CHAPTER V

RESULTS AND DISCUSSION

This chapter is divided into four sections. The first section, section 5.1, concerns about the effect of two types of acid and dipping time on surface of monolith in surface pretreatment. The second section, section 5.2, deals with the effect of various conditions on washcoat slurry preparing. The last two sections, section 5.3 and 5.4, are aimed at the study of some important properties, i.e. thermalshock resistance and abrasive strength, of washcoated monolith.

5.1 Comparison of the effect between acetic acid and nitric acid in surface pretreatment step

In this section, the study begins with soaking a 0.5×0.5×3 cm. monolith in two different acid solutions. Then, the effect of acid on surface of monolith sample in various soaking times from 0-9 hours was evaluated.

5.1.1 Effect of surface pretreatment in acetic acid and nitric acid solution on monolithic surface with time

Two different types of acid solution, Acetic acid [CH_3COOH] and Nitric acid [HNO_3], were used to prepare 2.5 wt% acid solution. The monoliths were soaked in each acid solution for 0 - 9 hours.

Figure 5.1 shows the surface of monolith before pretreatment. It was found that poresize on rough surface was in the range of 5-30 μm.

Figures 5.2(a) and 5.2(b) show the monolithic surface soaked in 2.5 wt% acetic acid and nitric acid solution for 2 min.,respectively. It was observed that poresizes on the surfaces of both pieces of monolith remarkably increased from the poresize shown in figure 5.1. This means that the surface of the monolith could be corroded by the acids. The difference between monolithic surface shown in figures 5.2(a) and 5.2(b) is that the monolith pretreated in acetic acid solution has larger poresizes (5-100μm.) than the other pretreated in nitric acid solution (5-60μm.). The reason is that nitric acid, strong acid , quickly corroded the surface of monolith in a short time. On the contary, acetic acid, weak acid , can continuously give H^+ ions , thus its acidic strength reduced slower . Thus, the surface could not be suddenly severely corroded

Figures 5.3(a) and 5.3(b) display the monolithic surfaces which were pretreated in 2.5 wt% acetic acid and nitric acid solution for 5 min., respectively. It was found that the monolith which was pretreated in nitric acid solution had poresize about 5-60 μm. which is the same size as of the sample treated for 2 min., slightly smaller than the sample pretreated in acetic acid solution(5-50μm.).

Figures 5.4(a) and 5.4(b) show the surface of monoliths that were pretreated in 2.5 wt% acetic acid and nitric acid solution for 10 min., respectively. There is a much difference between poresizes on the monolithic surface. For the surface pretreated in nitric acid solution, the surface of monolith was corroded and released because of strong acidity of HNO_3. Thus, there was small pores on the surface in the range of 5-20 μm. For acetic acid surface pretreatment, the pores are in the range of 5-50 μm.

Figures 5.5(a) and 5.5(b) show the monolithic surface that was soaked in 2.5 wt% acetic acid and nitric acid solution for 30 min.,respectively. Large pores , about 100 μm., on monolithic surface that was pretreated in acetic acid was observed. The appearance of the large pores was due to the corroded surface still came off. Then, the inner large pore appeared which was corroded slower.

For monolithic pretreatment in nitric acid solution, the pore on the surface of monolith has the size of 5-20 μm. It was the same size as of the sample pretreated in nitric acid for 10 min and smaller than that of the samples treated for 2 and 5 min. The reason is H^+ ions was almost used up, therefore, the corrosion of monolithic surface was nearly constant.

Figure 5.6(a) shows that the monolith soaked in 2.5 wt% acetic acid solution for 1 hour. This sample has poresize on surface in the range of 5-50 μm. It was due to the same reason that the upper surface was driven. The similar result of surface pretreatment in nitric acid solution was found in Figure 5.6(b) which show the surface of monolith pretreated in 2.5 wt% nitric acid for 1 hour.

Figures 5.7(a) and 5.7(b) show the monolithic surface soaked in 2.5 wt% acetic acid and nitric acid solution for 2 hour, respectively. The poresize on the surface of monolith pretreated in acetic acid solution had sharply reduced to the range of 5-20 μm. but the very large pore still appeared. On the otherhand, monolith which was soaked in nitric acid solution shows the same pore sizes (5-20μm.) as of the sample pretreated for 30 min. and 1 hour.

Figures 5.8(a) and 5.8(b) show the surface of monolith which were pretreated in 2.5 wt% acetic acid and nitric acid solution for 9 hour, respectively. There is no difference between both samples. The pores on surface of the monolith soaked in acetic acid are in the range of 5-30 μm. No large pores was observed.

For the monolithic surface pretreated in nitric acid , the poresizes are in the same range(5-20μm.) as of the samples pretreated for 30 min, 1 hour and 2 hour. However , both of poresizes showed in figures 5.8(a) and 5.8(b) have a larger sizes than the pores on the surface of the unpretreated monolith.

From the above results, it can be noted that the surface pretreatment of monolith in acid solutions has the effect on poresizes on the surface. When monoliths were pretreated for a short time, the poresize had remarkably increased and the poresize had reduced almost to the poresize of the unpretreated monolith. Then, it was continued to observed the appropriate time required for surface pretreatment.

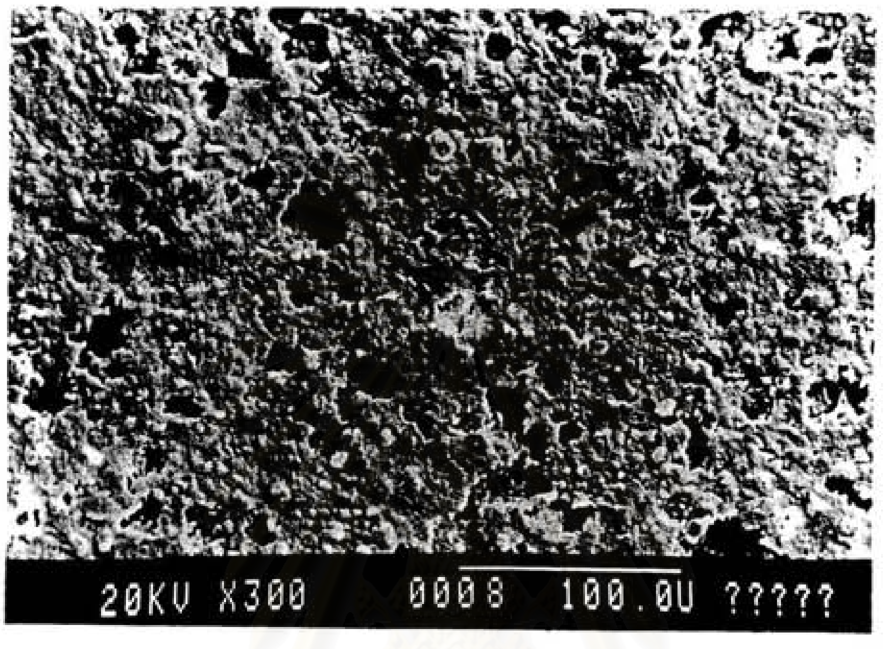


Figure 5.1 Surface of monolith

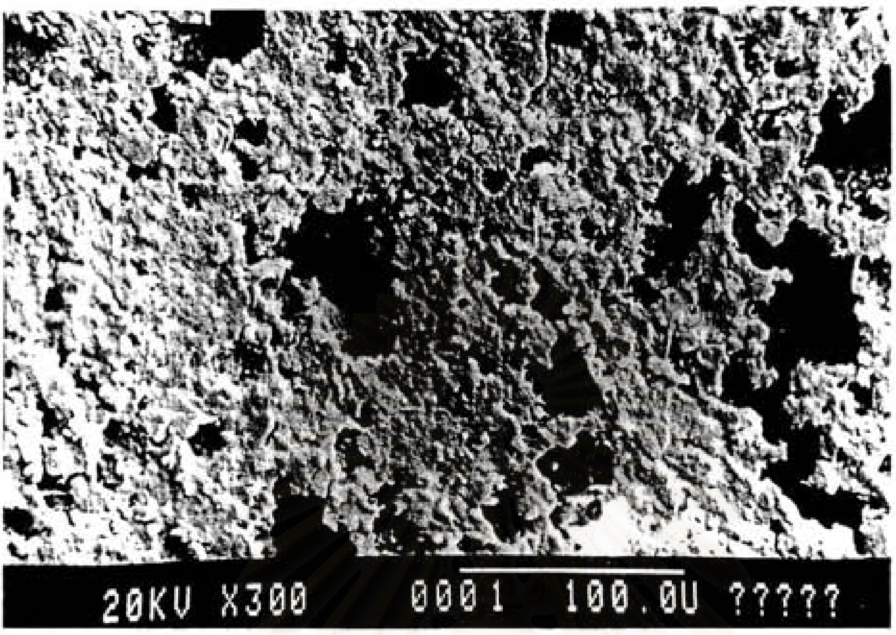


Figure 5.2(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 2 min.

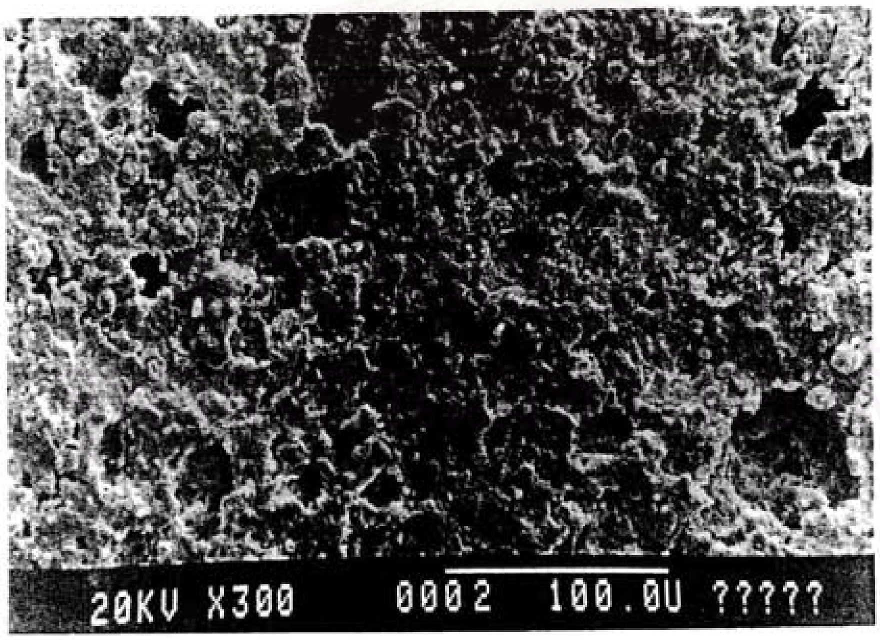


Figure 5.2(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 2 min.

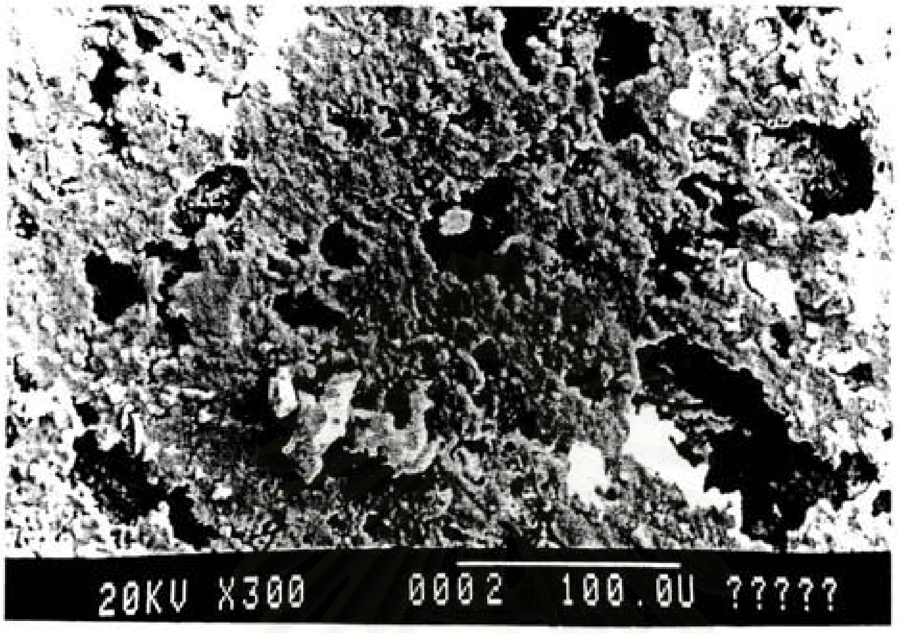


Figure 5.3(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 5 min.

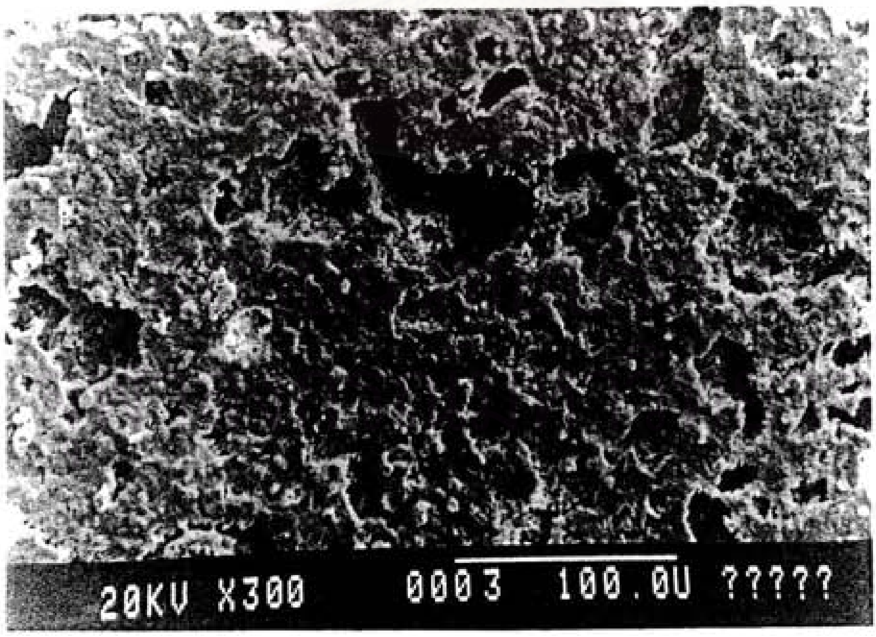


Figure 5.3(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 5 min.

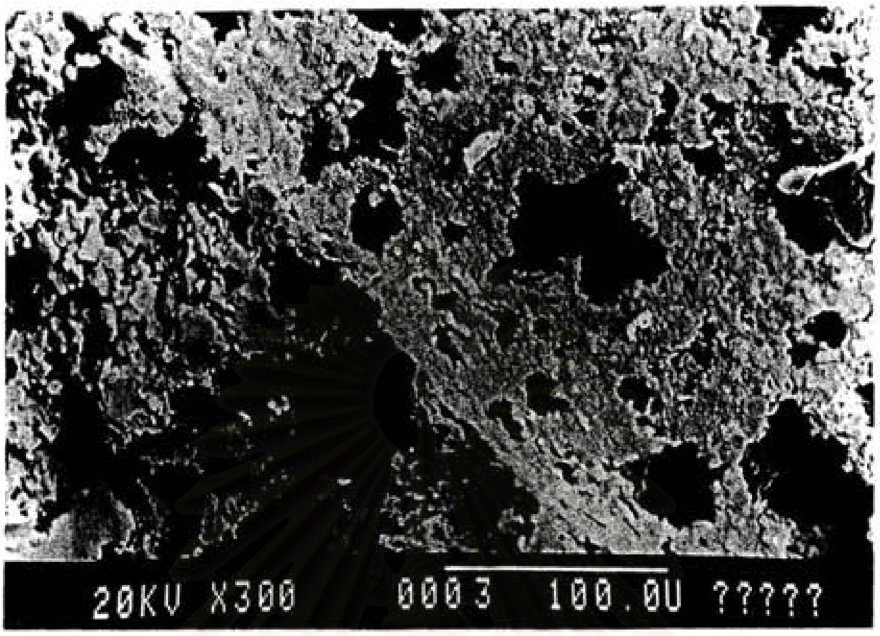


Figure 5.4(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 10 min.

