



Comparative study of the corrosion resistance of thermally sprayed ceramic coatings and their bulk ceramic counterparts

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Abstract

A comparative study of the corrosion properties of thermally sprayed ceramic coatings (Al_2O_3 , Al_2O_3 – TiO_2 with different ratios, mullite, and ZrSiO_4) and their sintered bulk ceramic counterparts was performed. The coatings were deposited on corrosion-resistant steel substrates using atmospheric plasma spraying (APS) and high velocity oxy-fuel (HVOF) spraying processes. The corrosion properties were investigated in 1 N solutions of NaOH and H_2SO_4 at 85 °C, respectively. The coating microstructures and phase compositions, as well as the corrosive environment were shown to have a strong effect on the corrosion resistance of the coatings. Al_2O_3 –coatings were more sensitive to these factors than Al_2O_3 – TiO_2 coatings were.

The corrosion resistance of the bulk ceramics was superior to that of the thermally sprayed coatings. This is mainly because the coatings exhibited specific microstructure and contained amorphous and/or metastable phases not appearing in the bulk ceramics.

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1. Introduction

Thermal spray processes represent an important and rapidly growing group of surface modification technologies. Besides metals and hardmetals, ceramics are the most important materials used in the preparation of thermally sprayed coating solutions.¹ Atmospheric plasma spraying (APS) has traditionally been the most common spray process for preparation of coatings from high melting temperature ceramic materials. High velocity oxy-fuel (HVOF) spraying, another widely used thermal spray process, is characterized by significantly lower process temperatures but by higher particle velocities, thus resulting in higher kinetic energies. Coatings prepared by HVOF show denser microstructures, lower porosity, higher bond strength, and lower roughness.¹

Thermal spray technologies allow large components to be protected from corrosion at reasonable costs using oxide materials. Normally, when the corrosion resistance of thermally sprayed coatings is discussed, it refers to the ability of the coatings to protect the substrates from corrosion.² As a rule, sealing technologies are applied to oxide coatings used under such conditions. Up to now only a very limited amount of information concerning the corrosion properties of oxide coatings themselves is available. Comparison of the behavior of the coatings and the corresponding bulk ceramics is very important to yield an understanding of the respective advantages and disadvantages. Consequently, the choice of appropriate technologies and materials as well as improvement of coating characteristics (i.e., microstructure, phase compositions) can be made for a specific application. Due to their denser microstructures, a higher corrosion resistance can be expected for HVOF-sprayed coatings than for APS-sprayed coatings.

Al_2O_3 plays an important role both as a bulk ceramic and as a thermally sprayed coating. Bulk Al_2O_3 ceramics components are widely used in industrial applications, in particular due to their good mechanical, wear, and corrosion resistance. A major difference between sintered bulk Al_2O_3 ceramics and

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thermally sprayed Al_2O_3 coatings is that sprayed coatings are composed of different thermodynamically unstable forms of Al_2O_3 (e.g., γ - and δ - Al_2O_3) even though all commonly used feedstock powders are composed of stable α - Al_2O_3 .¹ This rather uncommon material behavior has been known for a long time and was described in detail by McPherson.^{3,4} Thermally sprayed coatings are currently produced with these phase transformations being either ignored or accepted.¹ Direct comparison between bulk ceramics and coatings is difficult to perform, since bulk materials consisting of the metastable forms do not exist. However, significant differences, e.g., regarding the long-term stability in humid environments, are empirically well known. Several authors^{5–7} affirmed that the presence of the γ -phase combined with porosity and substrate characteristics drastically changes the corrosion properties of the coating compared with α -alumina. Corrosion resistance of the coatings can be changed by different post-spray treatments,^{8–11} but all of them are associated with additional costs and are technologically unfavorable.

In addition, not only the microstructures and phase compositions strongly influence the corrosion properties of ceramic materials, but also the impurities have to be taken into account. In the case of bulk ceramics, it is known that even a low content of impurities¹² or other oxides (i.e., ZrO_2)¹³ significantly modifies the corrosion behavior.

