

NOVEL PROCESS FOR CARBON COATING OF CERAMIC PARTICLES

M. Inagaki and H. Miura

Faculty of Engineering, Hokkaido University, Kita-ku, Sapporo, 060 Japan

Introduction

Polyvinyl chloride (PVC) is well known to be carbonized through liquid phase with low viscosity. When PVC powders were heated in an alumina crucible up to 1000 °C, the inside wall of the crucible was found to be homogeneously coated by residual carbon. We were interested in this phenomenon because it suggested us some possibility to coat ceramic particles, such as alumina, with carbon starting from PVC.

In the present work, carbon coating of fine particles of some ceramics was found to be successfully carried out by a simple mechanical mixing of ceramic particles with PVC powders, followed by heating up to 1000 °C in inert atmosphere.

Experimental

The starting ceramic powders and PVC powders used in the present work were listed with a range of their particle size and specific surface area in Table 1. Different particle sizes of α -Al₂O₃ and γ -Al₂O₃ were employed. The particles of the other ceramics were not defined, but they had a large range in size and morphology. PVC powder with average degree of polymerization of 700 (PVC-A) was mostly used, but those with different degrees shown in Table 2 were also used.

These ceramic particles were mechanically mixed with PVC powder in different ratios by using spatula and then heated up to 1000 °C in an alumina boat in a flow of argon gas with a rate of 40 ml/min for 1 hour. After heat treatment, the color, size and morphological aspect of the particles recovered were examined by eyes, optical microscope and SEM. The structure of ceramic particles before and after carbon coating was studied by X-ray powder diffraction. The amount of residual carbon after heating was determined from the weightloss by thermogravimetry up to 1000 °C in a flow of oxygen.

Results and Discussion

Alumina particles

When an appropriate mixing ratio between alumina particles and PVC, starting white particles changed to completely black, as will be explained below, but no carbon coating was observed on the wall of alumina boat. In Fig. 1, SEM photographs of α - and γ -alumina particles after carbon coating are shown. No changes in apparent size and morphology were observed, suggesting homogeneous coating of particle surface by thin carbon film.

In Fig. 2, the morphological aspect and the amount of residual carbon are summarized as a function of mixing ratio of PVC in starting powder mixtures. From the mixtures with high PVC contents, coagulation of particles was observed by eyes and also the deposition of carbon on the wall of alumina boat. The powders obtained from the mixtures with low PVC contents showed gray color and was found white uncoated particles under optical microscope. Between these two regions, homogeneous coating of particle surface by carbon was obtained, no coagulation being detected under microscope and all parts being real black.

It has to be mentioned that γ -alumina has much wider range for homogeneous carbon coating than α -alumina, independent of particle size. This difference on two kinds of alumina particles is reasonably supposed to be due to a big difference in their specific surface area. High surface area of γ -alumina was kept after carbon coating; surface area of 95 m²/g on the starting particles was changed to 58 m²/g after carbon coating. After coating at 1200 °C, γ structure was transformed to α structure, though no structure change occurred at 1000 °C. Carbon coated γ -alumina changed its structure to α -alumina in a flow of oxygen at 1200 °C, but all carbon could not be burned out, black spots were remained inside of the particles with α structure. This experimental fact suggests the permeation of carbon into porous γ -alumina particles during coating process and the occlusion of carbon inside of α -alumina particles after phase transformation.

PVC powders with different molecular weights gave the same results for carbon coating of α - and γ -alumina particles, much wide range of PVC content for homogeneous coating being observed on γ -alumina.

It was experienced that the mixing of fine powders of ceramics and PVC is not essential. Even when PVC

powders were placed on the layer of alumina powders, the coating was performed though a little inhomogeneity was observed between the particles near PVC layer and far from it.

Other ceramic particles

Carbon coating of silica, titania and magnesia particles was successfully done by the same procedure for alumina. PVC content in the starting mixtures for obtaining homogeneous coating without coagulation was not so wide as γ -alumina and rather similar to α -alumina, and the amount of carbon residues was also the same as alumina shown in Fig. 2.

From carbon-coated particles, magnesia was dissolved

out in sulfuric acid and got hollow carbon shells were obtained (Fig. 3), which were burned out in oxygen flow, giving a direct evidence of the formation of carbon layers on the ceramic particles.

In the case of titania with anatase structure, phase transformation to rutile was observed during carbon coating at 1000 °C

In the case of calcia, its reactions with carbon dioxide and water in the atmosphere with 80 % humidity at 25 °C were detected after carbon coating. This shows that the formed carbon layer on the ceramic particles by the present procedure is porous.

Table 1 Ceramic particles used

Ceramics	Particle size μm	Crystal structure	Specific surface area m^2/g
$\gamma\text{-Al}_2\text{O}_3\text{-A}$	50 ~200	spinel	95
$\gamma\text{-Al}_2\text{O}_3\text{-B}$	40~ 50	spinel	160
$\gamma\text{-Al}_2\text{O}_3\text{-C}$	100~200	spinel	130
$\alpha\text{-Al}_2\text{O}_3\text{-A}$	30~100	corundum	1.1
$\alpha\text{-Al}_2\text{O}_3\text{-B}$	30~ 45	corundum	1.1
$\alpha\text{-Al}_2\text{O}_3\text{-C}$	60~70	corundum	1.2
SiO_2		amorphous	---
TiO_2		anatase	---
MgO		rock salt	---

Table 2 Polyvinylchloride powders used

Code name	Average degree of polymerization
PVC-A	700
PVC-B	500
PVC-C	1000

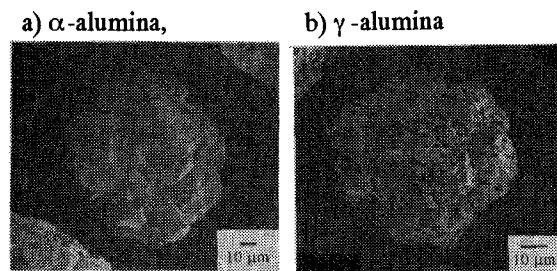


Fig. 1 SEM of the particles after carbon coating.

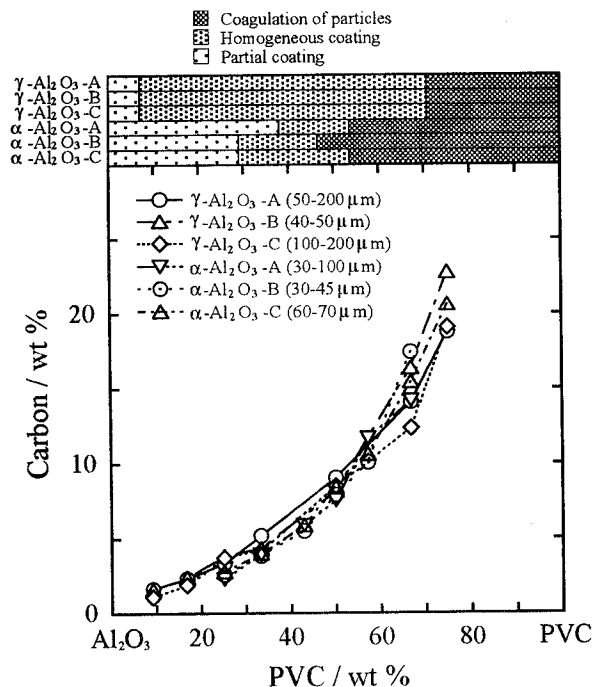


Fig. 2 Range of PVC content in the starting mixture for homogeneous coating and amount of carbon residues.