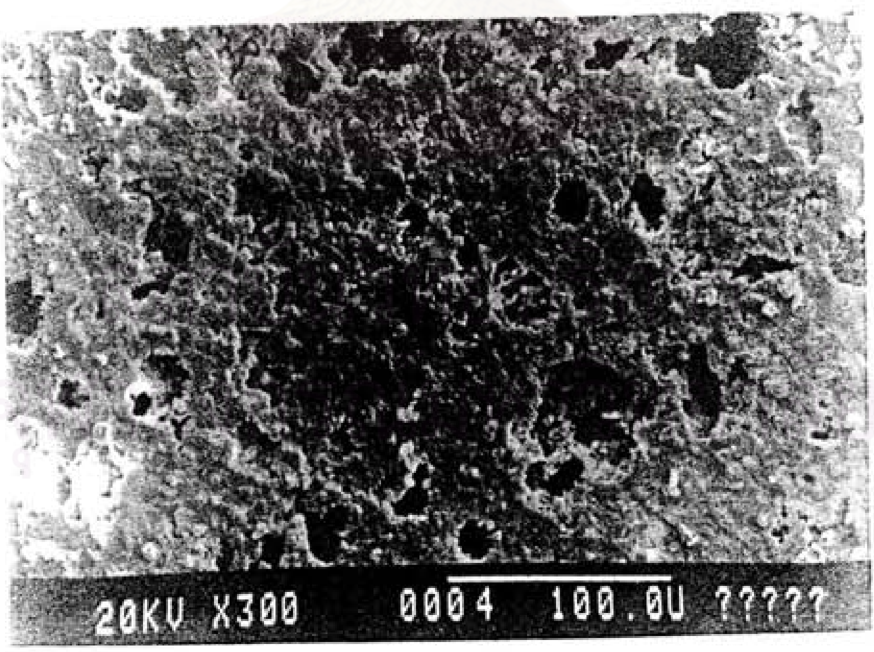


Figure 5.4(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 10 min.

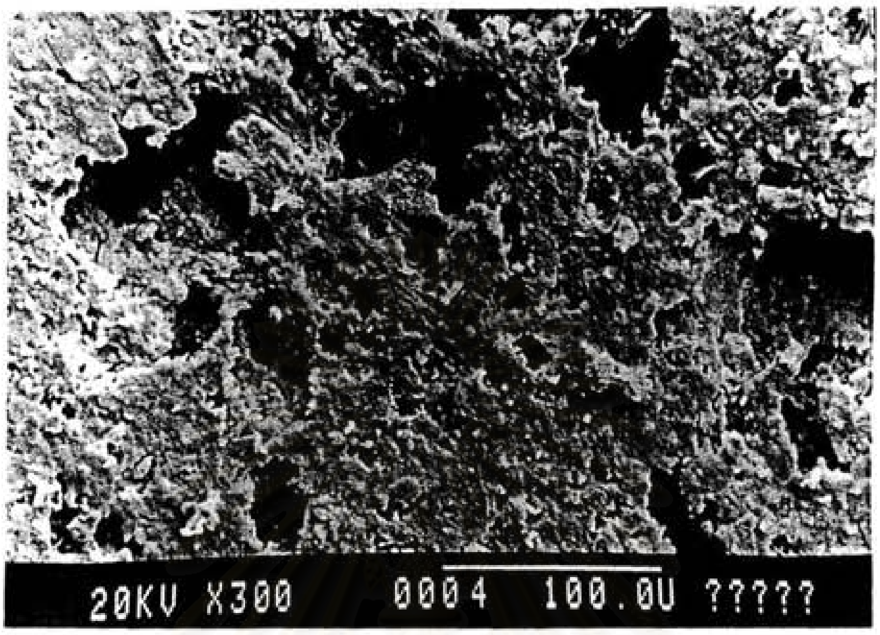


Figure 5.5(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 30 min.

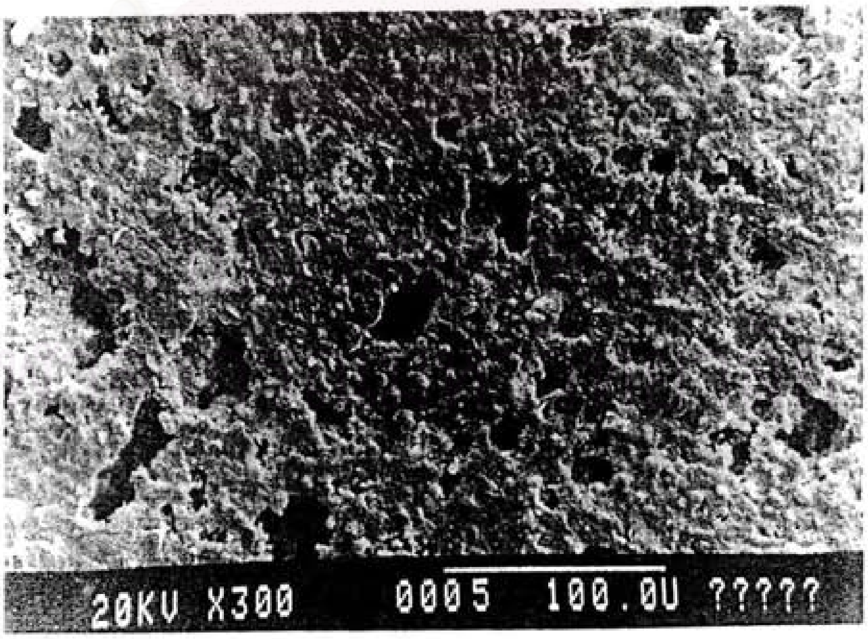


Figure 5.5(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 30 min.

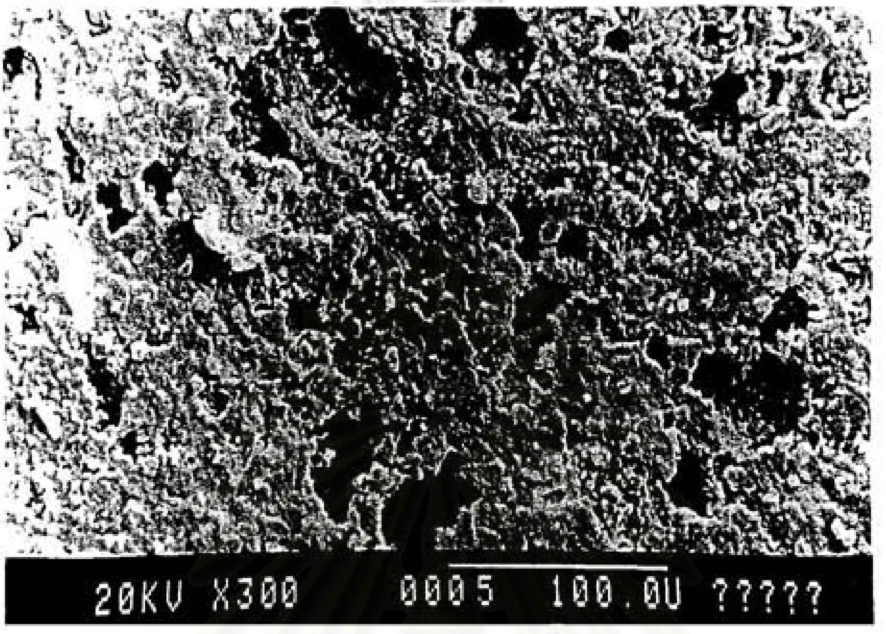


Figure 5.6(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 1 hour

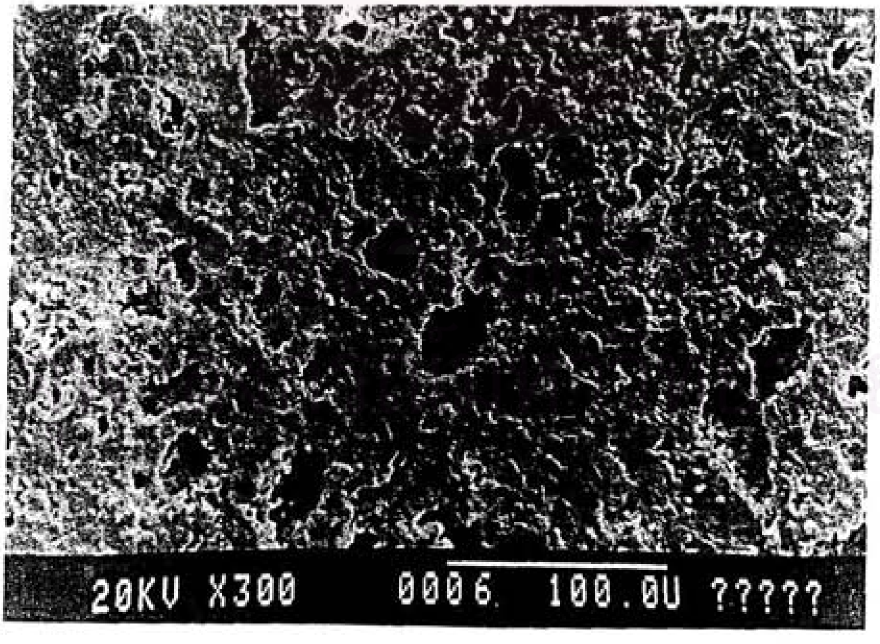


Figure 5.6(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 1 hour

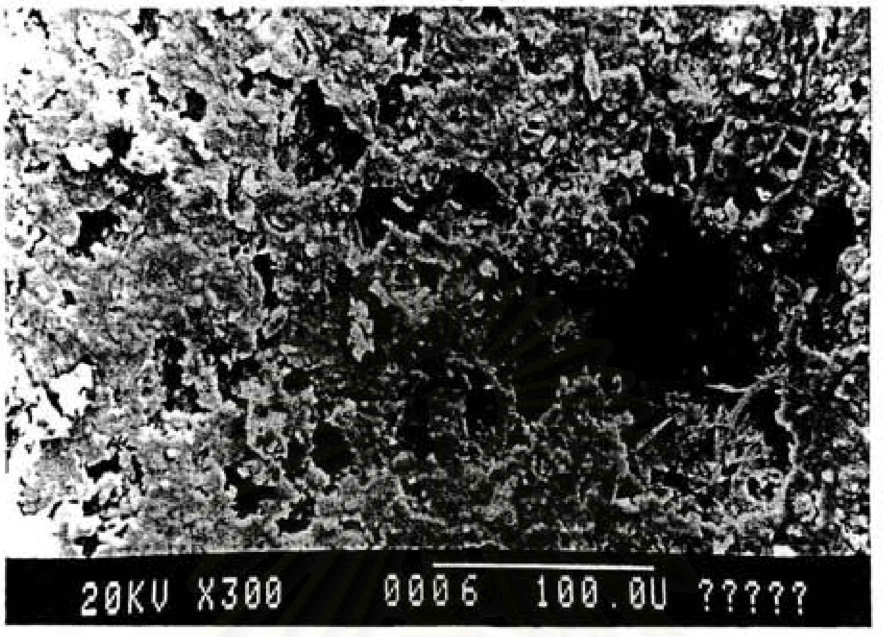


Figure 5.7(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 2 hour

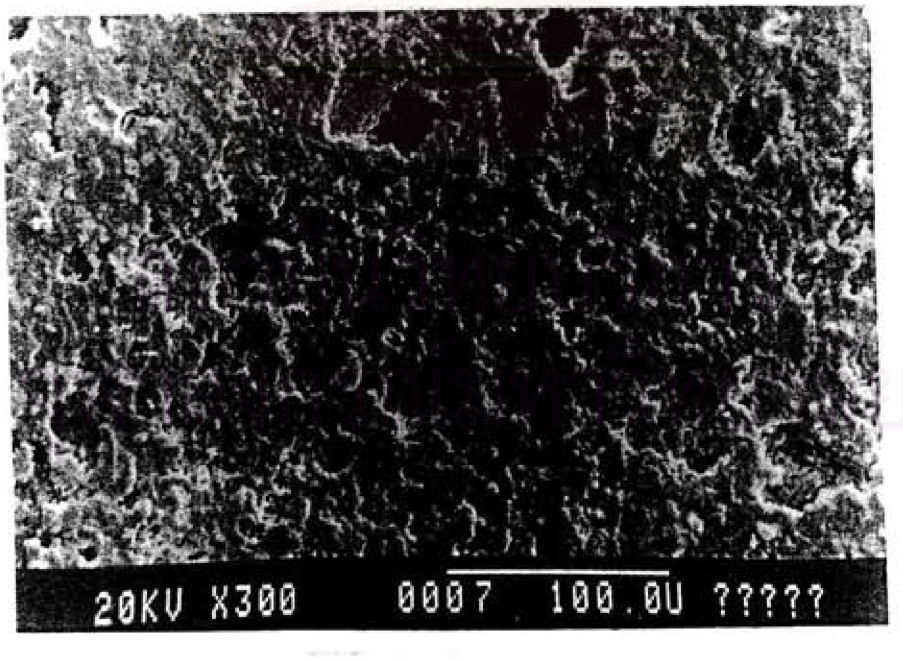


Figure 5.7(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 2 hour

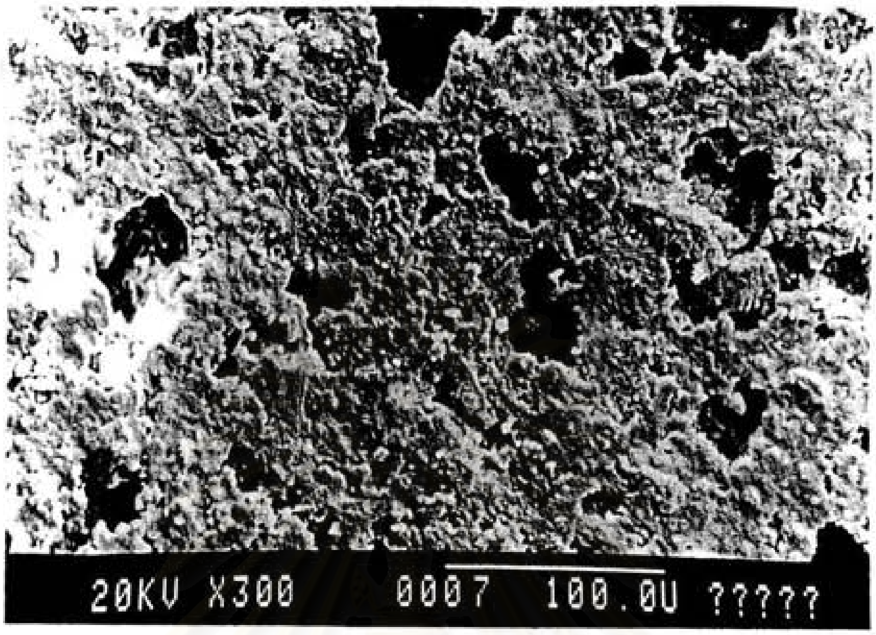


Figure 5.8(a) Surface of monolith pretreated in 2.5 wt% CH_3COOH for 9 hour

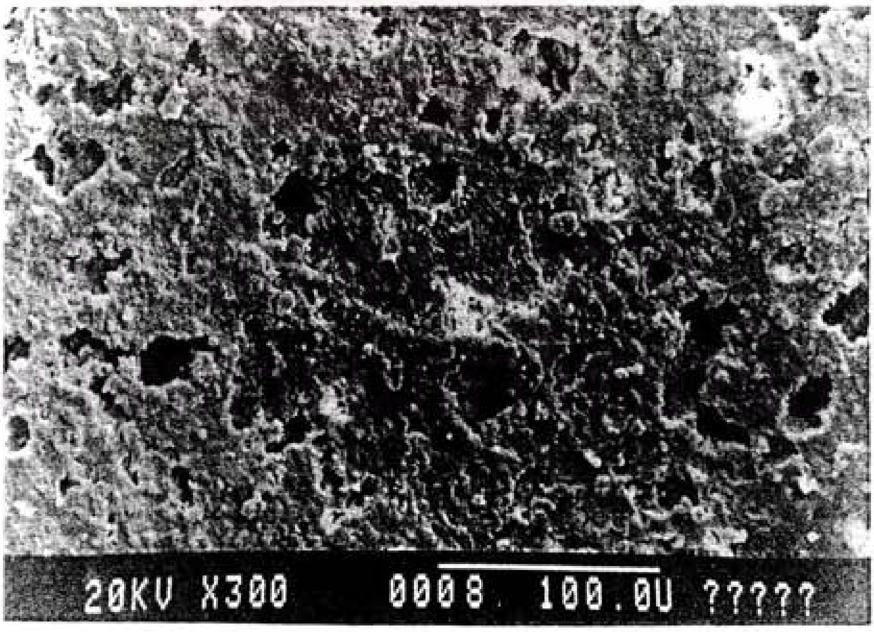


Figure 5.8(b) Surface of monolith pretreated in 2.5 wt% HNO_3 for 9 hour

5.1.2 Effect of surface pretreatment time on monolithic surface

In this part, the monoliths were pretreated in two acid solution, 2.5 wt% acetic acid [CH_3COOH] and nitric acid [HNO_3], in the holding times same as the previous part, 2, 5, 10, 30 min. 1, 2 and 9 hour.

Figures 5.9-5.16 show the effect of increasing surface pretreatment time on surface of monolith soaked in 2.5 wt% acetic acid solution for 2, 5, 10, 30 min. 1, 2 and 9 hour,respectively. Figures 5.9-5.13 show that poresize on the surface of monolith remarkably increased for soaking time from 2-30 min. to the range of 80-100 μm. After that, their sizes(5-30 μm) gradually reduced from 1-9 hour as displayed in figures 5.14-5.16 , but had larger poresizes and surface were smoother than the unpretreated monolith shown in figure 5.9. It should be noted here that the monoliths soaked for 2-30 min. had larger poresize than those soaking for 1-9 hour. This is because acetic acid corroded the surface of monolith and the pores were enlarged. After that, the rough surface was released and poresize became small.

