

2.45 GHz Microwave Plasma Source μ SLAN



Abb. 1: μ SLAN

Plasma CVD of thin Al_2O_3 films on powders in a circulating fluidized bed

International Conference on Metallurgical Coatings and Thin Films - ICMCTF 2000

April 10 – 14, 2000 USA

The work described below was performed using a PlasmaConsult μ SLAN microwave plasma source.

Paper A 4-5

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Easy handling and high surface area brought powders into a dominating position among industrial goods. Bulk properties and surface properties can be controlled independently by coating the particles with a thin film. Aluminum oxide films are known to have excellent

properties as diffusion barriers, even at high temperatures, and protect particles during high-temperature applications such as thermal spray coating. Among the various film deposition techniques, plasma CVD is particularly promising, since low-temperature plasmas provide a powerful source of reactive species at relatively low temperatures. Such plasma processes have mainly been applied to flat surfaces so far, whereas no satisfactory process is currently available for the coating of powders.

We have recently shown that circulating fluidized bed, operating under vacuum conditions, can be used to expose the powder to plasma in a very efficient way¹. The coating reactor is a vertical tube (40 mm I.D., i.e. a μ SLAN !), where the particles are blown through by the reaction gas. The plasma is generated in situ in the upper part of this riser tube. The entrained particles are separated from the gas phase in a cyclone, recirculated to the reactor and enter a new deposition cycle. 1 kg of powder can be coated per batch. Circulating the powder enables efficient heat dissipation out of the plasma zone and improves the uniformity of the coating.

SiC particles of 150 μm diameter were coated with about 1 μm aluminum oxide films using aluminum sec.-butylate as precursor and an argon/oxygen-mixture as process gas. The aluminum precursor was mixed with sec.-butanol for flow ability enhancement and subsequently vaporized in a heated injection nozzle. Coating time was 3 – 5 hours. Chemical surface analysis of the coated SiC powder was performed using X-ray photoelectron spectroscopy (XPS). The diffusion barrier effect was investigated with a high temperature treatment of the coated powder in oxygen. It could be shown by electron microscopy that the coatings are dense and uniform. Variations of film thickness were small and corresponded to the calculated variation of residence time in the reactor. The deposited material consisted mainly of Al_2O_3 , and low carbon impurity levels were reached. High-temperature oxidation of the SiC bulk material was reduced by an order of magnitude. These results demonstrate that uniform, dense, thin coatings can be obtained by PECVD and circulating fluidized bed technology.

- 1) M. Karches, C. Bayer, Ph. R. von Rohr, in:
Circulating Fluidized Bed Technology VI
(Ed.: J. Werther) p. 537, DECHEMA,
Frankfurt a. M. 1999

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