POWDER DENSIFICATION IN A TRANSFERRED ARC REACTOR

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Résumé

Pour améliorer la projection plasma de particules poreuses de zircone produites par atomisation-séchage, les poudres subissent un pré-traitement de densification avant projection. Cette densification doit permettre d'augmenter la tenue mécanique des particules et améliorer leur fusion lors de la réalisation des dépôts. Le pré-traitement de la poudre est réalisé dans un réacteur plasma à arc transféré. Avant et après traitement, les poudres sont caractérisées par leur morphologie, leur répartition granulométrique, leur densité, leur composition chimique et leur résistance mécanique. En jouant essentiellement sur le débit et la nature du gaz plasmagène et sur le débit du gaz porteur de poudre, des particules de forme sphérique avec une densité proche de la densité théorique et une faible teneur en phase monoclinique ont été obtenues.

Abstract

To improve the plasma spraying of porous zirconia particles prepared by spray drying, a pre-treatment of powder has been carried out in a transferred arc plasma. The densification of the agglomerated particles must raise the resistance of agglomerated particles against mechanical and thermal loads during plasma spraying. Before and after treatment, powders are characterized with regard to morphology, grain size distribution, density, phase composition and mechanical resistance. With the plasma transferred arc reactor, particles with spherical shape, nearly theoretical density and low content of monoclinic phase are produced provided that the particles don't receive a too high thermal flux.

1 Introduction

Recent works /1, 2/ have shown that the properties of coatings produced by plasma spraying depend on the way the powders are produced. Among production methods, the micropelletization of primary products by spray drying offers some advantages as the wide range of particle size and particle porosity that is possible to obtain, or the possible combination of a lot of different components /3/.

However these agglomerated particles may be desintegrated in the plasma jet because their mechanical strength is not sufficient. Figure 1 /4/ for example, shows the evolution of the velocity of zirconia agglomerated particles along their mean trajectory. As it can be seen, the treatment of the powder within the plasma jet is characterized by two particle velocity distributions ; one is representative of the micropellets which have penetrated into the plasma core and the other of the small particles obtained by the desintegration of the micropellets either during the pneumatic transport or at the injection port ; these small particles are afterwards sucked in the plasma jet with the surrounding gas and reach the jet axis at about 45 mm from the nozzle exit.

To raise the resistance of these agglomerated particles against mechanical and thermal loads during plasma spraying the spray-dried powder may be densified.

The densification of micropellets by plasma treatment must produce dense and spherical particles, and reactions between the primary components may occur during particle heating and/or melting.

In this paper is presented a study carried out on powder densification by plasma treatment in a transferred arc reactor which is characterized by short residence times of the solid particles to be treated and high temperature gradients.

Figure 1: Agglomerated particle velocity evolution versus particle mean trajectory under plasma spraying conditions

The starting powder is agglomerated ZrO₂-MgO (76/24) spray dried and sintered at the Material Science Institute of Aachen (FRG) /3/. The grain size range is between -63+20 μm. Before plasma densification, the micropellets are thermally treated in an open furnace (at 1400°C during 2 h) to raise the particle strength and remove the binder (see figure 2).

Figure 2: Starting powder (spray dried and sintered)

2 Experimental

The transferred arc device is confined in a double wall water cooled chamber equipped with viewing ports for the arc control and the optical diagnostics. This controlled atmosphere chamber is 2 m long and 0.2 m in internal diameter. The plasma device is made up of a plasma torch and a rotating auxiliary anode. The axis of this water cooled anode tube (12 mm in diameter) is decentred with respect to the plasma jet axis in order to avoid hot particles sticking on the anode tube and the distance between the nozzle exit and the anode ranges between 40 to 100 mm. The configuration with a blown arc between the cathode and the nozzle of the torch at a potential lower than that of the rotating anode has been chosen (it allows to keep ionized the gap between the nozzle exit and the rotating anode and control easily the transferred arc current between a few tens and a few hundreds amperes). The powder is injected close to the torch nozzle exit with one injector and is collected by a tube 40 mm in internal diameter which can be moved up and down along the chamber axis. A schematic diagram of the experimental set up is given in figure 3.

Figure 3: Schematic diagram of transferred arc process

3 Characterization of the processed powders

The characterization of powders was carried out for starting and densified powders with regard to morphology, grain size distribution, density, phase composition, and mechanical resistance.

The morphology of the particles (surface and cross section) is examined by scanning electron microscope after embedding them in resin. Their shape and size distribution are determined by an image analyzing technique (the particle size and shape factor distributions are calculated taking into account the particle volume which is more representative of the powder mass). The phase composition of the powder is analyzed by XRD, and its density by picnometry with phtalate dibutyl as liquid. Before and after processing, the mechanical stability of the powder (or more precisely its tendency to break up) is assessed using an ultrasonic bath (the powder is put in acetone solution and exposed during 20 minutes to ultrasonic waves).

4 Results and discussion

Five sets of experiments were conducted with zirconia powders to determine the characteristics of the treated powders (shape, size distribution, density ...) under various operating conditions which are summarized in table 1.