Figures 5.17-5.24 show the effect of surface pretreatment time on surface of monolith soaked in 2.5 wt% nitric acid solution for 2, 5, 10, 30 min. 1, 2 and 9 hour, respectively. It was found that poresizes on the surface of monolith sharply increased when soaking for 2-5 min. The poresizes are in the range of 10-60 μm. Then, poresizes decreased after soaking for 10 min.- 9 hr. This is because nitric acid corrosion as same as acetic acid which demonstrated in figures 5.22-5.24.

It should be noted here that there is a difference in surface pretreatment time that poresizes turned to reduce. Monoliths that were soaked in nitric acid solution took a little time for a large poresize to change to a smaller poresize. This can be explained by the fact that, nitric acid is a so strong acid so surface of monolith was corroded quickly. After that, poresizes were nearly constant because H^+ ions were consumed almost completely in a short time. But acetic acid ,weak acid, in the same time still had H^+ ions. The surface of monolith soaked in acetic acid solution was slowly corroded.

It can be concluded that the monolith poresizes became large when pretreat monolith in acetic acid solution for a short time. Then, alumina washcoat can easily deposit on the monolith. The longer the surface pretreatment time the weaker the monolith structure. The appropriate time required for surface pretreatment of monolith is 2 min. However, the other factors such as weigth loss of monolith during in surface pretreatment was also studied.

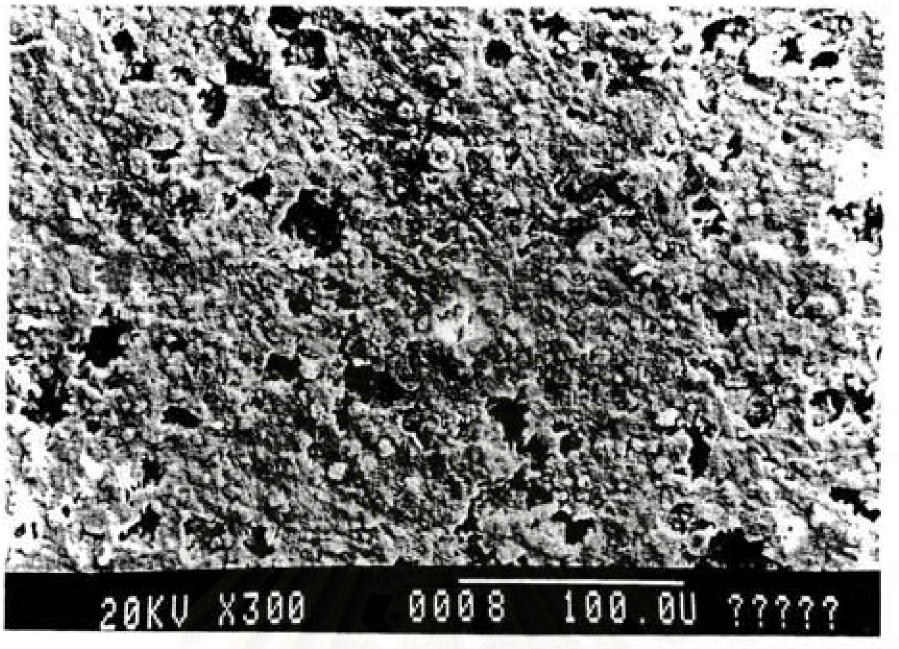


Figure 5.9 Surface of monolith

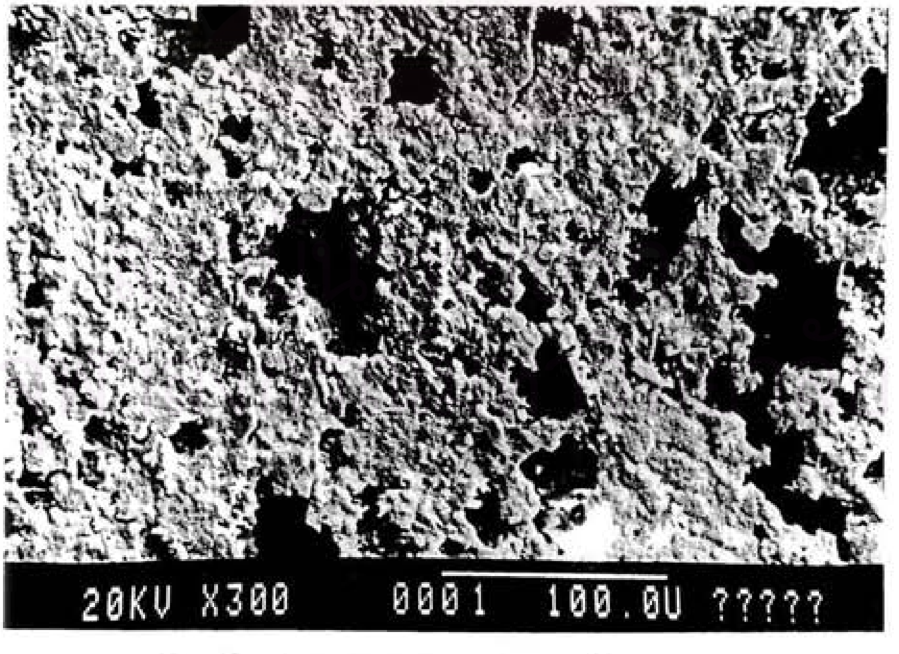


Figure 5.10 Surface of monolith pretreatment in 2.5 wt% CH_3COOH for 2 min.

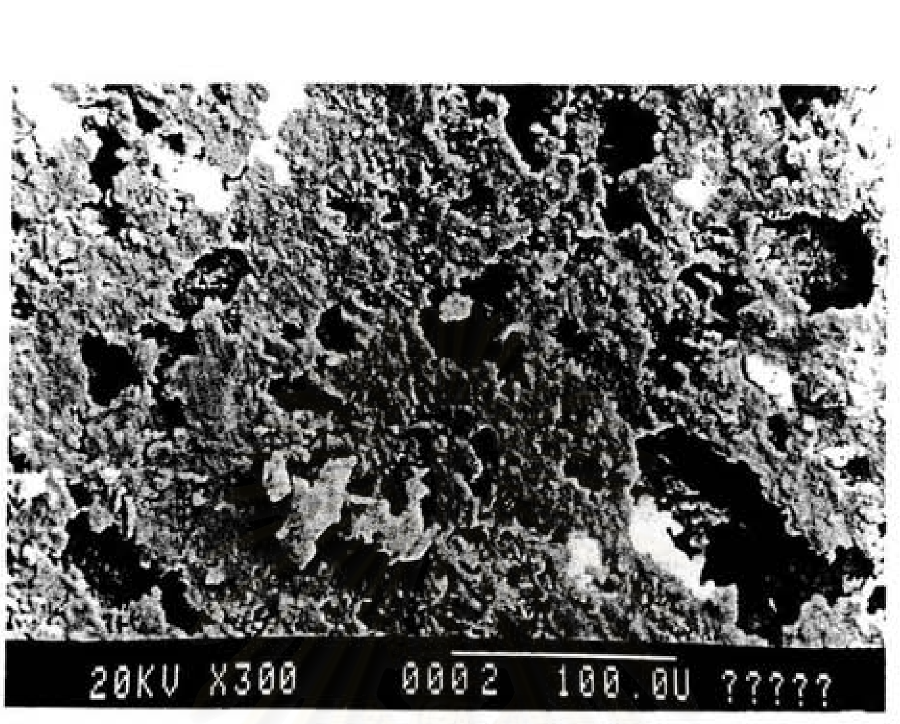


Figure 5.11 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 5 min.

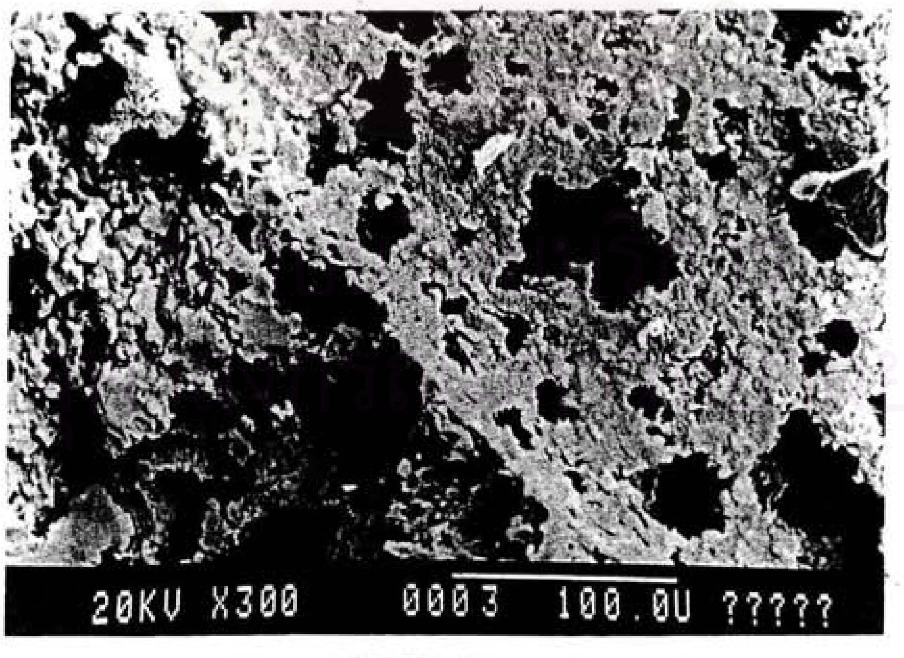


Figure 5.12 Surface of monolith pretreatment in 2.5wt%CH_3COOH for 10 min.

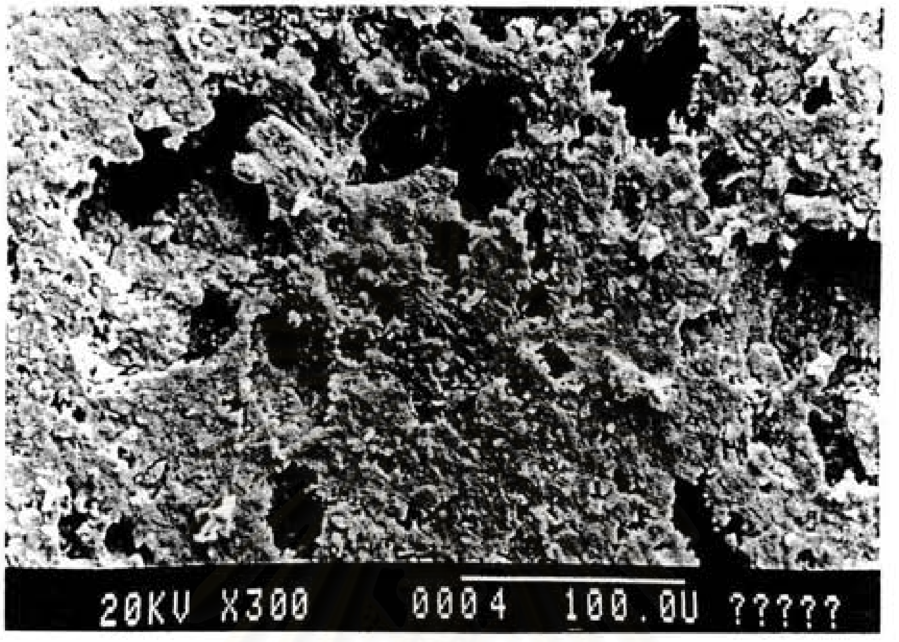


Figure 5.13 Surface of monolith pretreatment in 2.5wt%CH_3COOH for 30 min.

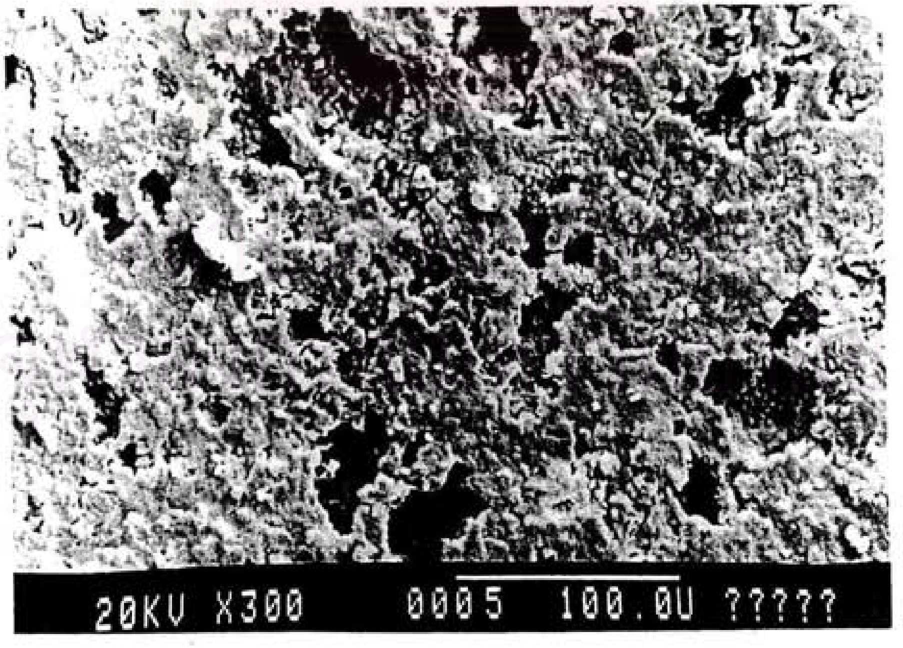


Figure 5.14 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 1 hr.

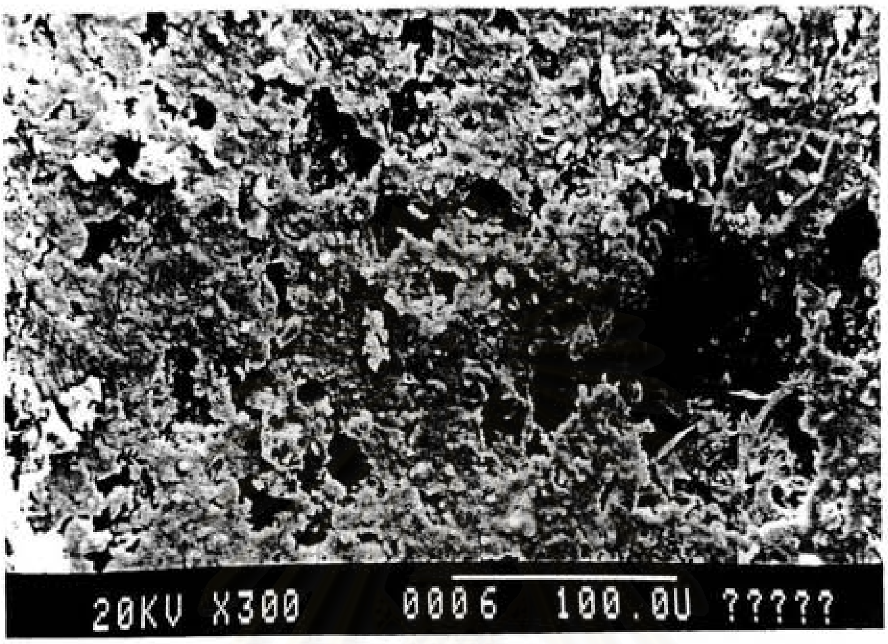


Figure 5.15 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 2 hr.

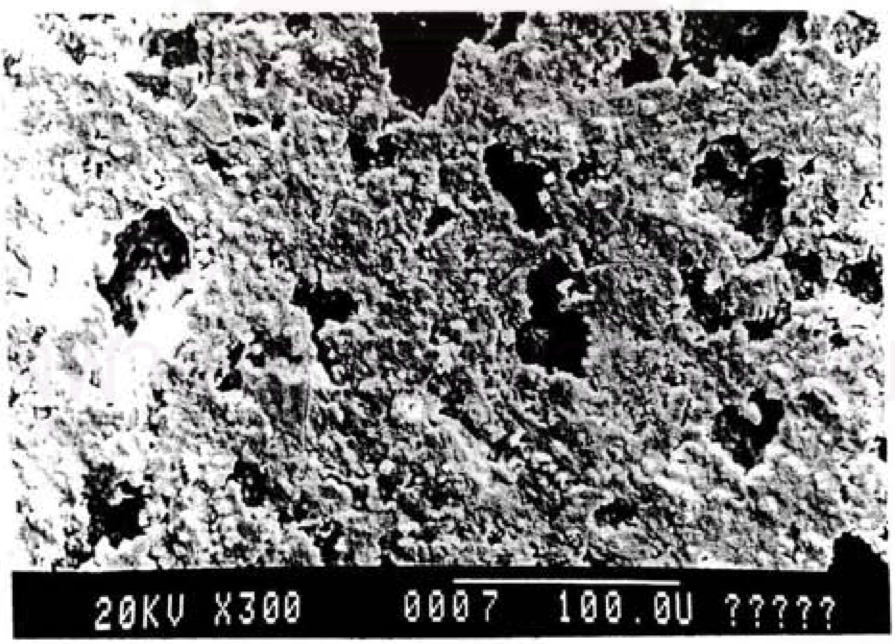


Figure 5.16 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 9 hr.

