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≫ 연구논문 ≪

플라즈마 분사 처리에 의한 Cr_2O_3 조립분말의 구상화에 대한 연구

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Spherodization of Granuled Cr_2O_3 Fine Ceramic Powder by Plasma Spray

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요약

Spray dried Cr_2O_3 분말은 겉보기밀도를 향상시키기 위해 plasma flame에 투입하여 실험을 진행 하였다. 첫번째 고밀도화 공정에서의 분말은 입자내부 공간까지 완전히 용해되어지지 않았으며, 두번째 공정 이후 완전히 용해가 되었다. 두번째 공정 결과 분말 입도는 작아졌으며, 용해 및 표면 연화에 의해 겉보기 밀도와 유동도는 향상이 되었다. 두번째 고밀도화 공정이 후 부분적으로 입자들이 30 μm 이상의 hollow structure을 보여주고 있다. 분말의 이러한 고밀도화는 plasma flame에 의해 응집되어진 응집체내의 열전도율 및 내부 가스압의 관점에서 정량적으로 논의 하였다.

주제어 : Cr_2O_3, 플라즈마 프레임, 겉보기 밀도, 고밀화

Abstract

Spray dried Cr_2O_3 powder having an agglomerated structure of particles was twice treated into a plasma flame to increase its apparent density. The powder subjected to the first densification treatment did not show the entirely melted state keeping inner particle hollows, and it was fully melted after the second processing only. The powder size as a result of the second treatment decreased, and the apparent density as well as flowability were increased due to melting and surface smoothing effects. But a part of particles after the second densified treatment showed the hollow structure, especially those which were above 30 μm in size. This densification behavior of the powder has been qualitatively discussed in terms of the thermal conductivity and inner gas pressure within aggregates exposed to the plasma flame.

Key words : Cr_2O_3, Plasma frame, Apparent density, Densification

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1. INTRODUCTION

The spray drying process is widely used for agglomerating the fine ceramic powder into that with larger spherical particles to improve the powder flowability[1)]. Especially it fulfills the role of a convenient process to fabricate uniform sized thermal spraying ceramic powders, and even gives the possibility to produce composite powders[2)]. But due to porous structure of its particles, the spray dried powder shows a lower apparent density than that of the fused+crushed ceramic powder for thermal spraying.

Thus, the densification process by melting in a plasma flame was used to improve the apparent density of Al_2O_3, Cr_2O_3 powders and other ceramic systems[3-5)]. However, the studies on the thermal spray coatings with densified powder are usually devoted to the mechanical properties and microstructure evaluation of coated layers. On the other hand, details of the microstructure change in treated powders and their densification behavior are not fully understood.

In this study, the morphology, cross sectional microstructure and some characteristics of the spray dried Cr_2O_3 powder densified by a plasma flame investigated, and the densification behavior of aggregates into plasma jet was qualitatively discussed.

2. EXPERIMENTAL

The spray dried powder was produced in a centrifugal disk atomizer with the disk diameter of 66 mm, its rotation speed of 11,000 rpm and the chamber temperature of 110°C, using the slurry made of the clean water, raw Cr_2O_3 powder (~ 1 μm) and PVA (polyvinylalcohol) binder. The powder content, PVA concentrations in the powder and the slurry feed rate were fixed as 20 wt.%, 2 wt.% and 85 g/min, respectively, taking into account results of the previous work[2)].

The spray dried powder was twice melted into the flame of the METCO 9MB plasma gun, collecting it under cold water. The plasma jet power was 43 kW, plasma gas pressures were 100 psi for Ar, and 25 psi for H_2. The powder feed rate was 90 g/min. The flowability and apparent density of spray dried and densified powder were measured by the standard method of ASTM B 213-83 and 214-86, and the morphology and cross sectional microstructure were observed by a scanning electron microscope.

3. RESULTS AND DISCUSSION

3.1. Examination of spray dried and densified powder

In spray drying process, agglomerated powders are produced through three steps: 1) transferring the slurry to the rotating disk, 2) atomizing the slurry into droplets by centrifugal force owing to the disk rotation, 3) formation of spherical aggregates dried by evaporating water out of slurry droplets inside the hot chamber. The effect of process parameters on the properties of a spray dried powder was in detail discussed in the previous paper[5)].

The SEM morphology and cross sectional microstructure of the produced spray dried Cr_2O_3 powder are given in Fig. 1. It shows the spherical shape particles with the well agglomerated inner structure.

The SEM morphologies and cross sectional microstructures of the spray dried Cr_2O_3 powder after the first and second densification treatments in the plasma flame are presented in Fig. 2 and Fig. 3, respectively. After the first spraying processing, the powder exhibits the partially melted state only. It was observed that the fraction of spherodization by melting was nearly 60% level in the first plasma treatment. The particles become fully melted as a result of the repeated powder treatment, but there are still many hollow particles. This implies that the shrinkage of the particles due to melting is

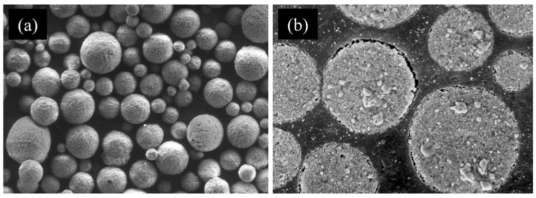

Fig. 1. SEM morphology (a) and cross sectional microstructure (b) of the spray dried Cr_2O_3 powder.