Additions of 3, 13, and 40% TiO_2 to alumina are frequently used in thermal spraying.^{1,2,14–16} Through the formation of a eutectic phase, the TiO_2 addition to Al_2O_3 involves the formation of a liquid phase at a significantly lower temperature. The amount of liquid phase depends on the TiO_2 content (see phase diagram¹⁷). At a content of 44% TiO_2 , which corresponds to a mole ratio Al_2O_3 : TiO_2 of 1:1, the thermally instable compound Al_2TiO_5 (aluminum titanate, mineralogical name tialite) is formed. The process conditions, in particular heating and cooling rates, determine whether or not Al_2TiO_5 can be found in feedstock powders and coatings. A new phase, $\text{Al}_6\text{Ti}_2\text{O}_{13}$, that exists only in a small temperature range above 1400 °C has recently been described for the Al_2O_3 – TiO_2 system.^{18,19} Vargas et al.²⁰ mentioned the existence of this phase in feedstock powders and sprayed coatings. Another phase ($\text{Al}_2\text{Ti}_7\text{O}_{15}$ with ordered cations in M_3O_5 structure) and replacement of Al by Ti in Al_2TiO_5 was described by Monnereau et al.²¹

The phase composition of the coatings is normally not in equilibrium and therefore consists of a mixture of different phases of Al_2O_3 , titanium suboxides TiO_x (in an ordered state: $\text{Ti}_n\text{O}_{2n-1}$), and Al_2TiO_5 .²² This fact is frequently ignored when properties are evaluated, and generally, only Al_2O_3 – TiO_2 coatings are spoken of. It was affirmed that the additions of TiO_2 to Al_2O_3 ^{16,23,24} or use of Al_2TiO_5 ²⁵ decreases corrosive attack by dilute acids.

Sintered mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) ceramics are known for their corrosion resistance in solutions, melts, slags, and gases.²⁶ According to investigations performed by Lee et al.,²⁷ thermally sprayed mullite coatings have a high amorphous phase content, with the amorphous phase crystallizing after holding of the coatings at high temperatures. The mullite coatings are suitable for preparation of so-called environmental barrier coatings.^{28,29}

They have been shown to exhibit a high resistance to corrosion by molten salts and by water vapor at high temperatures and pressures.^{30,31}

Like mullite, zirconium silicate (ZrSiO_4) as alternative ceramic material shows interesting corrosion resistance but it was little studied for the preparation of thermally sprayed coatings.

This paper presents a comparison of the corrosion stability of APS- and HVOF-sprayed ceramic coatings Al_2O_3 , Al_2O_3 – TiO_2 with different ratios, mullite, and ZrSiO_4 and their sintered bulk ceramic counterparts in different corrosive conditions. Information concerning the corrosion stability of the coatings at different temperatures and under hydrothermal conditions is given elsewhere.^{2,16} Knowledge of the feedstock materials behavior during the spray process and investigations of the corrosion properties of the coatings can be used in the selection of appropriate thermal spray ceramic coating and bulk ceramic materials and processes for application in corrosive media.

2. Experimental

2.1. Preparation of the coatings

Commercial fused and crushed powders Al_2O_3 (99.7% purity), and Al_2O_3 –3% TiO_2 , Al_2O_3 –13% TiO_2 , and Al_2O_3 –40% TiO_2 (Ceram GmbH, Albrück-Birndorf, Germany) with appropriate particle size distributions for both APS (–40 + 10 μm) and HVOF spraying (–25 + 5 μm) were used for coating preparation. Commercial fused and crushed mullite (Al_2O_3 –30% SiO_2) and zirconium silicate (ZrO_2 –33% SiO_2) powders (Ceram GmbH, Albrück-Birndorf, Germany) with particle size ranges of –40 + 20 μm were used to produce APS coatings. A summary of the composition and the crystalline phases of the feedstock powders are given in Table 1.

APS coatings were sprayed with an F6 plasma gun (GTV mbH, Luckenbach, Germany) using an Ar/ H_2 plasma gas mixture. HVOF coatings were sprayed with a TopGun system (GTV mbH) using ethylene as the fuel gas. The spray parameters were optimized in order to produce low-porosity coatings. Details of the spray parameters can be found elsewhere.^{2,16} Coatings were deposited on round substrates 25 mm in diameter made of corrosion-resistant stainless steel 1.4462 (X2CrNiMoN 22-5-3). All of the substrates were thoroughly cleaned, degreased, and grit-blasted with corundum prior to coating. The coating thickness was about 200–250 μm . All specimens were used in the as-sprayed condition, i.e., without any further machining or sealing, for the corrosion tests.

2.2. Preparation and properties of the bulk ceramics

For preparation of the bulk ceramic materials, the spray powders (except Al_2O_3 –3% TiO_2) were hot-pressed in Ar (HP25; FCT Systeme GmbH) with varying temperatures, holding times, and pressures to form disks of diameter 80 mm and thickness 5 mm. Density was determined using Archimedes' principle.

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