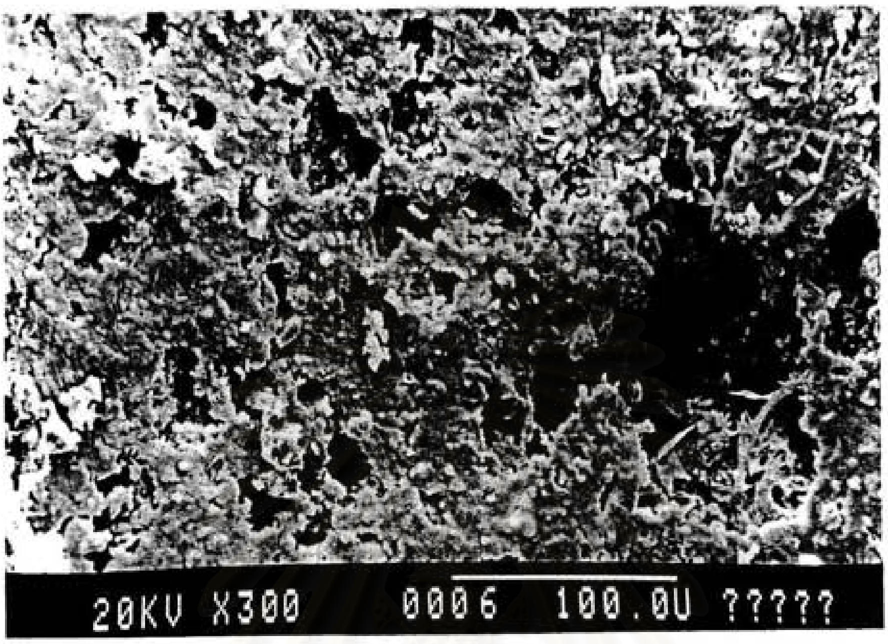


Figure 5.15 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 2 hr.

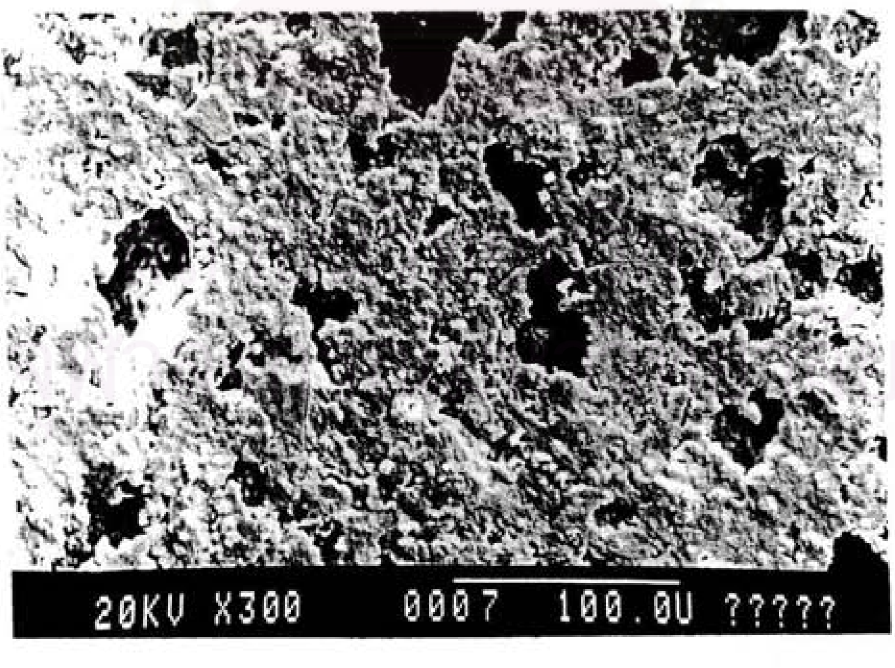


Figure 5.16 Surface of monolith pretreatment in 2.5 wt%CH_3COOH for 9 hr.

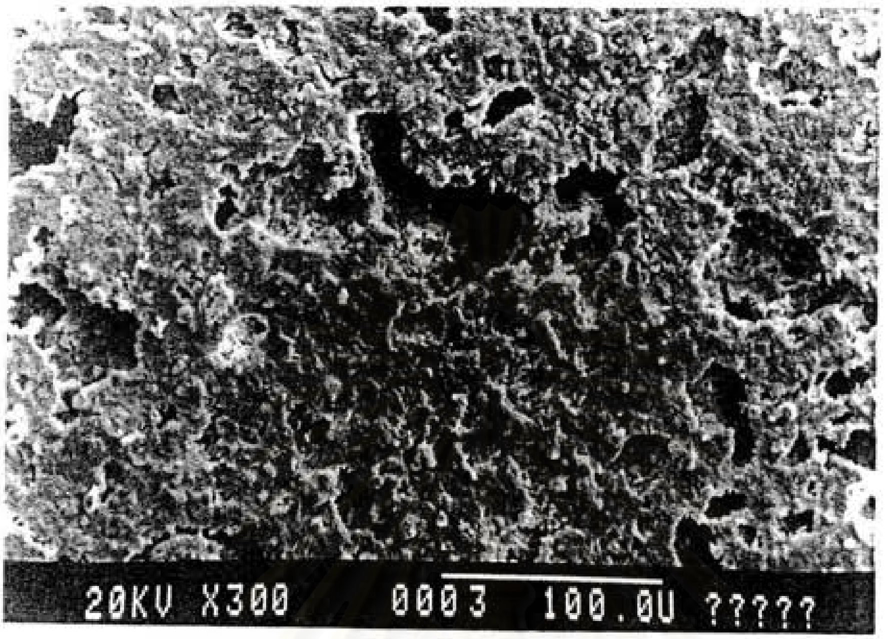


Figure 5.19 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 5 min.

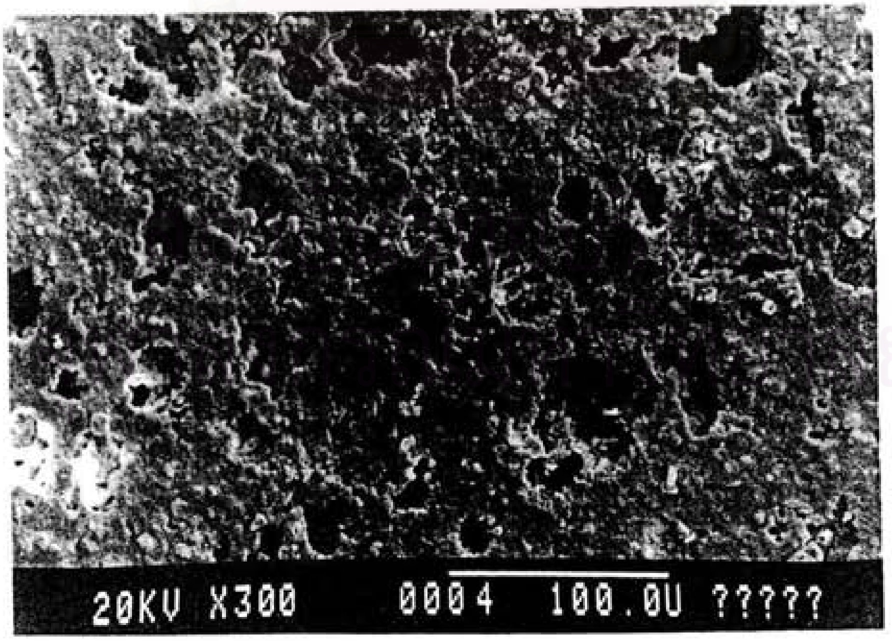


Figure 5.20 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 10 min.

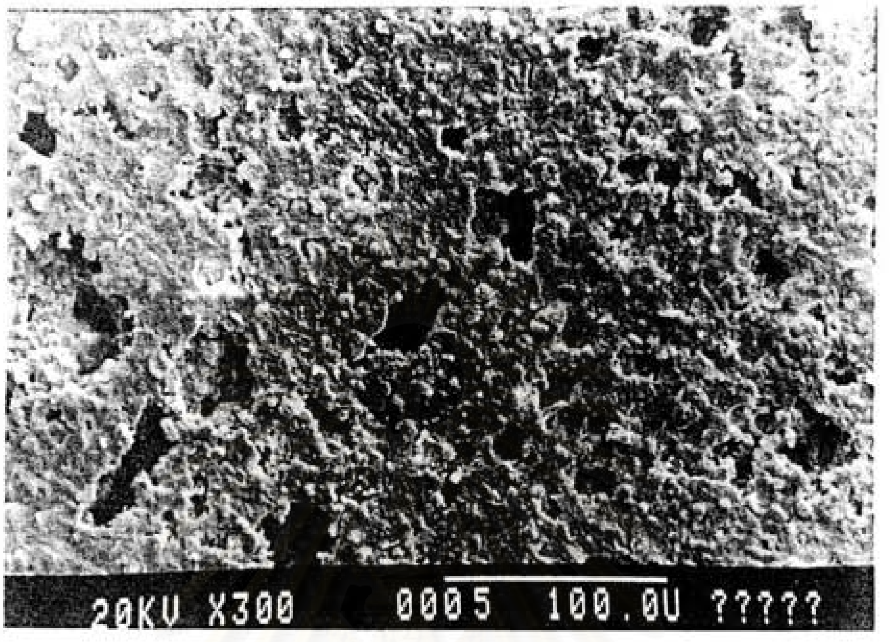


Figure 5.21 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 30 min.

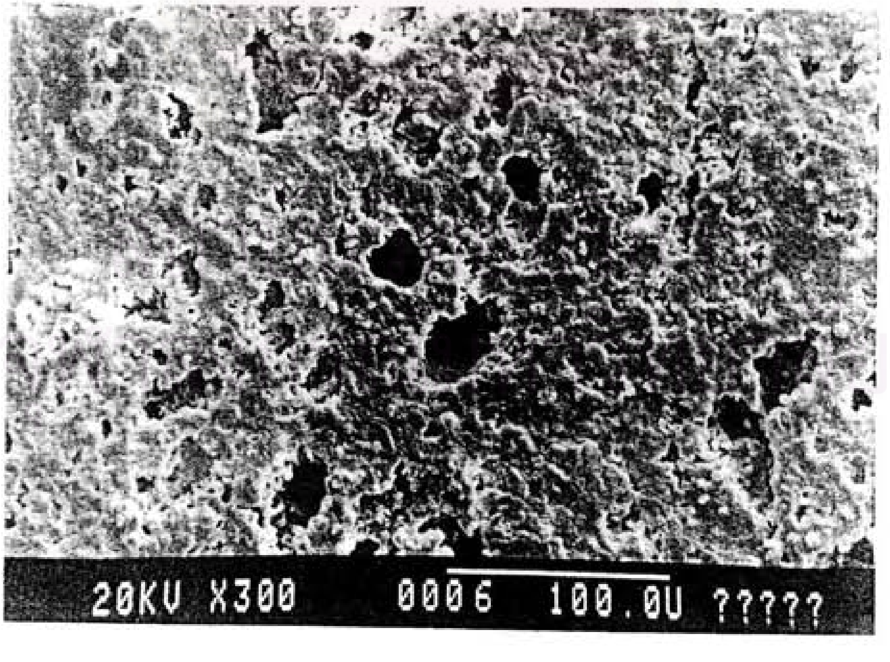


Figure 5.22 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 1 hr.

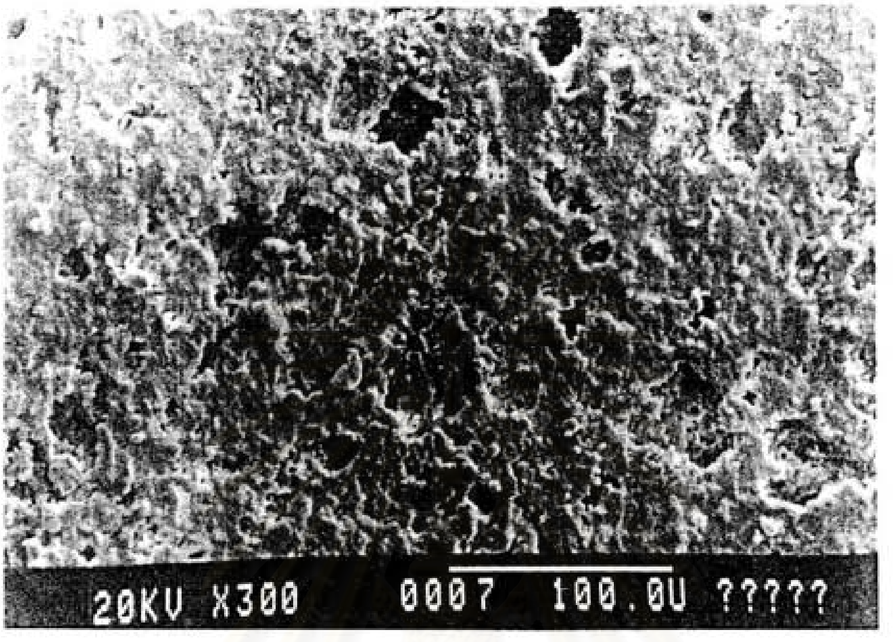


Figure 5.23 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 2 hr.

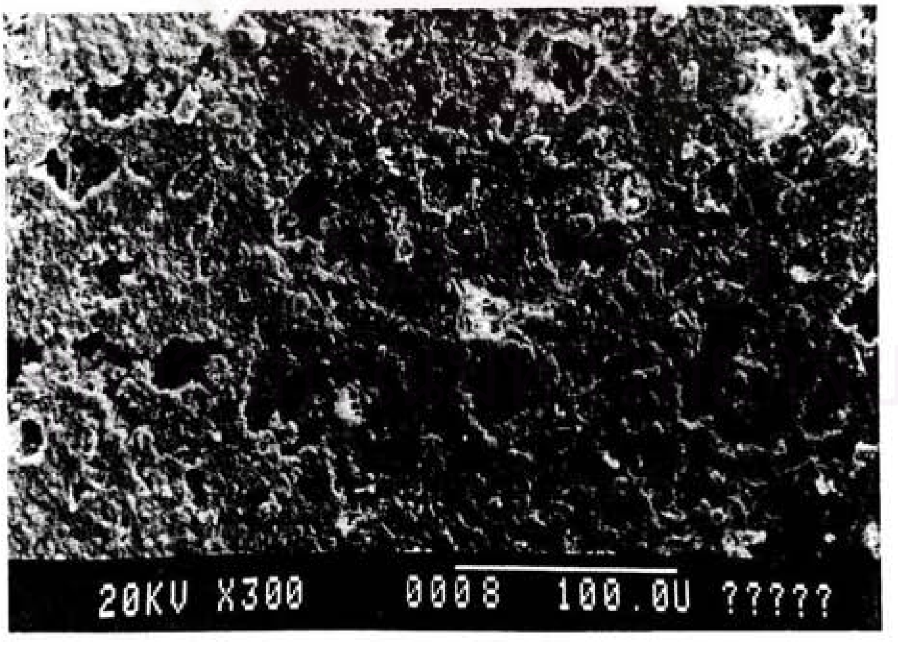


Figure 5.24 Surface of monolith pretreatment in 2.5 wt% HNO_3 for 9 hr.

5.1.3 Effect of surface pretreatment time on weight loss of monoliths

In this part, five pieces of 0.5×0.5×3 cm. monolith were soaked in 2.5 wt% acetic acid solution for the same series of soaking times as previous. Then, the samples were dried at 110°C until their weight became constant.

Figure 5.25 exhibits weight loss of monolith in surface pretreatment step. It was found that %weight loss of the monolith almost constant and had a little value, 0.05-0.08 %weight loss of monolith, when compare with weight of monolith. This means that the weight of monolith dose not significantly change during surface pretreatment.

