CHAPTER IV

EXPERIMENTS

The experiments of the development of alumina coating on ceramic monolith are classified into three parts:

1. Preparation of washcoat and surface characterisation
2. Comparison of amount of γ-Al_2O_3 deposited on monolith in the variety of coating conditions.
3. Physical properties testing of washcoated monolith

The detail of the experiments are explained in the following sections.

4.1 Preparation of washcoat and surface characterisation

4.1.1 Materials

The alumina powder used for preparing washcoat and the support are listed below:

1. Alumina powder (γ-Al_2O_3, size 5 μm) ,used for washcoating monolith, was obtained from Sumitomo Co., Ltd., Japan. (specification see Appendix B)

2.Cordierite Monolith, used as substrate, was obtain from N-COR, Ltd., Nagoya , Japan.

4.1.2 Preparation of monolith sample

The monolith test sample was cut into bar shape, 3 mm width ×3 mm height × 250 mm lenght from cordierite monolith (400 cells/in^2)

4.1.3 Surface Treatment

Monolith test samples were weighted and soaked in 2.5% by weight acetic acid or nitric acid solution for 2, 5, 10, 30, 60, 120, 540 min. Then, the

monolith samples were washed by distilled water several times to remove residual acid solution and dried in the oven at 110 °C until the weight becomes contant.

4.1.4 Characterization of surface-pretreated monolith by Scanning Electron Microscopic

The surface of the monolith samples which were treated by acid was enlarged by a scanning electron microscopic at Scientific and Technological Research Equipment Centre, Chulalongkorn University and National Metal and Materials Technology Center (MTEC). SEM was used to measure size of large pore on monolith surface and grainsize of washcoat.

4.2 Comparison of amount of γ-Al_2O_3 deposited on monolith in the variety of coating conditions

4.2.1 Preparation of slurry for washcoat

Alumina powder (TA) was added to 2.5 % by weight acetic acid solution to give 30-50% w/v alumina washcoat and stirred for 5-10 min.

4.2.2 Monolith coating procedure

Monolith test samples were dipped into the prepared washcoat for 2 min. and blown by compressed air in order to remove extra slurry washcoat in the cells of the monolith, followed by drying at 110°C over night in the oven. Some of the samples were repeatedly dipped in the washcoat 2-3 times and calcined at 500 °C - 600°C for 2-4 hr. in air atmosphere.

4.3 Physical properties Testing of coated monolith

4.3.1 Thermalshock Resistant

Thermal shock resistant of the monolith was tested by placed the sample in the oven at 600-800°C for 10 min. and rapidly took out to the air at room temperature. A fan was used to cool down the monolith for 10 min. Then, the test sample was placed back in the oven again. The monolith samples were tested for 5 times.

The surface of washcoated monoliths after thermalshock experiment were investigated by a scanning electron microscopic.

4.3.2 Abrasive Strength

The washcoated monolith was weighted and tested using a stainless tube, inside diameter = 0.95 cm. The hot air, 800°C, 200 ml/min was fed through the washcoated monolith for 24, 48, 72 hr. After that the sample was weighted again. The difference between the sample weight before and after the experiment represents amount of washcoat released from monolith surface.

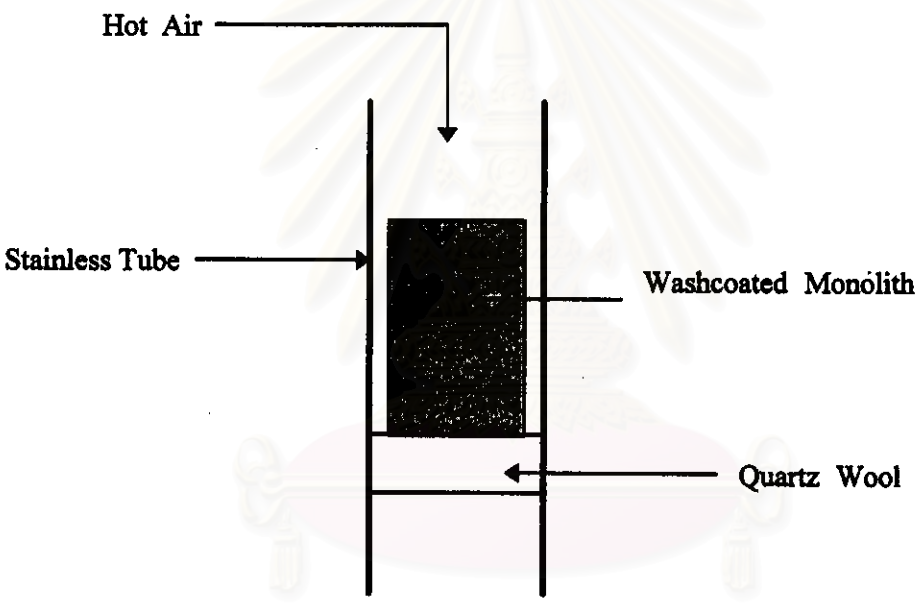

Figure 4.1 Flow diagram of the abrasive strength testing