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Effects of suspension properties on the microstructure of Al_2O_3 coatings deposited onto macroporous SiC substrate

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ABSTRACT

In this paper the effects of the suspension preparation conditions on suspension coating of porous substrates were investigated. Alumina suspensions were prepared using varying types of additives, including non-ionic polyethylene glycol (PEG), cationic polyethylene imine (PEI), and anionic sodium carboxymethyl cellulose (CMC). Prepared suspensions were coated on porous SiC substrates by spin coating. The flow curves of the suspensions were measured, whereas their particle dispersion states were also observed directly using an optical microscope. In addition, the surface and cross-section of each coating were observed by SEM and analysed by EDX. Finally, the air permeability of each coating was measured.

For the suspension with PEG, alumina particles dispersed well in the suspension and the added PEG formed a network structure, resulting in a homogeneous coating on the surface of the substrate with less particle penetration into the substrate pores. For the suspension with CMC and the suspension with PEI, since particles flocculated in the suspensions, particle penetration into the substrate pores hardly occurred, however, the formed coating was inhomogeneous.

1. Introduction

Recently, porous ceramic materials have been widely used in various industries such as hot gas separation [1], direct methanol fuel cell [2], carbon dioxide capture and storage [3], particulate filters for diesel engines [4], and waste water treatment [5] industries. As such, much research related to porous ceramics has already been conducted. For practical use, a fine catalyst or catalyst-supporting powder is coated on the surface of porous ceramic substrates. The state of this coating on the substrate affects its performance. For example, if the fine particles form aggregates, the catalysis reaction efficiency decreases; if the fine particles penetrate into the pores of the substrate, the fluid permeation resistance increases. Therefore, the coating state, including the distribution of fine particles on and in porous substrates, needs to be controlled.

One of the coating techniques is dip coating of porous substrates into suspensions in which fine particles are dispersed in an appropriate medium. Usually these fine particles are dispersed in a medium with a dispersant and a binder, and dipping of the porous substrate into the prepared suspension is followed by drying and heating to immobilize the particles. Compared to coatings on glass or metal substrates with relatively smooth surfaces, few papers exist on coatings of fine particles on porous substrates. Paunovic et al. reported about silica coating on porous substrates [6]. Zhang et al. achieved a silicon coating on porous Si_3N_4 [7]. Agrafiotis et al. have coated a ceria-doped alumina on ceramic honeycombs [8]. Itoh et al. have coated $Ca(Ti,Fe)O_3$ on porous substrates [9]. Zwinkels et al. have described ceramic coatings on metal substrates [10]. However, the optimal properties of suspensions used for coating of porous substrates have not been identified yet, because these substrates often have relatively uneven surfaces and pore sizes that are larger than the size of fine catalytic particles. In addition, the desirable structure of fine particles coated on substrates varies by final product; thus, control of the structure is needed to be able to freely fabricate coatings with different structures. In previous papers on dip coating of porous substrates, mainly the apparent viscosity was investigated in order to optimize the slurry conditions for alumina coatings [11,12], Cu/Mn/ZnO catalyst coatings [13], γ-alumina coatings [14], and yttria-stabilized-zirconia coatings [15], while other suspension properties related to the particle dispersion state were not discussed. It was also reported that, in some cases, the apparent viscosity did not correspond well with the particle packing ability of a suspension [16–20], an important observation since the apparent viscosity is one of the most important parameters for the coating process. Therefore, further investigation of suspension properties is necessary to establish optimal suspension preparation guidelines to control the coating state. In this paper, the relationship between the suspension properties and the microstructure of coatings on porous substrates was examined by changing the type of suspension

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Nomenclatures

k	Viscosity constant in Eq. (1) [Pa•s^n]
n	viscosity index in Eq. (1) [−]
$\dot{\gamma}$	shear rate for flow curve measurement [s^{-1}]
τ	shear stress for flow curve measurement [Pa]
τ_c	yield stress (intercept of flow curve) [Pa]
ε_0	permittivity of free space [F•m^{-1}]
ε_r	relative permittivity [−]
ξ	zeta potential of particle [V]
u_{ave}	average particle mobility for electrophoresis [m^2•V^{-1}•s^{-2}]
μ	medium viscosity [Pa•s]
Φ	final volume fraction of formed sediment [−]
H	final height of formed sediment [m]
ϕ	initial volume fraction of slurry for settling test [−]
h	initial height of slurry for settling test [m]