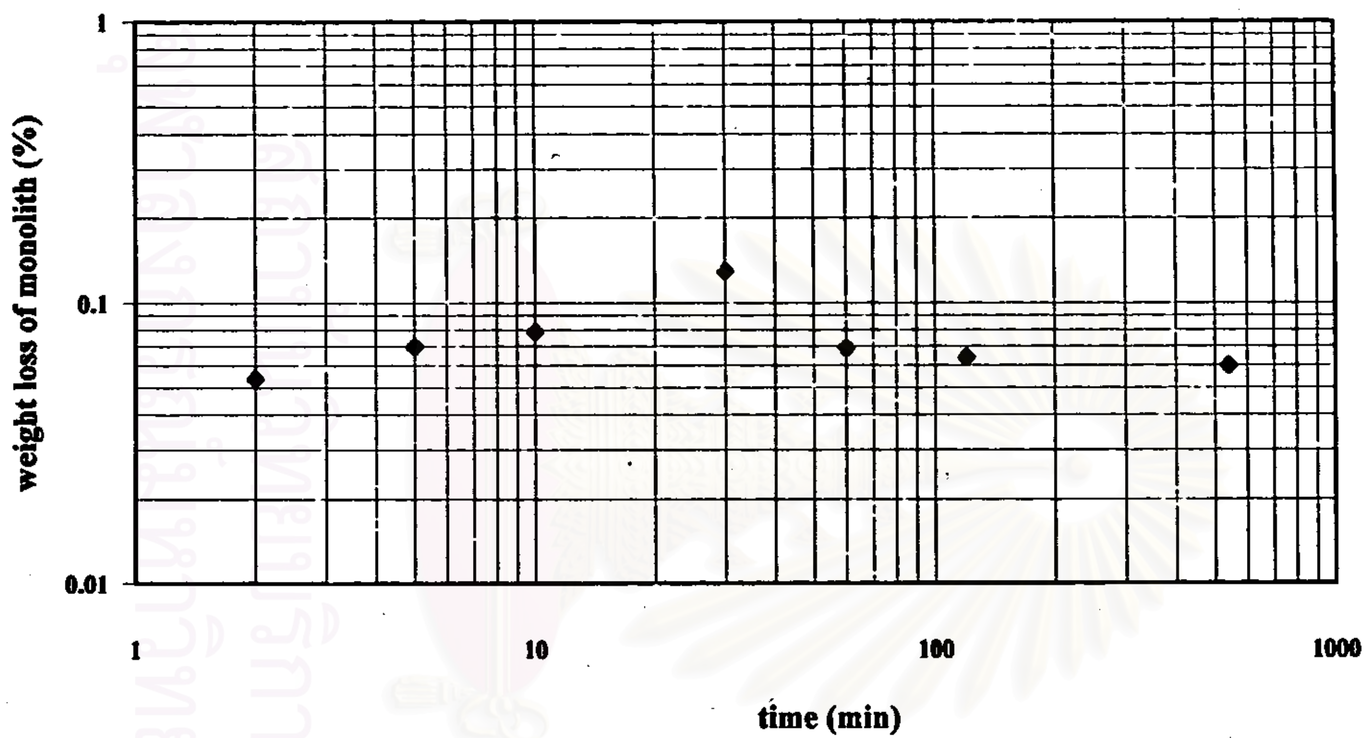


Figure 5.25 Weight loss of monolith in acid surface pretreatment

5.2 Effect of various conditions on washcoat preparing

In the previous section, section 5.1, it was showed that the 2.5wt% acetic acid solution can be used to prepare washcoat slurry and the surface pretreatment time of 2 min was prefer because of large poresizes.

In this section, the effect of alumina content in washcoat slurry will be investigated first. Then, the effect of times of dipping on surface coating was studied.

5.2.1 Effect of alumina content in washcoat slurry on surface coating

The monoliths were dipped in different washcoat slurry concentration by varying alumina content ranging between 30%-50% of alumina. The weight of washcoat deposited on monolith are listed in Table 5.1

Table 5.1 Weight of washcoat deposited on monolith in various alumina contents

% Alumina in washcoat	30	40	50
% Alumina deposited on monolith	4.11	11.68	28.14

It was observed that % by weight of alumina deposited increased with % alumina in washcoat slurry. To prepare washcoat slurry, 30% alumina content is not appropriate because a little of alumina can deposited on monolith.

At 50 % alumina content, the washcoat slurry had so high viscosity that when the washcoat slurry so strongly stuck on the cell of the monolith that it was so hard to purge the excess slurry from the cell. Hence, 40% alumina content is favorable. For this reason, the following study is chosen at 40% alumina in washcoat slurry to be main washcoat concentration.

5.2.2 Effect of times of dipping on surface coating

The monoliths were dipped in 40 wt% alumina in washcoat slurry by varying times of dipping ranging between 1-3 times. The weight of washcoat deposited on monolith are listed in Table 5.2

It was noted that the % weight of washcoat deposited on monoliths raised in a linear fashion with times of dipping

Table 5.2 Weight of washcoat deposited on monolith in various times of coating

number of coating	1	2	3
% weight of washcoat	11.68	20.11	29.81

5.3 Thermalshock Resistance of washcoated monolith

The purpose of this study is to study the thermalshock resistance property of washcoated monolith. First, the study began with the effect of varying times for coating washcoat on monolith, holding time in calcination step, calcination temperature and ended with the effect of thermalshock temperature on monolith prepared by previously mentioned procedure. The obtained experimental results are reported below.

5.3.1 Effect of times of coating washcoat on monolith

0.5×0.5×3 cm. monoliths were dipped in 40 wt% alumina washcoat slurry for 2 min. The times of coating were ranging between 1-3 times.

Figure 5.26 shows the surface of washcoat deposited on monolith for one time dipping. There was smooth surface and had small grains in the range of 100 μm.

Figure 5.27 shows the washcoated surface of a monolith for two times washcoat dipping. It was found on the smooth surface that the washcoat grains on the surface are in the range of 200-300 μm.

Figure 5.28 shows the surface of washcoat deposited on monolith for three times washcoat dipping. The smooth surface had large grains in the range of 200-1000 μm.

From the experimental results, it was found that the grains of washcoat deposited on monolith became larger when the monolith was coated several times, thus 3 times of washcoat dipping shows the result with the largest grains and the smallest grains were obtained from the surface of washcoated monolith for 1 time washcoat dipping. The thermal shock resistance property of washcoated monolith was studied later to observed the characteristics of washcoated monoliths.

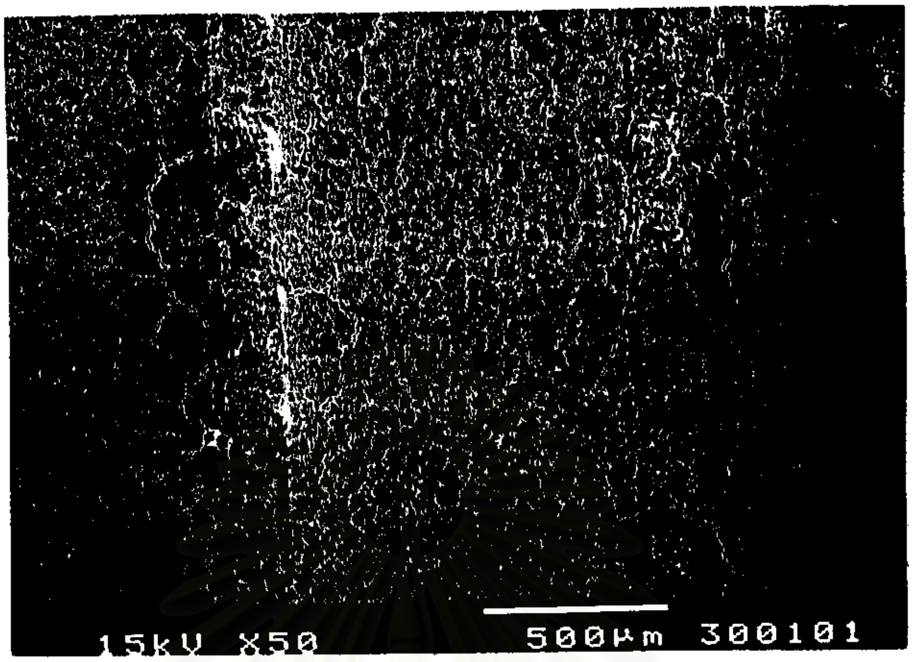


Figure 5.26 The surface of washcoat deposited on monolith for one time of washcoat dipping

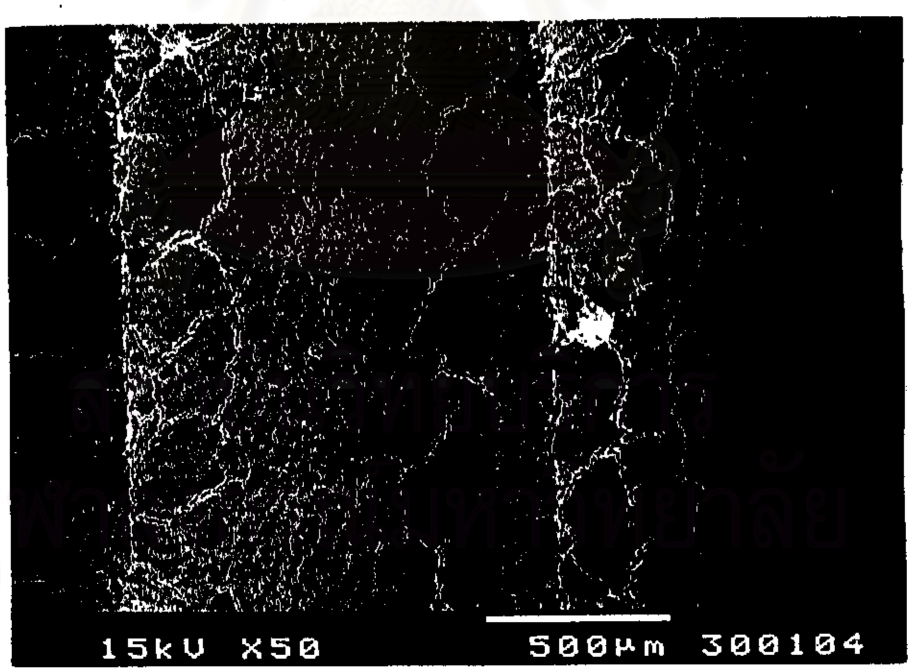


Figure 5.27 The surface of washcoat deposited on monolith for two times of washcoat dipping

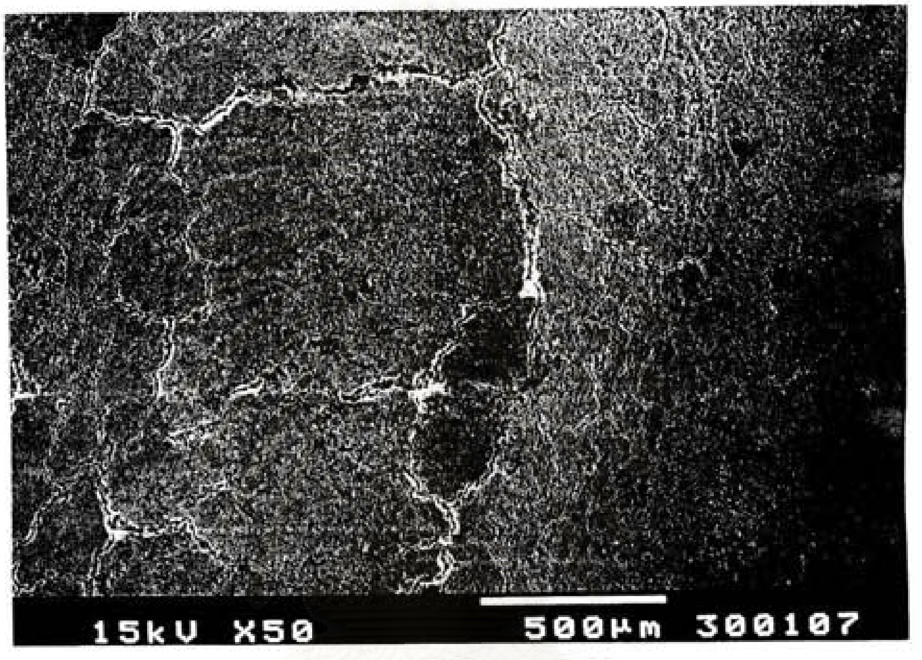


Figure 5.28 The surface of washcoat deposited on monolith for three times of washcoat dipping

5.3.2 Effect of holding time in calcination on washcoated monolith

In this part, monoliths which were dipped in washcoat slurry will be calcined at 500-600°C for 2-4 hr. and thermalshock resistance tested at 600 - 800 °C. The results are shown below:

5.3.2.1 Comparison the effect of holding time in calcination between 2 and 4 hr. for one times of coating

Figures 5.29 and 5.30 show the surface of washcoated monolith that was calcined at 500°C for 2 and 4 hr. and tested thermalshock resistance at 600°C., respectively.

For 2 hr. holding time in calcination step, the grainsizes are in the range of 20-100 μm. but for longer calcination time, 4 hr, the washcoat grains are about 5-20 μm. Note that all of the grains in figure 5.30 are almost in the same sizes and the grains of washcoat for both holding times had not released.

Figures 5.31 and 5.32 show the surface of washcoat deposited on monolith that were both calcined at 500°C for 2 and 4 hr. and thermalshock temperature at 800°C, respectively. The results agree with those observed from figures 5.29 and 5.30. Washcoat layers on the monoliths which were calcined for 4 hr. cracked to produce much smaller grains than the other calcined for 2 hr.

The washcoat grains of the sample tested at 600°C, figure 5.31, are in the range of 100-200 μm.

For the sample tested at 800°C, the washcoat on the monolith surface cracked into grainsizes of 10-20 μm. They were also much smaller grains than the other calcined for 2 hr.

It was noted that the longer the holding time of calcination the smaller the washcoat grainsize. This result can be observed for both thermalshock temperature at 600 and 800 °C and calcination temperature at 500 °C

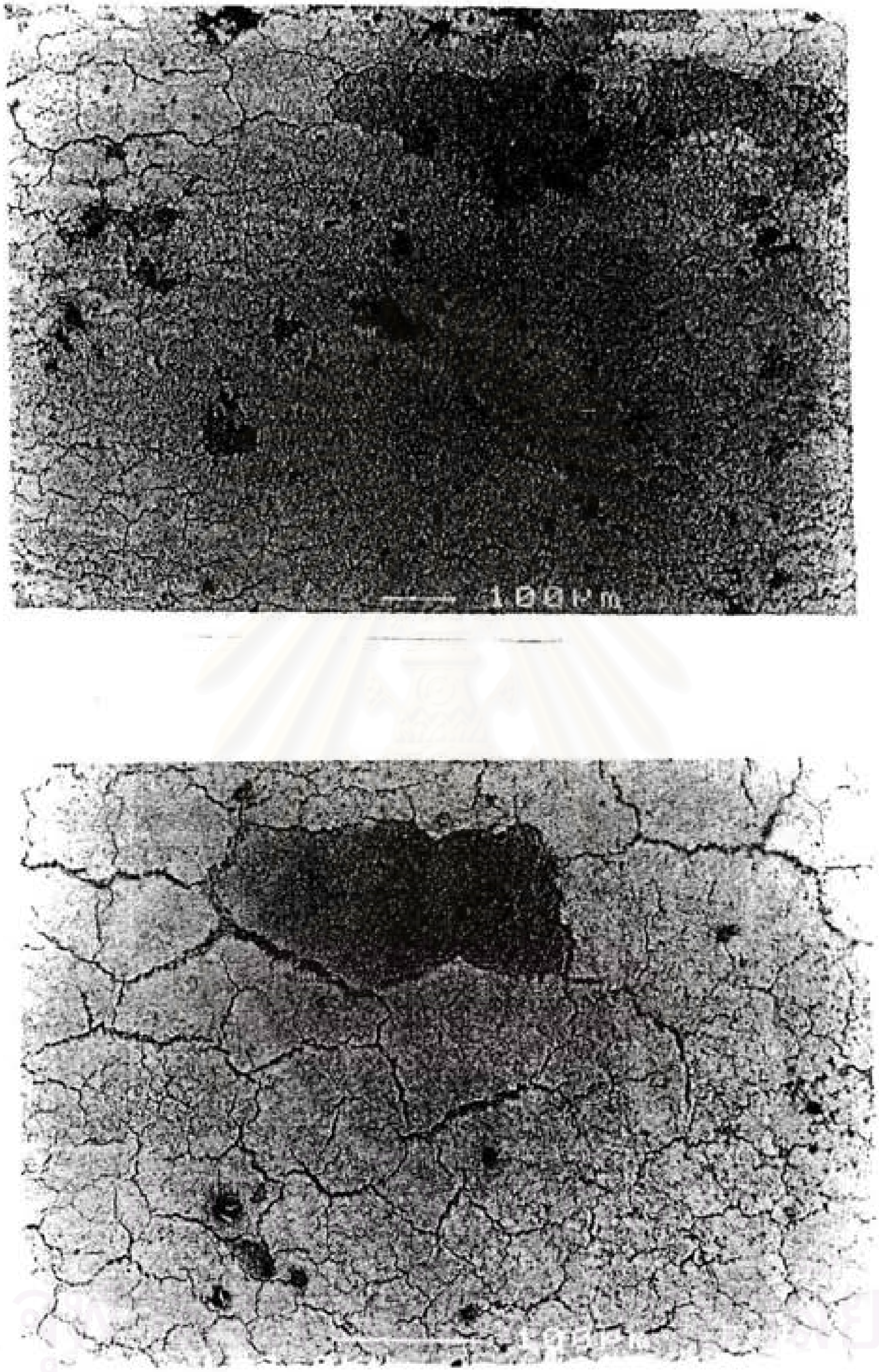


Figure 5.29 The surface of washcoat deposited on monolith dipped for one times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

