The coating was used to degrade of NO and NO_2 pollutants. The photocatalytic activity of obtained coatings were much better than that of commercial paint.The group of Hua Li from Ningbo in China [54][55], manufactured nanocomposite coatings of TiO_2/HA and TiO_2/HA/reduced graphene oxide obtained by flame spraying and tested for water disinfection and air purification.

1.4 Applications in Printing Industry (Corona and Anilox Rolls)

The APS method is widely used to spray coatings in the printing (*anilox rolls*) and packaging (*corona rolls*) industries. *Anilox rolls* are used to transfer ink to paper at printing. The ceramic coatings sprayed with the APS method replaced galvanic layers used previously. The specifications of coatings in such rolls are hard to reach. Namely, the surfaces of coatings must be smooth, free from defects, resistant to abrasion and corrosion, with high wettability [56]. The rolls are made of stainless steel. The metallic bond coat is sprayed on the substrate followed by a ceramic, mainly Cr_2O_3, top coat. The as-sprayed coatings are ground and polished. The laser engraving of small cells follows. The last stage is the final polishing. There is a tendency to increase the density of cells on the surface. The rolls with the surface texturing after spraying are also sometimes used [57]. The rolls are important and increasing part of thermal spray market [58]. It should be stressed up, that *anilox rolls* can be produced with other than Cr_2O_3 oxides such as e.g., Al_2O_3 + TiO_2 alloys [59]. On the other hand, the Cr_2O_3 coatings can be produced with use of HVOF spraying [60]. *Corona rolls* are used to increase the adhesive capacity of printing ink to the polyethylene surface by the use of plasma generated at corona discharge. The rolls used in the process were coated by Al_2O_3 sprayed using the APS method. The amount of metal (the inclusions of metals in sprayed coatings are mainly the droplets of W or Cu from the spray torch electrodes.) in sprayed alumina must be as small as possible. Therefore, it is very important to optimize parameters and to control the electrodes of the torch.

The coatings must have good dielectric properties and being thick enough to have high breakdown voltage [61]. This requires spraying of thick alumina coating. The APS-sprayed alumina coating on *corona rolls* can be sealed after deposition [62]. The rolls are an important part of the plasma spray installations market [63].

1.5 Other Applications

A rapid development of industrial technologies in the industrial sectors of aerospace, automobile, shipbuilding, etc., resulted in many applications of non-ferrous metals. Consequently, the joining of such metals became an important issue. The conventional welding processes are not adapted for joining the non-ferrous metals. A possible solution is an application of an additional interlayer. The popular techniques of interlayers deposition are hot dipping and galvanizing [64][65][66]. An interesting alternative is cold spraying technique. Winnicki et al. [67] used low pressure cold spraying (LPGS) to deposit composite coatings of Al + Al_2O_3, Al + Ni + Al_2O_3 and Ni + Al_2O_3. The authors found that the microstructure of Ni + Al_2O_3 interlayer after the resistance spot welding (RSW) remains unchanged (Figure 2) and that the shear strength of the joint was comparable to that of joint of Al + Al. Another opportunity to use the cold spray technology was a deposition of interlayers on non-metallic substrates made by Wojdat et al. [68]. The authors used metallic (Al and Cu) and metal matrix composite (MMC) (Al + Al_2O_3 and Cu + Al_2O_3) interlayers in soldering and concluded that the interlayers deposited by LCPS effectively limited formation of the reaction zones at the interface of interlayer with the soldered joint. Consequently, the mechanical properties of such joint could were improved. Li et al. [69] used cold spraying to obtain Sn-Cu coatings onto aluminum and on copper substrates. The results showed the improvement in soldering. The deposition of graphite-copper composite coatings onto aluminum alloy 6060 with the use of LPCS were analyzed in [70]. The authors added aluminum, aluminum with alumina and copper to exclude erosion of the graphite at cold spraying of composite coating.

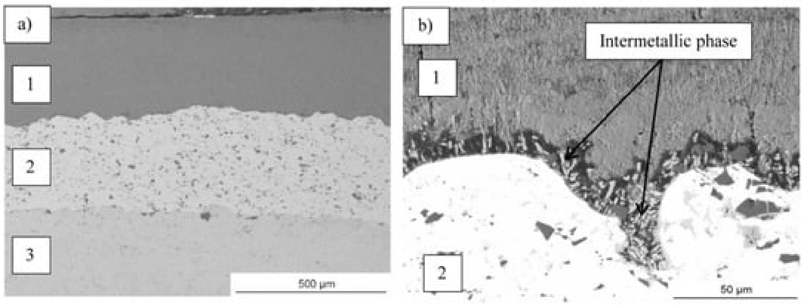

Figure 2. SEM-BSE micrographs of RSW aluminum—steel joint with Ni + Al_2O_3 interlayer (**a**,**b**). 1—aluminum alloy, 2—Ni + Al_2O_3 interlayer, 3—steel substrate [67].