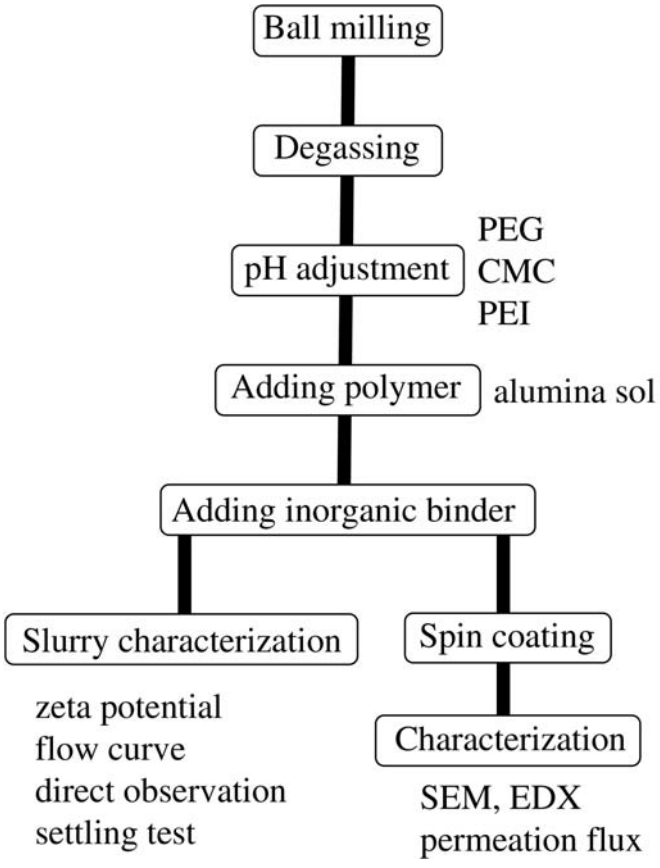

Fig. 1. Experimental flow.

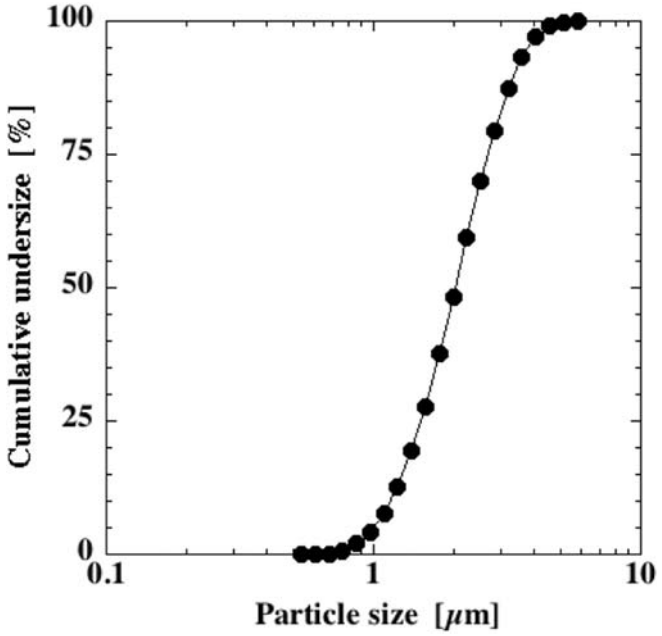

Fig. 2. Particle size distribution of alumina powder used in this study.

additives. For suspension characterization, not only apparent viscosity measurements, but also sedimentation tests were conducted. In addition, direct observations of particle dispersion and flocculation [21,22] were made. All results were analysed with regards to the final coating properties.

2. Experiment

Fig. 1 shows the experimental flow. In brief, experiments consisted of four parts: suspension preparation, suspension characterization, substrate coating, and coating characterization. Details of each aspect are described below.

2.1. Suspension preparation

An alumina powder with an average particle size of 1.98 μm was used as a model catalyst-supporting powder particle. The particle density, measured by pycnometry, was 2480 kg·m^{-3}. The alumina powder was mixed with deionized water to prepare a suspension with a particle concentration of 2.0 vol%. The prepared suspension was ball-milled for 4 h in a polyethylene pot with 1.5-mm-diameter zirconia beads. Fig. 2 shows the particle size distribution of the alumina powder after ball milling, measured by a laser diffraction particle size analyser (SALD-3100, Shimadzu Corporation). Subsequently, the suspension was degassed and its pH value adjusted to approximately 5.0 with 1 M HCl. An additive aqueous solution that was prepared in advance was then added to the suspension while stirring. The added amount was 2.4 wt% of the mass of the alumina powder in the suspension. The additives used in this study were the non-ionic polymer polyethylene glycol (PEG; M_w = 2000,000, Wako Pure Chemical Industries), the cationic polymer polyethylenimine (PEI; M_w = 70,000, Wako Pure Chemical

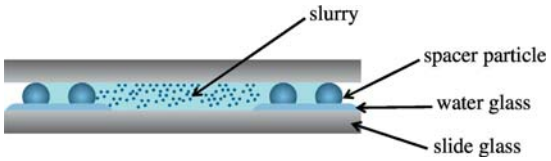

Fig. 3. Schematic illustration of sample configuration for direct observation of suspensions.