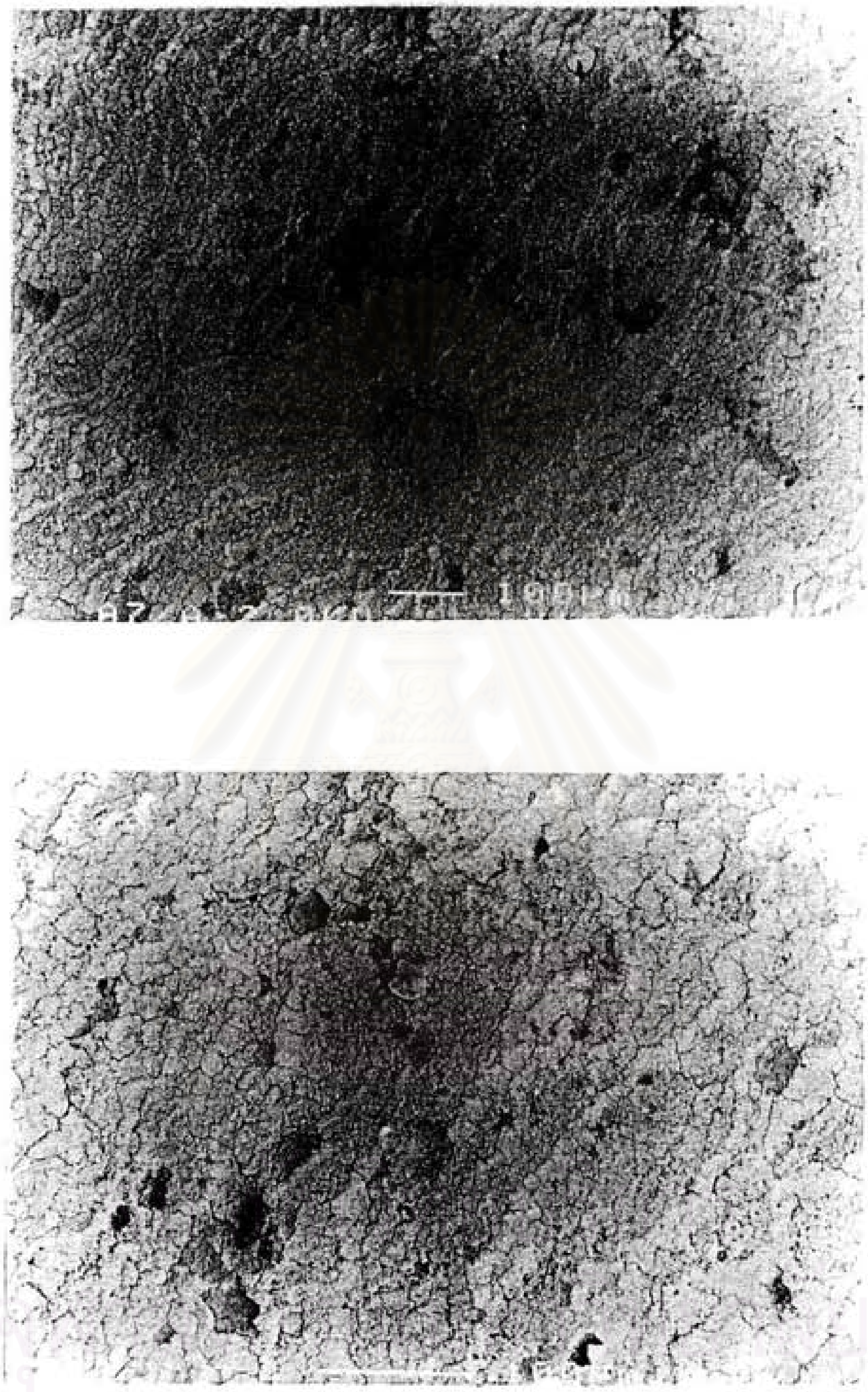


Figure 5.30 The surface of washcoat deposited on monolith dipped for one times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

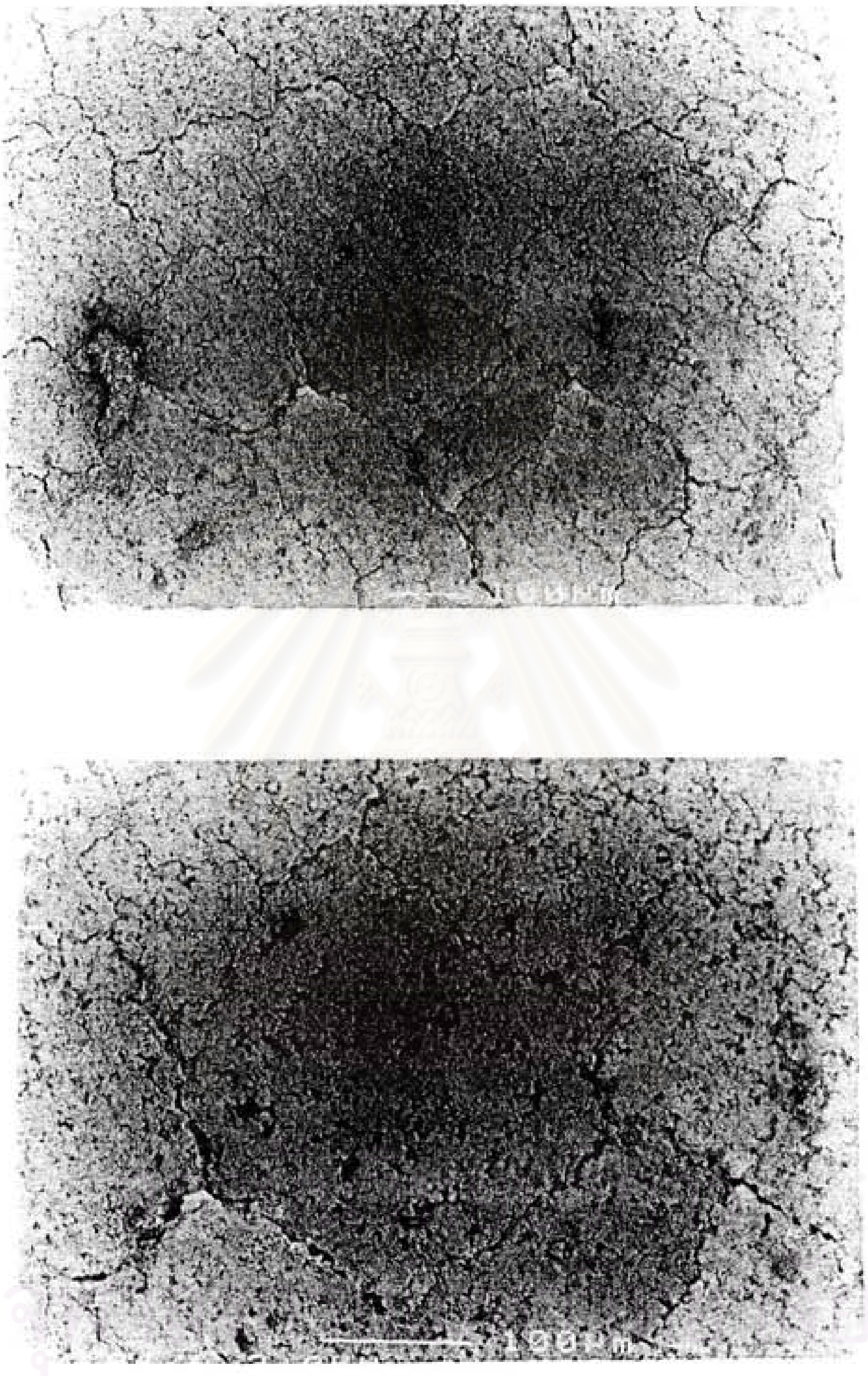


Figure 5.31 The surface of washcoat deposited on monolith dipped for one times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

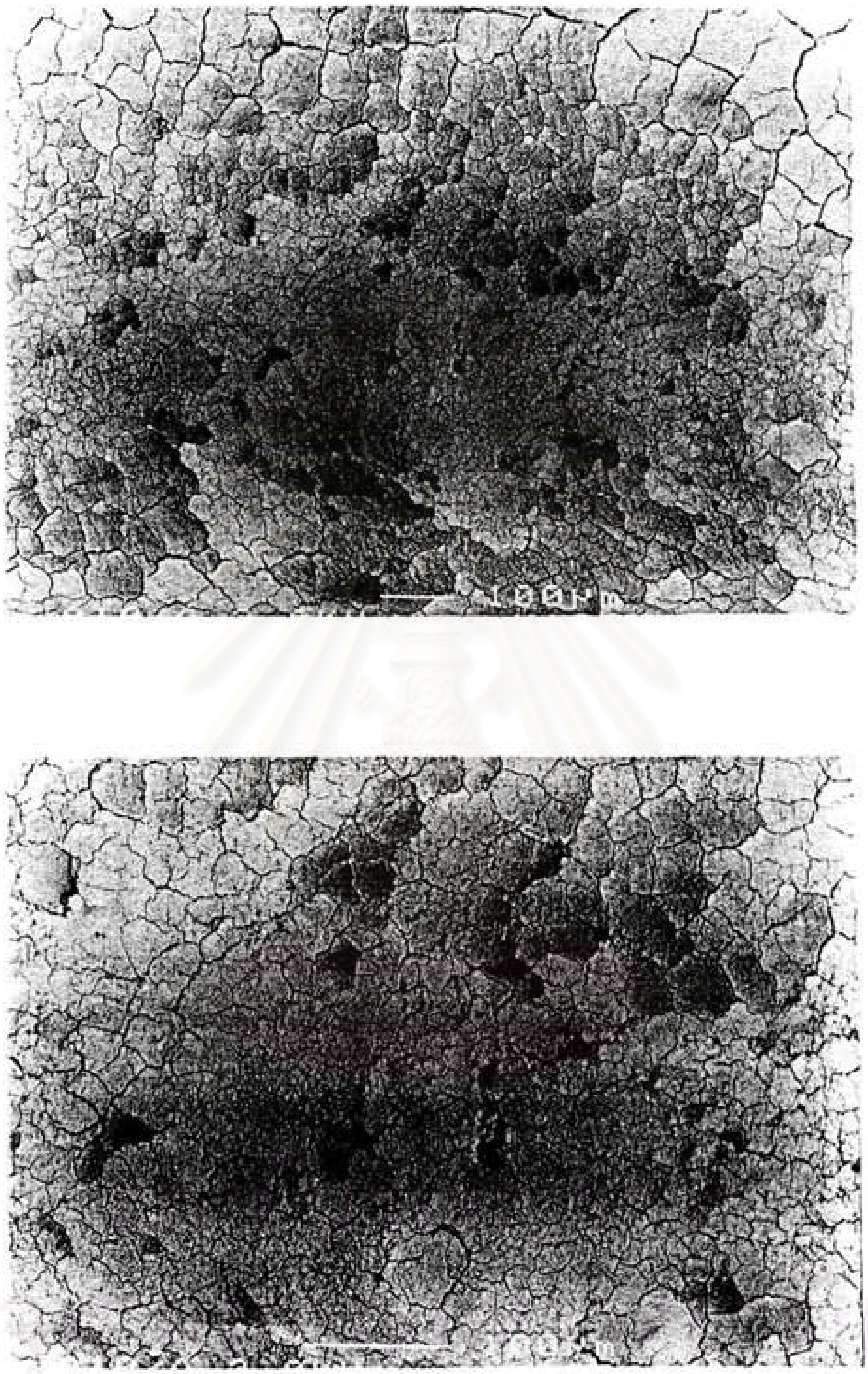


Figure 5.32 The surface of washcoat deposited on monolith dipped for one times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

5.3.2.2 Comparison the effect of holding time in calcination between 2 and 4 hr. for two times of washcoat dipping

The test samples used were bars of cordierite monolith, 0.5 cm × 0.5 cm × 3 cm. They were dipped in 40% alumina washcoat slurry for two times, then calcined at 500 °C for 2 and 4 hr. and tested thermalshock resistance at 600-800 °C

Figure 5.33 shows that the grains of washcoat deposited on monolith that was calcined at 500 °C for 2 hr. and thermalshock resistance tested at 600 °C has the grainsize about 100 µm. All of the grains stickily adhere on the monolith surface.

Figure 5.34 shows the surface of washcoat coated on monolith which was calcined at 500 °C for 4 hr. and tested thermalshock resistance at 600 °C. There were large cracked lines between grains. The grainsizes are about 100-200 µm.

Figure 5.35 shows the washcoat surface deposited on monolith that was calcined at 500 °C for 2 hr. and thermalshock temperature at 800 °C. The washcoat layer cracked into grainsizes about 100-200 µm.

Figure 5.36 shows the surface of washcoat on monolith that was calcined at 500 °C for 4 hr. and thermalshock resistance tested at 800 °C. It was observed that the washcoat cracked into large and small grains. Their sizes are about 50-200 µm.

For two times of washcoat coating, it can be concluded that the longer the calcination time the larger washcoat grains when test thermalshock resistance at 600 °C but for thermalshock temperature at 800 °C, the result was directly inverse. The effect of washcoat grainsizes will be studied later in the abrasive strength of the washcoated monolith.