Metal matrix composites (MMC) coatings are used frequently to improve resistance against erosion and wear. Most of thermal spraying processes are done at high temperature and, consequently, are associated with the phase transformation, oxidation and decarburization of ceramic reinforcement or soft metal matrix. Therefore, a low temperature process of cold spraying can be useful. The drawback of cold sprayed MMCs is low strength and ductility. The post-spray heat treatment process may help in eliminating these negative effects. Consequently, the post-spray friction stir processing (FSP) of MMC coatings deposited by cold spray was tested [71][72][73][74][75][76]. Such post-treatment resulted in reduction of interparticles distance and in refinement of the reinforcing particles. Peat et al. [75] showed that FSP post-spray treated cold sprayed Al + Al_2O_3 composite coating had satisfactory erosion resistance.

2. Perspectives of Development of Functionally Graded Coatings

2.1 Polymers as Substrates

Polymer are attractive in many applications because of their small density and small CTE [77]. However, the exposure to harsh environment such as ultraviolet (UV) radiation, moisture, or high temperature deteriorates their properties. Consequently, some coatings protecting their surface were useful [78]. Thermoplastic polymers are easier to be coated than thermosetting polymers because of their thermal softening [79][80][81]. However, particularly interesting for industrial applications are polymer matrix composite (PMC) such as e.g., such with the polyimide resin as a matrix. Such composite can tolerate long-term service temperature up to 400 °C and may be useful for the components in aerospace [82]. Thermally sprayed coatings on polymers may increase their service temperature and improve their thermal shock resistance [83]. Presently, the low melting point metals as zinc or aluminum are used as bond coat adhering to polymer surface and ceramics are used as top coats for the TBCs on the composite substrate [84]. The failure of such TBC's occurs as vertical cracks through the coating followed by its delamination [85]. To reduce the residual stresses between polymer substrate and ceramic coating the use of ceramic TC having the CTE close to that of the polymer substrate would promote thermal shock resistance [86]. The group around Ivosevic [87][88][89] initiated the studies of FGC on PMC substrates. They used HVOF sprayed graded polyamide/WC-Co coatings to improve adhesion between carbon reinforced PMC substrate and WC-Co top coatings. The bond strength of bond coat was relatively low with the value of 8 MPa. Similar value had the bond strength of coatings arc sprayed with cored wire consisting of low carbon steel skin and Ni–Cr–B–Si filler material on the substrate being graphite fiber reinforced thermo-setting polyimide [90]. Some investigations of sprayed FGC on polymer matrix composites were focused on modification of top layer of composite with metal-polymer mixture. Cui et al. [91] used epoxy resin filled with aluminum powder interlayer to deposit aluminum coating with detonation gun on PMC. The bond strength of Al top coat was about 8.6 MPa. Rezzoug et al. [92] sprayed different coatings including e.g., pure epoxy resin layer and its composites with copper, stainless steel and aluminum and reached adhesion varying between 2.7 and 6.5 MPa.

2.2 New Applications of FGC

The number of applications of functionally graded coatings is still growing. In this section only a few examples are briefly described. The development of new materials and new deposition methods enables predicting many new implementation of FGC systems.

Presently, the bond coats of TBC are usually deposited using expensive vacuum plasma spraying (VPS). This spray technique can be replaced by cold spraying and by HVOF spraying [93][94][95]. The emerging technology on this field is cold spraying. Karaoglanli and Turk [94] tested cold spraying bond coats in the TBC for isothermal oxidation behavior. The authors confirmed the usefulness of cold spraying for bond coat manufacturing. Khanna and Rathod [95] found that bond coat manufactured by cold spraying has satisfactory tribological properties and high oxidation resistance in high temperatures. Go et al. [96] presented cold sprayed coatings using Cr_2AlC powder for TBC bond coat. Another example of bond coat deposition by cold spraying was a FGC system deposited onto polymer substrate for biomedical applications [97]. The authors describe the bond coat of titanium produced on the biocompatible PEEK substrate. The nanostructured TiO_2 crystallized, as anatase was a top coat. The new idea of metal-doped HA thermally sprayed coatings was studied to improve antibacterial resistance of bone prostheses, dental implants, and macroporous scaffolds [98]. Sergi et al. produced HA doped with Zn coatings by solution precursor plasma spraying (SPPS) method. The tests carried out revealed the coatings bioactivity and their efficiency against some bacteria. The enhanced antibacterial properties of plasma sprayed HA coatings doped with Sr and Zn showed the reduction of cytotoxic effect of Zn^{2+} ions by addition of Sr^{2+} as observed Ullah et al. [99]. Copper can be a useful dopant to the HA improving bactericidal properties sprayed coatings. The characterization of Cu- and Ag-doped HA coatings obtained by APS was carried out by Lyasnikova et al. [100]. The HA with Cu cermet obtained by SPPS technique was characterized by Unabia et al. [101]. The improvement of biomedical properties of plasma sprayed HA coatings doped with Mg or with Mg and Sr were found in [102][103]. Another popular dopant of HA coatings is silver. The Ag addition to HA coatings improves bactericidal capacities and decreases the risk of infections after surgery. This type of biomedical coatings were obtained by many thermal spray methods such as vacuum plasma spraying, flame spraying, radio frequency plasma spraying, and suspension plasma spraying [104][105][106][107][108]. The new generation of biomaterials uses magnesium alloys as a substrate on which HA coatings doped with niobium were deposited [109].

To finish, let us have a look on an interesting application of high-pressure cold sprayed alumina composites with aluminum. The coatings were characterized by low thermal conductivity, low solar radiation absorption, comparatively high infrared emittance, and oxidation stability which rendered them useful for application in outer space [110].

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