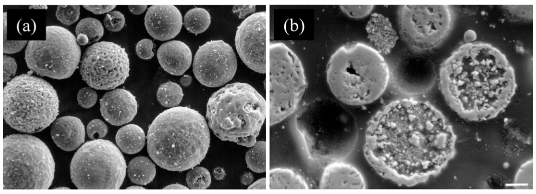

Fig. 2. SEM morphology (a) and cross sectional microstructure (b) of the spray dried Cr_2O_3 powder after the first plasma treatment.

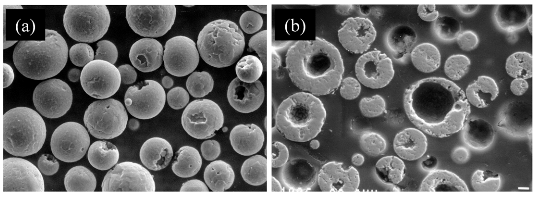

Fig. 3. SEM morphology (a) and cross sectional microstructure (b) of the spray dried Cr_2O_3 powder after the second plasma treatment.

accompanied by hollow formation inside some of them. Presumably, such a powder behavior is caused by the effect of a gas pressure inside hollows due to PVA evaporation in agglomerates during their melting in the plasma flame.

Dependence of the average size, apparent density and flowability of the powder on the treatment steps is shown in Fig. 4. The mean size decreased, and the apparent density and flowability increased after each treatment step due to the melting effect. But nearly all the increase of flowability is yielded in the first densification treatment. This was probably due to the fact that the powder particles have become smooth enough to maximize the flowability resulting from solidification of the liquid film on their surface.

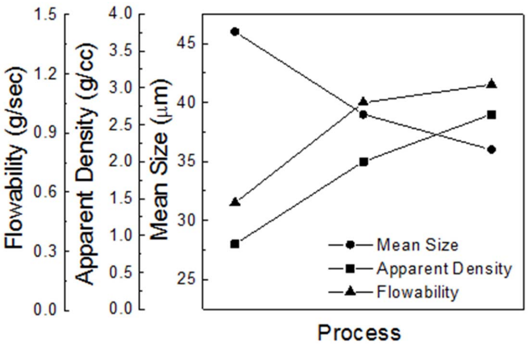

Fig. 4. Effect of the first and second plasma treatments on characteristics of the spray dried Cr_2O_3 powder.

3.2. Densification behavior

The cross sectional microstructure of particles after the second plasma treatment of the powder is given in Fig. 3(b). The mean aspect ratio of spherical particles treated was measured to about 0.98. The relatively large particles have the hollow structure, while the small ones with sizes below approximately 20 μm are fully densified because of melting without any visible inner voids. This speculation well corresponds with the results of measurement of the particle size distribution (Fig. 5). The distribution curves for densified powders have the bimodel feature, and new pulses appear below the particle size of around 25 μm. It means that after the first treatment the small particles of the spray dried powder shrink without any hollows inside them, forming a new pulse on left side of the curve. On the other hand, the large particles do not fully shrink due to formation of inner hollows, and finally there results in the appearance of the bimodel particle size distribution.

A reason causing the inner void formation within solidified particles is probably the presence of a gas inside hollows captured and sealed by the melted layer over the particle surface during treatment of the powder in the plasma flame. In a plasma jet with the extremely

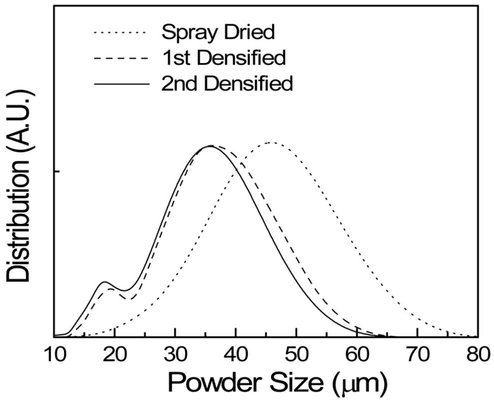

Fig. 5. Size distribution change of the spray dried Cr_2O_3 powder as a result of the first and second plasma treatments.

high velocity of particle movement of around 200 m/sec and temperature of 20,000°C, the flying aggregates can reside in plasma flame during very short time of about 0.003 sec[6)]. For this period, the agglomerated particles injected from the gun exit may initially transfigure with forming a surface liquid film like in Fig. 6. Here, the binder inside micropores between very small agglomerated grains forming the more coarse particles of the raw spray dried powders, evaporates into gas state. As the melting progresses, the wall thickness of liquid films increases, and the pressure of the binder vapor and environment gas trapped in micropores may also increase due to decreasing volume of both the particles and isolated inner hollows. So, reducing the particle size will be retarded, and the raw powder particles with the liquid walls will continue to melt toward the inner wall surface. Finally aggregates solidify forming a shell structure of large particles of the powder treated in the plasma flame.