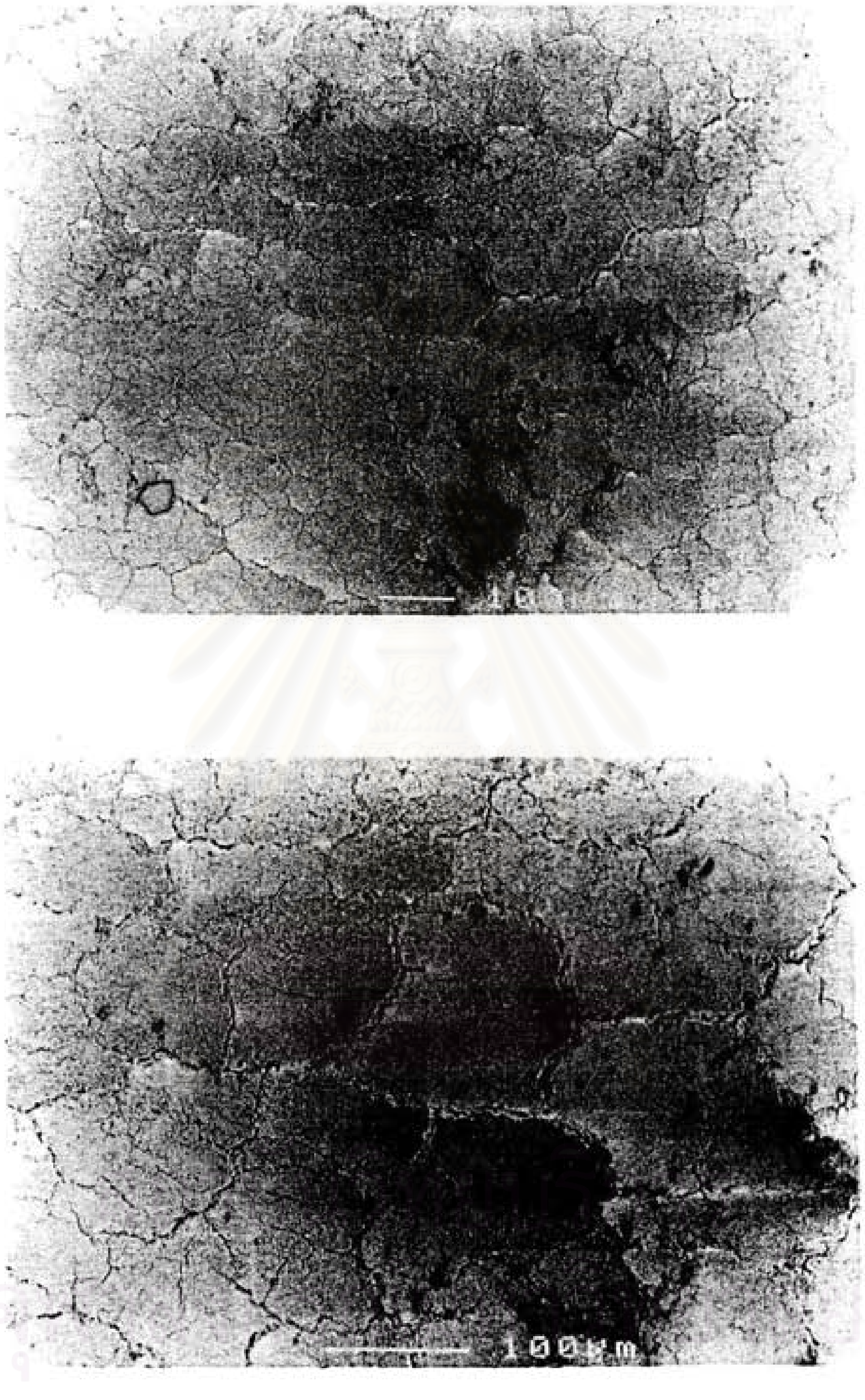


Figure 5.33 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

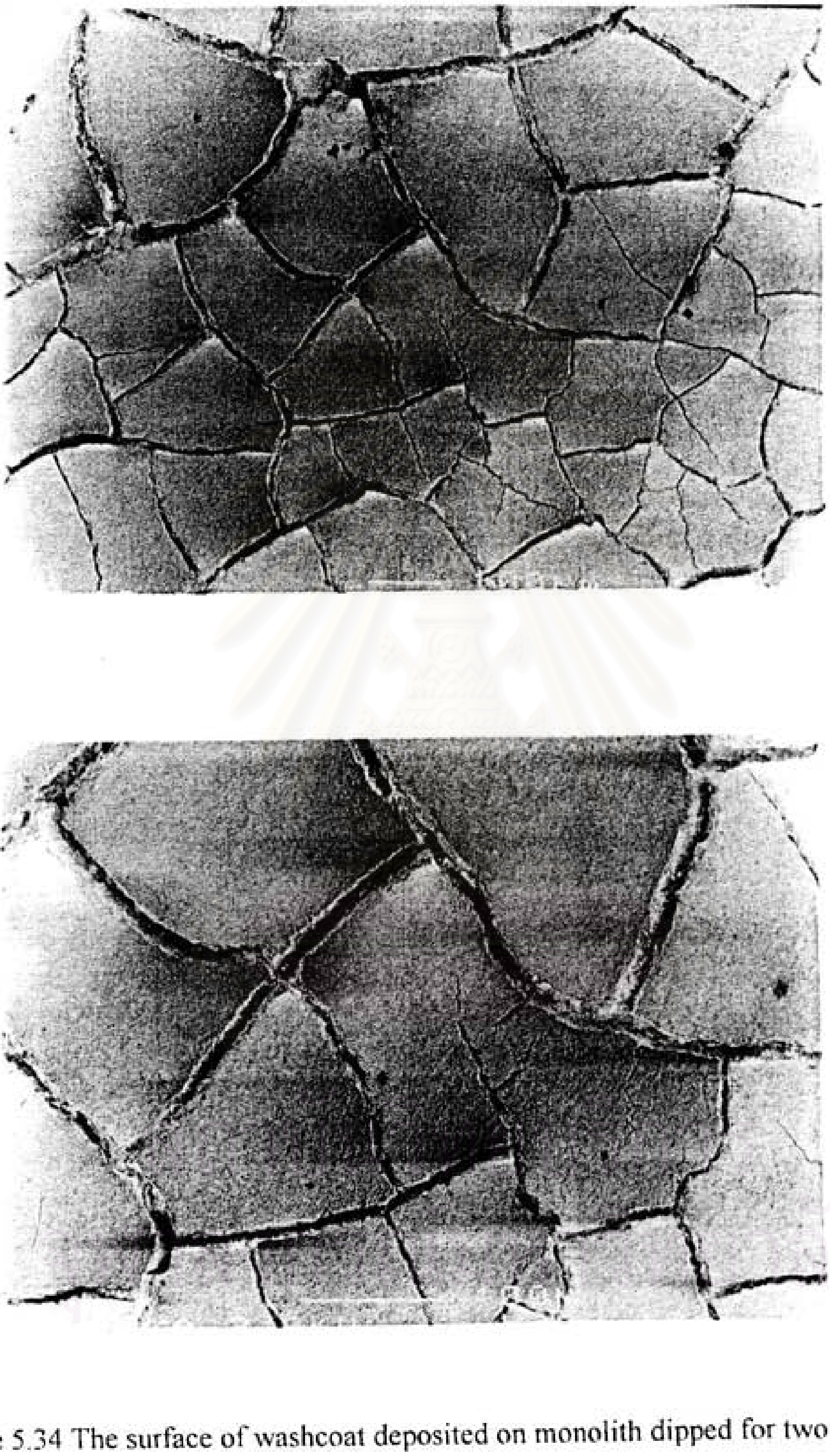


Figure 5.34 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 600°C

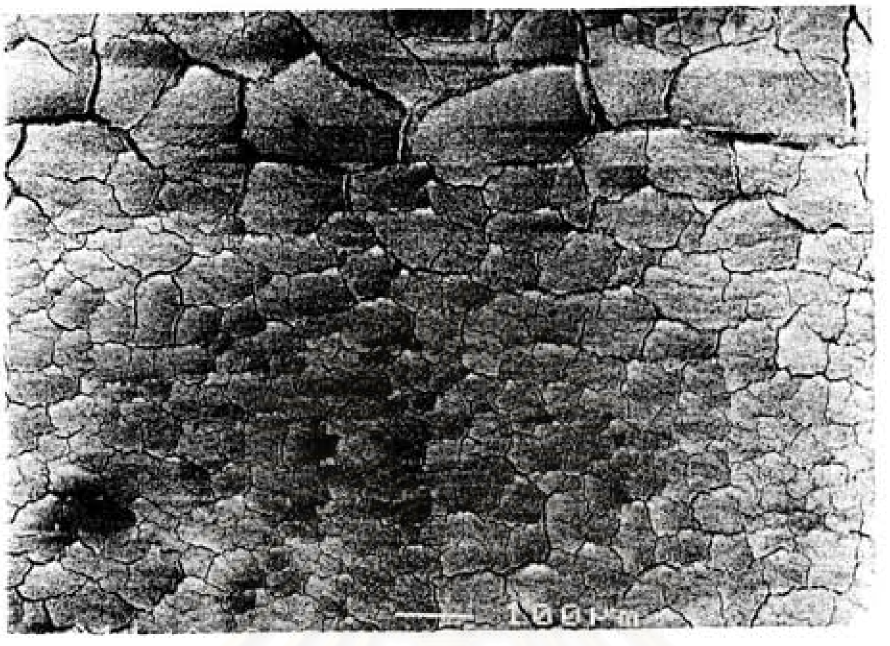
Top : Magnification 100 times

Bottom : Magnification 200 times

Figure 5.35 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times



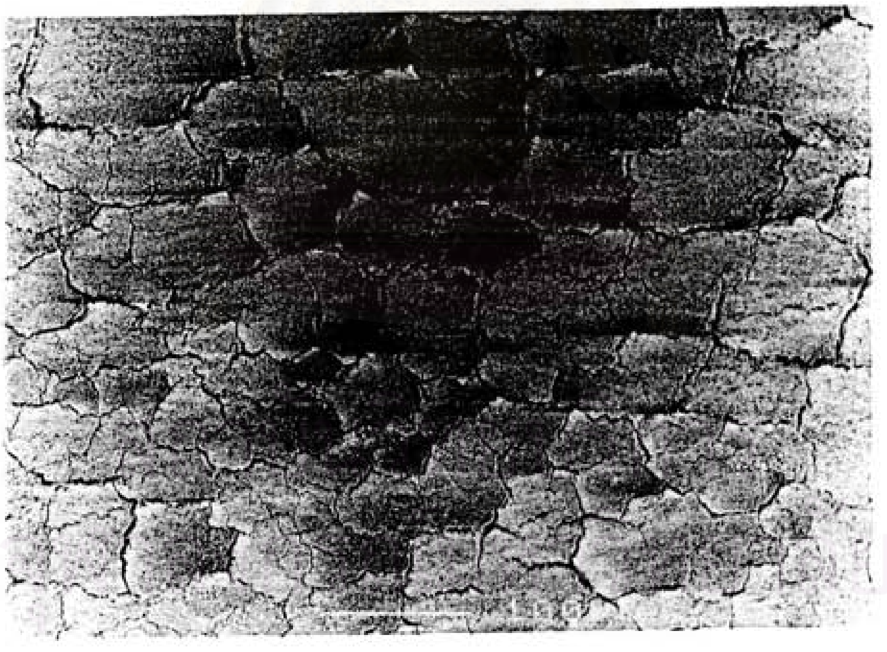


Figure 5.36 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

5.3.2.3 Comparison the effect of holding time in calcination between 2 and 4 hr. for three times of washcoat dipping

This experiment involves an investigation of the monolith that was dipped in 40% alumina washcoat slurry for three times, calcined at 500 °C for 2-4 hr. and tested thermalshock resistance at 600 - 800 °C

Figure 5.37 shows the surface of washcoat coated on a monolith which was calcined at 500 °C for 2 hr. and thermalshock tested at 600 °C. It was found that the washcoat grains are small and the grainsizes are in the range of 10-50 μm. The washcoat grains stickily adhered on the monolith surface.

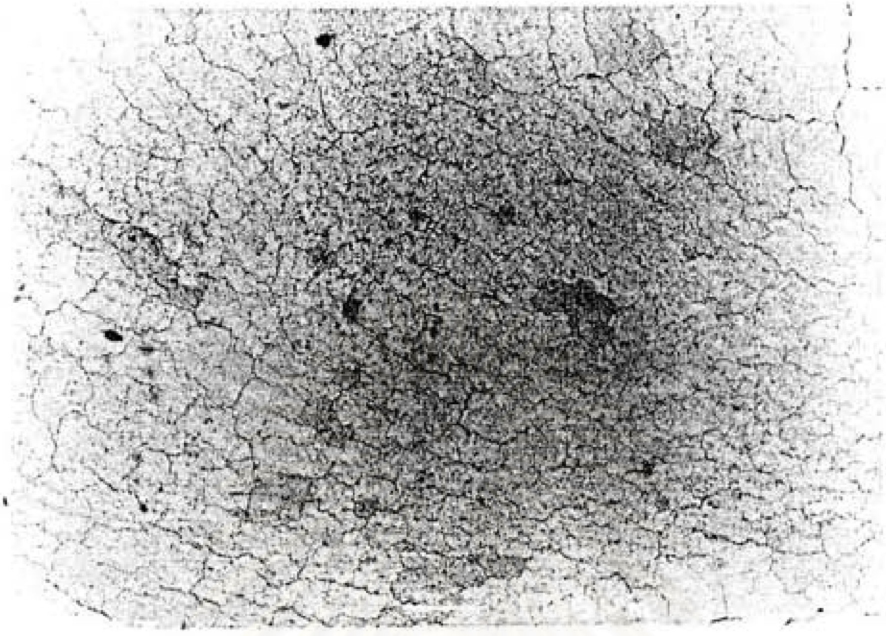
Figure 5.38 shows the alumina washcoat deposited on a monolith that was calcined at 500 °C for 4 hr. and tested thermalshock resistance at 600 °C. It can be seen clearly that some of washcoat grains released from the monolith surface.

Figure 5.39 shows that the washcoat on a monolith which was calcined at 500 °C for 2 hr. and thermalshock resistance tested at 800 °C cracked into large grains. Their size are about 200 μm.

Figure 5.40 shows the surface of washcoat on a monolith that was calcined at 500 °C for 4 hr. and tested thermalshock resistance at 800 °C. It was observed that the washcoat surface was not smooth because some of the washcoat grains came off the monolith surface.

A possible reason is that the washcoat layer was too thick when the monolith was coated in washcoat slurry for three times. Not only that, the thermalshock resistance testing at high temperature, 800 °C, caused the washcoat cracked into small grains. Thus, the grains slip off the monolith surface.

It can be concluded that the long calcination time will cause the less stability of washcoat deposited on the monolith.



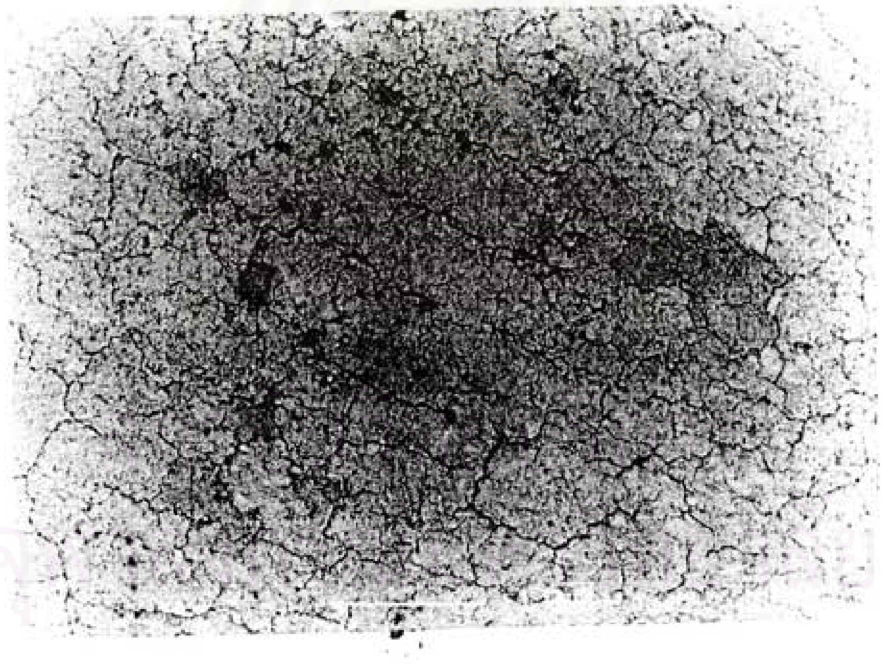


Figure 5.37 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

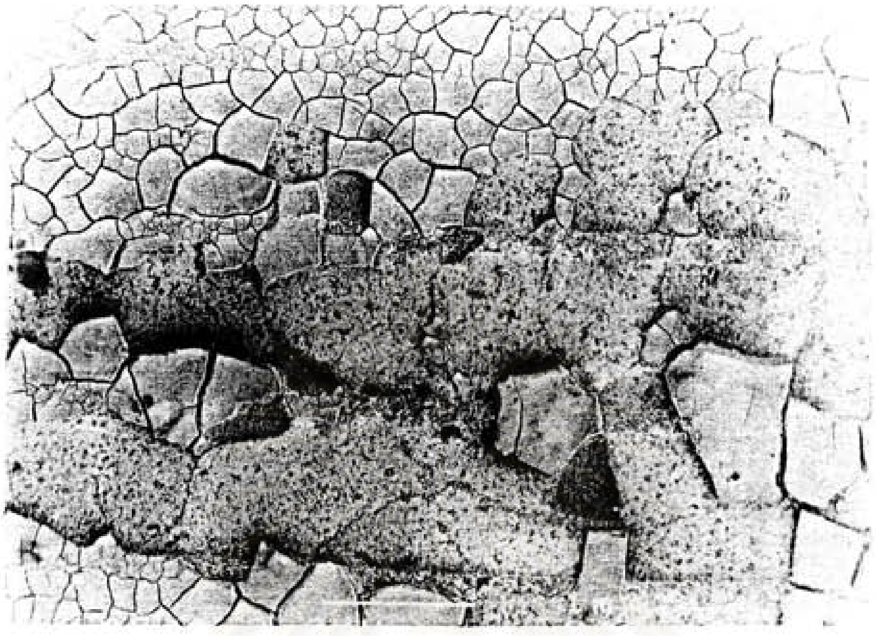


Figure 5.38 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 600°C

Top : Magnification 20 times

Bottom : Magnification 20 times

Figure5.39 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

Figure 5.40 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 4 hr.and tested thermalshock resistance at 800°C

Top : Magnification 20 times

Bottom : Magnification 20 times

5.3.3 Effect of calcination temperature on washcoated monolith

Monoliths were coated with alumina washcoat then calcined at 500-600 °C for 2 hr. and tested thermalshock resistance at 600 - 800 °C

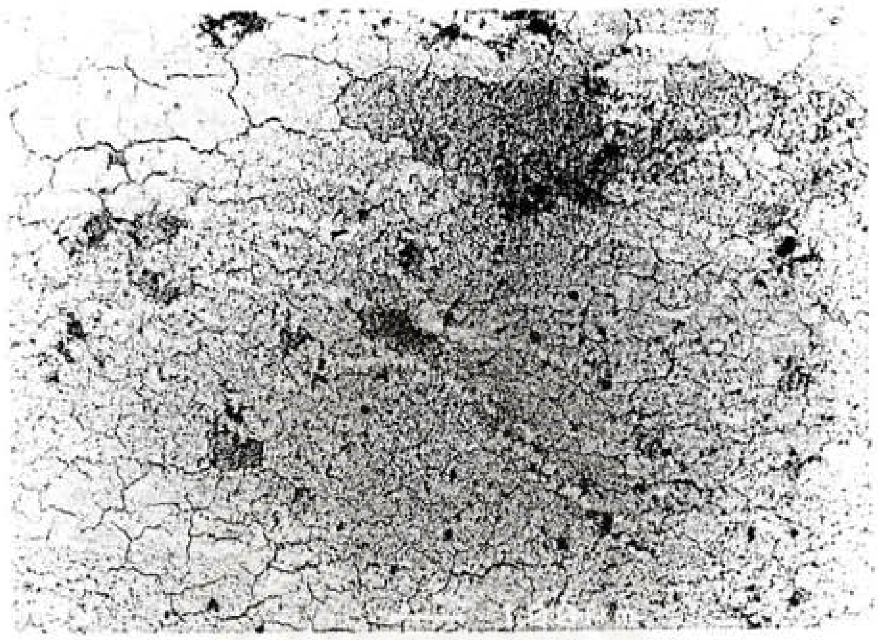
5.3.3.1 Comparison the effect of calcination temperature between 500 and 600 °C for one time of washcoat coating

Figures 5.41 and 5.42 show the washcoat surface on monoliths that were calcined at 500 and 600 °C for 2 hr. and tested the thermalshock resistance at 600 °C, respectively. The results are clearly different between the monoliths calcined at 500 and 600 °C that the washcoat deposited on the monolith which was calcined at 500 °C cracked into small grains in the range of 10-100 μm. and the washcoat stickily adhered on the monolith surface. But, there were some large washcoat grains and some of the grains released from the monolith when calcined the washcoated monolith at 600 °C.

Figures 5.43 and 5.44 show the surface of washcoat on monoliths that were calcined at 500 and 600 °C for 2 hr. and tested the thermalshock resistance at 800 °C, respectively. There is a similar trend as the above results that the washcoated monolith that was calcined at 500 °C cracked into large grains about 200 μm. and the narrow cracked lines separated between the washcoat grains. The grains still sticked on the monolith surface.

