Dielectric properties through-thickness mapping on extremely thick plasma sprayed TiO2

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Plasma spraying of self-supporting plates (SSP), tubes and variously shaped ceramic parts is performed by WSP process for a long time. First results were published nearly 20 years ago, when Al2O3, ZrO2, mullite and zircon ZrSiO4 were tested as the first materials. Later, various other materials as garnets, basalt, forsterite or diopside were tested. TiO2 was applied for thick wall tubes production as well as for thinner coatings for photocatalytic tests. This material is relatively insensitive to spray torch parameters, and when sprayed using a natural rutile feedstock, is it also inexpensive. The difference in thermal expansion between substrate and coating materials is most typically used for thick self-supporting plates production.

Rutile TiO2 received as a natural mineral was sieved to obtain feedstock powder for spraying (size 100-170 μm”). Plates were sprayed using the water-stabilized plasma spray system WSP 500 (IPP ASCR, Prague, Czech Republic). For the described experiments the voltage 320 V and current 480 A were set. The powder was fed in through two injectors and thickness of the produced coatings was as high as 15 mm. Massive aluminum blocks were used as substrates. To restore the stoichiometry, thermal post-treatment (annealing) was applied. The samples were heated in a laboratory furnace in the air atmosphere with heating and cooling rates 6 °C/min and with the dwell time of 3 hours on the maximum temperature 1100 °C. Such a temperature is high enough for a stoichiometry restoration and also for substantial changes in the microstructure, and in dielectric properties. Annealing changed the character of porosity. After annealing the porosity is higher but the mean size of pores is smaller. Correspondingly, the number of pores per square millimeter is higher. The pores are less flat and more globular as the annealed microstructure is approaching a sintered microstructure.

The extremely high coating thickness enables us to cut the SSP into three parallel “horizontal” slices (i.e. cuts were perpendicular to the spray direction): one was close the substrate, second one in the middle and the third one close to the surface. This sectioning was performed on as-sprayed deposit as well as on annealed one. Usually the low coating thickness does not allow such a slicing. The layers close to the surface of the annealed SSP exhibited dielectric relaxation at frequency 100 kHz while the central slice and the layers close to the substrate exhibited tendency to relax at frequencies over 1 MHz. Typical relative permittivity of the annealed material was around 150 and loss tangent around 0,05. The volume resistivity in the order 109 Ωm and the surface resistance of the individual slices in the order 1012 Ω were measured. Before the annealing the volume resistivity was in order of only 1 Ωm. This conductive character of the sample was due to the plasma-induced reduction of the TiO2 ceramics into TiO2-x, as reported by the authors in the past. For the restoration of stoichiometry and giving the dielectric character to the material, the thermal annealing is necessary.