The wall thickness in solidified particles may be increased by decreasing the air and PVA fraction in the raw dried powder, which causes a gas pressure inside hollows. The small fraction of the trapped air and binder can be obtained in more dense aggregates, which have also the better thermal conductivity. In the more dense aggregates, the core temperature can be higher than that in agglomerates with lower density, and the more thick particle walls will be achieved.

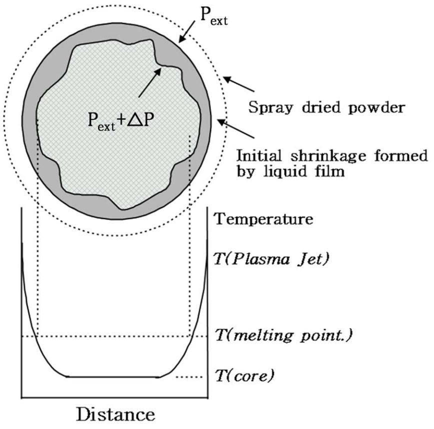

Fig. 6. Scheme of the temperature distribution inside a spray dried Cr_2O_3 powder particle in plasma flame.

In its turn, the spray drying condition may be well controlled to maximize the apparent density of the spray dried powder, so that the inner particle voids during densification could be minimized. It is known that in the spray drying condition, the increase of the powder concentration in the slurry improves the densification of aggregates and increases the mean size of spray dried powder particles, whereas the increase of the slurry feed rate and speed of disk rotation decrease their sizes[6)]. Thus, to produce the spray dried powder with the smaller particles providing the better densification, the spray drying process has to be regulated by choosing the proper powder concentration and slowing down the slurry feed rate and speed of disk rotation.

Figure 3(b) shows the cross sectional microstructure of the fully melted relatively large particles. Here, all hollow particles seem to have nearly the same wall thickness of around 12 μm even though their outer diameters are different. It means that any other spray dried powder with the aggregated particles of different sizes having almost the same ability to shrink causes the similar temperature gradient in particles during the plasma treatment. On the other hand, the final mean wall thickness is thought to depend on the powder materials with different thermal conductivity.

In the spray dried Cr_2O_3 powder fully melted as a result of the second plasma treatment, particles reduced in the mean size around 23% from 47 to 36 μm (Fig. 5). As it follows from Fig. 3(b), after the second treatment the mean wall thickness of the hollow particles changes to a small extent with changing the average particle size. In this study, a mean wall thickness is about 11,5 μm. Therefore, the size of a fully dense particle without any inner hollow is equal to 23 μm (Fig. 7). Since the particle size reduces near to 23% after the second treatment, the initial size of this particle of the critical size was approximately 30 μm.

Thus, it is important that the wall thickness of spray dried powder particles after the melting treatment can be predicted in beforehand. According to this study, the initial spray dried powder is required to be produced with agglomerates below 30 μm in mean size. An

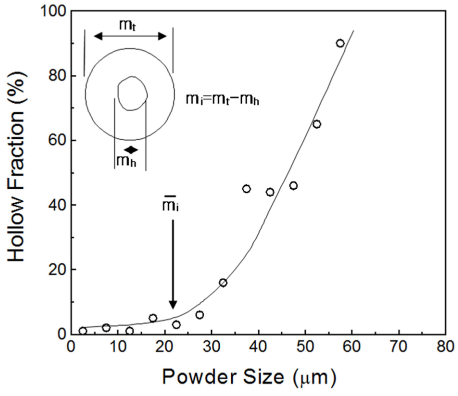

Fig. 7. Dependence of the hollow particle fraction on the mean size of the spray dried Cr_2O_3 powder twice treated in plasma flame.

analytical approach to this concept has been developed in the studies of other powder systems like Cr_2O_3+TiO_2, Al_2O_3, Al_2O_3+TiO_2[7,8].

4. CONCLUSION

The agglomerated Cr_2O_3 powder made by the spray drying process was densified via two treatments in plasma flame. The mean size of powder particles was decreased, while the apparent density and flowability were improved by the plasma treatments. The powder particles were not fully melted as a result of the first treatment, and partly unmelted inner structure of the particles was observed. The powder particles were fully melted after the second treatment only, and particles with sizes above 23 μm still had the hollow structure with the nearly constant wall thickness of 12 μm. During the plasma treatments, the formation of the shell structure of particles was due to stopping their shrinkage by the gas presence inside hollows. The critical size of the spray dried powder which can be melted without the inner void formation is to be below 30 μm.

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	년회비	30,000원		년회비	50,000원
종신회원	입회비	10,000원	종신회원	입회비	10,000원
	종신회비	300,000원		종신회비	500,000원
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