Table 1 : summary of experimental conditions for the transferred arc reactor

Run	Trans. ferred arc		Blown arc		Plasma gas		Torch rotating anode distance	carrier gas
	Is A	Vs V	It A	Vt V	Ar slm	He slm	mm	slm
1	216		55		60	0	50	4
		33		107				
2	178		60		60	0	50	2.5
		30		104				
3	205		80		45	0	53	4
		38.8		112				
4	180		95		45	0	53	2.5
		31		100				
5	200		60		30	30	53	4
		46		112				
Powder mass flow rate : 15 g/min								

Runs 1 and 2 differ essentially from one another in powder carrier gas flow rate. The electric parameters and the plasma gas flow rate correspond to the most stable conditions as shown by preliminary experiments.

Run 3 is intended to get a better powder treatment by increasing the particle dwell time and the heat flux received by particles within the plasma flow.

Run 4 is essentially a repetition of run 3 with a lower carrier gas flow rate.

And in test 5, helium is added to argon plasma gas in order to increase the thermal conductivity of the mixture and so to get a more efficient treatment for powders.

For all the tests, about 20 % in weight of the powder is collected in the chamber outside the collecting tube (see figure 3). The analysis of this powder part shows that the particles are identical to the starting ones and have undergone no modification.

The particles collected in the tube have always an average diameter smaller than the one of the original ones (figure 4), that is explained partly by a desintegration of the micropellets within the plasma jet and partly by their densification.

Figure 4: Particle diameter and shape factor before and after treatment (X=mean value ; S=root mean square) for runs 1, 2 and 4 depicted in table 1

Runs 1 and 2 are characterized by a low treatment of the powder : only the smallest particles (diameter less than 20 μm) are fused ; the powder density is increased up to 4.5 against 4 for the spray dried starting powder and the proportion of monoclinic phase is still high as shown in table 2 giving the height of the principal peaks.

Table 2 : height of the principal peaks as measured by XRD analysis using a copper anode

Run	Cubic phase	Monoclinic phase	MgO
2 theta position	30.599	28.167 31.384	42.829
Starting powder	0	8.6 6	2.5
1	8.6	2.3 1.8	1.6
2	7.6	5.4 3.8	2.7
3	8.8	0.8 0.5	1.8
4	8.9	0.3 0.2	1
5	8.9	0.5 0.3	1.1

The powder carrier gas flow rate conditions the trajectories of the particles. When the flow rate used is 4 slm, the particles penetrate into the plasma jet and follow trajectories close by the jet axis. When it is 2.5 slm, they pass in periphery of the plasma flow. For the two first sets of experiments, the density and the morphology of the particles are nearly the same for both carrier gas flow rate ; the velocity of the particles passing through the plasma core is too high due to the plasma gas flow rate and in both cases the treatment is not efficient.

On the contrary for runs 3 and 5, the particles follow axial trajectories and receive a higher heat flux, so that the powder treatment is too high as revealed by powder analysis. All the particles are fused and some exhibit an internal porosity that may be explained by the blowing of the particle molten shell by the included gas. For these two tests, the powder density is lower than in the preceeding tests (figure 5).

The better results are obtained in test 4. Most of the particles are fully molten and have no porosity as seen in figure 6. In this case, the density measured by pycnometric technique is 5.5 instead of 6 for theoretical density (cubic phase), and the percentage of monoclinic is low (see table 2).

Figure 5: Density of the powder as measured by pycnometry method

— 41.6 μm x 240

Figure 6: Morphology of powder collected in run 4

With the working conditions, the particles pass in periphery of the plasma arc with a lower velocity than in cases 2 or 1, due to the lower flow rate of the plasma gas and of the carrier gas. Therefore they receive a more "gentle" thermal flux than in test 1 or 3 but sufficient to be molten.

5 Conclusion

The pre-treatment of zirconia powder before spraying in a transferred arc reactor has been carried out to improve mechanical properties of coatings sprayed with atomized powders. An example of plasma coating produced with powder obtained with run 2 (see table 1) which is characterized by a low gain in powder density, is shown in figure 7 and it can be seen that rather dense coatings are obtained.

However these tests have shown the necessity to modify the reactor to limit the mixing of the treated and untreated powders and also to classify the powder after treatment to eliminate the smallest particles (diameter < 10 μm). Similar experiments are in progress with a plasma heated fluidized bed which is known for its uniformity in temperature and its long powder residence time which can be easily controlled /5/

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Spray distance 150 mm

x 200

Figure 7: Cross section of a zirconia coating formed with powder densified by transferred arc process (spraying conditions : torch PTF4, I = 600 A, nozzle diameter 6 mm, Ar = 35 slm, H₂ = 12 slm, powder injector diameter = 1.6 mm) /6/

References

- /1/ MEYER-GRUNOW H., NICOLL A.R., Proceeding 1st Plasma Technik Symposium, Lucerne, 1, (1988), p 12
- /2/ INWOOD B.C., CHANDLER P.E., Thermal Spraying Conference, London, UK 4-9, proceedings published by the Welding Institute, 1, (june 1989), p 91
- /3/ E. LUGSCHEIDER, H. ESCHAUER, A. NISCH, 12th International Thermal Spraying Conference, London, UK 4-9, proceedings published by the Welding institute, 1, (june 1989), p 91
- /4/ D. BERNARD, M. VARDELLE, P. FAUCHAIS, A. VARDELLE, IEEE Transactions on Plasma Science (submitted for publication)
- /5/ D. ABOULKASSIM, B. PATEYRON, P. FAUCHAIS, 5th International Symposium on Plasma chemistry, Pugnuchiuso, Italy, ed. R.D. AGOSTINO, (4-8 sept. 1989), L-51
- /6/ BRITE Report (period 01.10.89/28.02.90) from MTU partner P-2280-1-87