For the calcination temperature at 600 °C, there were large cracked lines between each grains. The grains were larger than the washcoat grain that was calcined at 500 °C. Their sizes are in the range of 200 - 500 μm. Some of the washcoat grains slip off the monolith surface.

From the above results, it can be concluded that the calcination temperature at 500°C is appropiate for the washcoated monolith. This is because the washcoat grains can adhere to the monolith surface and the surface of washcoat layer is smooth.



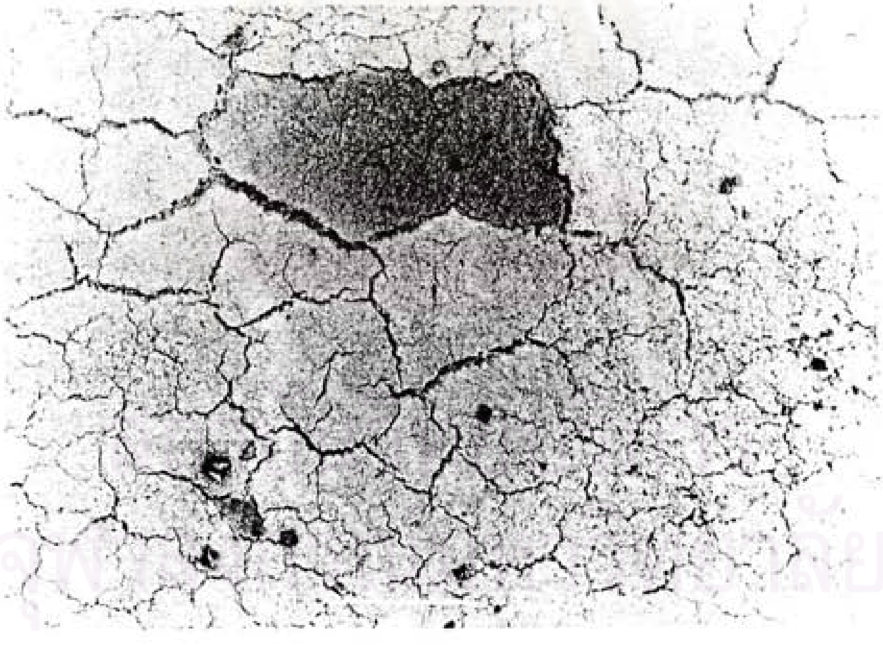


Figure 5.41 The surface of washcoat deposited on monolith dipped for one times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

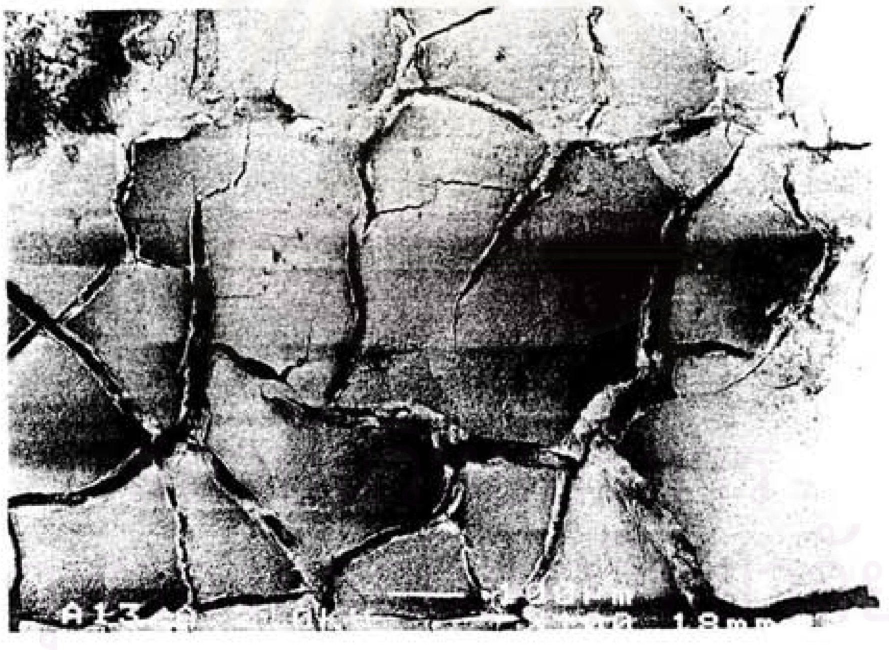
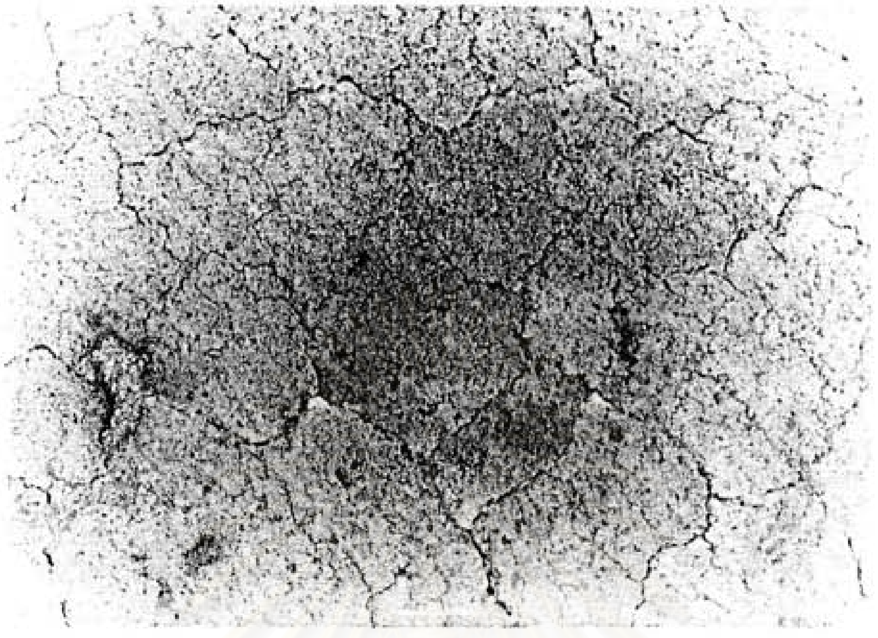


Figure 5.42 The surface of washcoat deposited on monolith dipped for one time, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 50 times

Bottom : Magnification 100 times



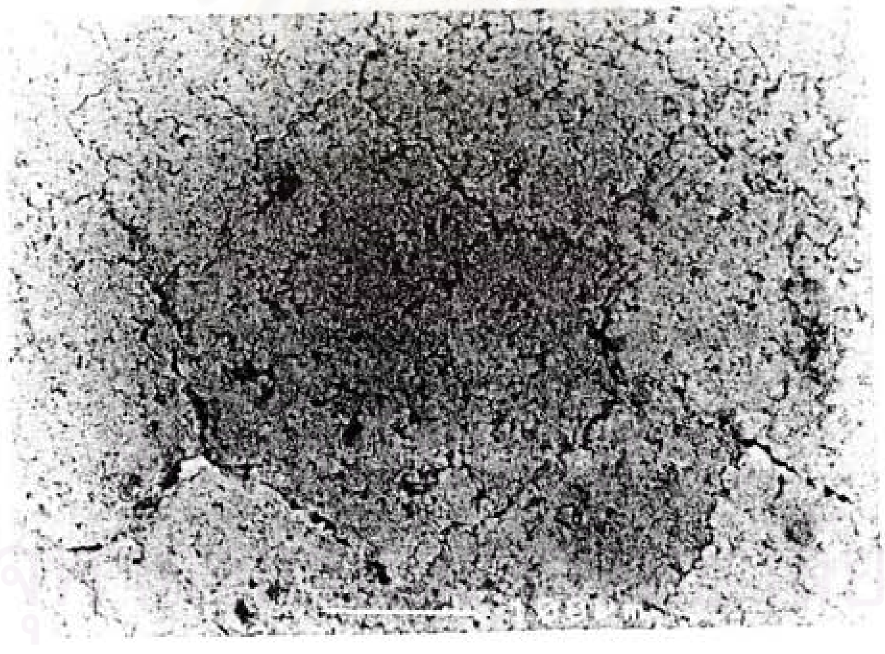


Figure 5.43 The surface of washcoat deposited on monolith dipped for one time, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

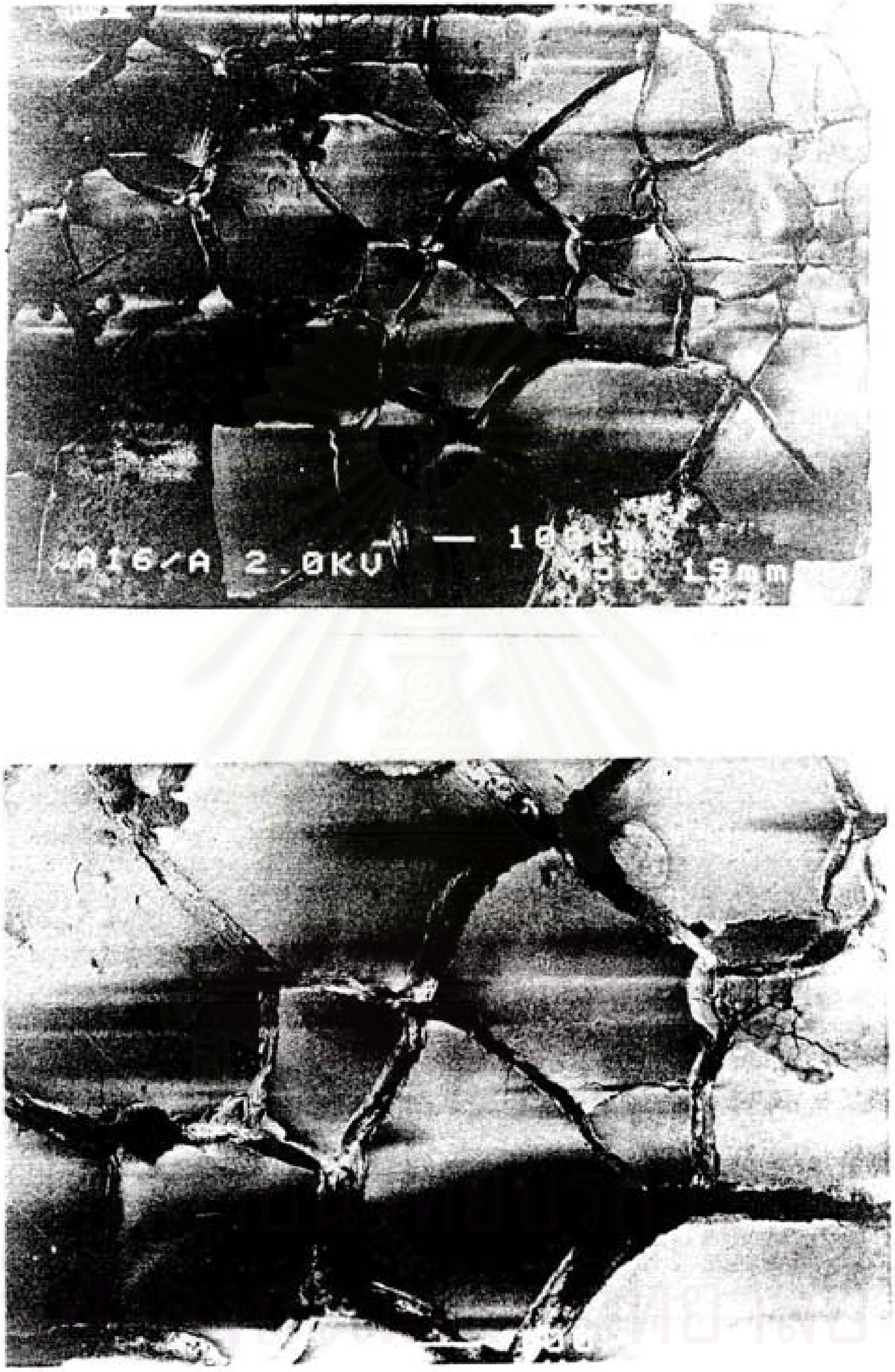


Figure 5.44 The surface of washcoat deposited on monolith dipped for one time, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 50 times

Bottom : Magnification 100 times

5.3.3.2 Comparison the effect of calcination temperature between 500 and 600 °C for two times of washcoat coating

Figures 5.45 and 5.46 show the surface of washcoat on monoliths that were calcined at 500 and 600 °C for 2 hr.and tested thermalshock resistance at 600 °C, respectively.

The similar trend as surface coating in washcoat slurry for one time was observed in figures 5.45 and 5.46.

For calcination temperature at 500 °C, the washcoat grains are about 100 μm. and there was no grain released from the monolith surface.

For calcination temperature at 600 °C, there were several small grains of washcoat about 10-50 μm.

Figures 5.47 and 5.48 show the washcoat surface on monoliths that were calined at 500 and 600 °C for 2 hr. and thermalshock resistance tested at 800 °C, respectively. The washcoat deposited on the monolith which was calcined at 500 °C cracked into grainsize about 200 μm.

It was found that some of the washcoat grains released from the monolith that was calcined at 600 °C. Therefore, the calcination temperature at 500°C result to the denser and smoother washcoat layer on the monolith than at 600°C.

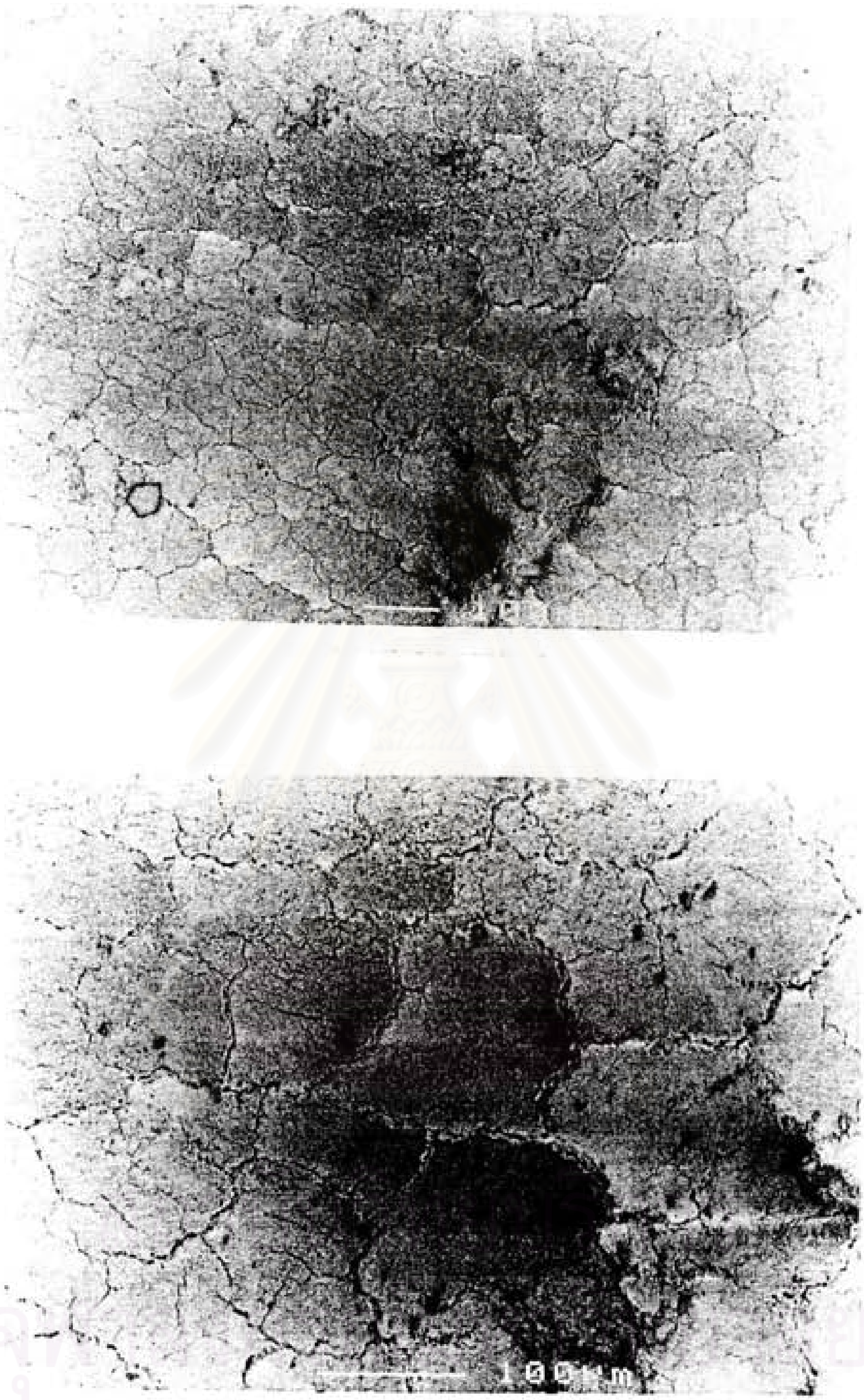


Figure 5.45 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 200 times

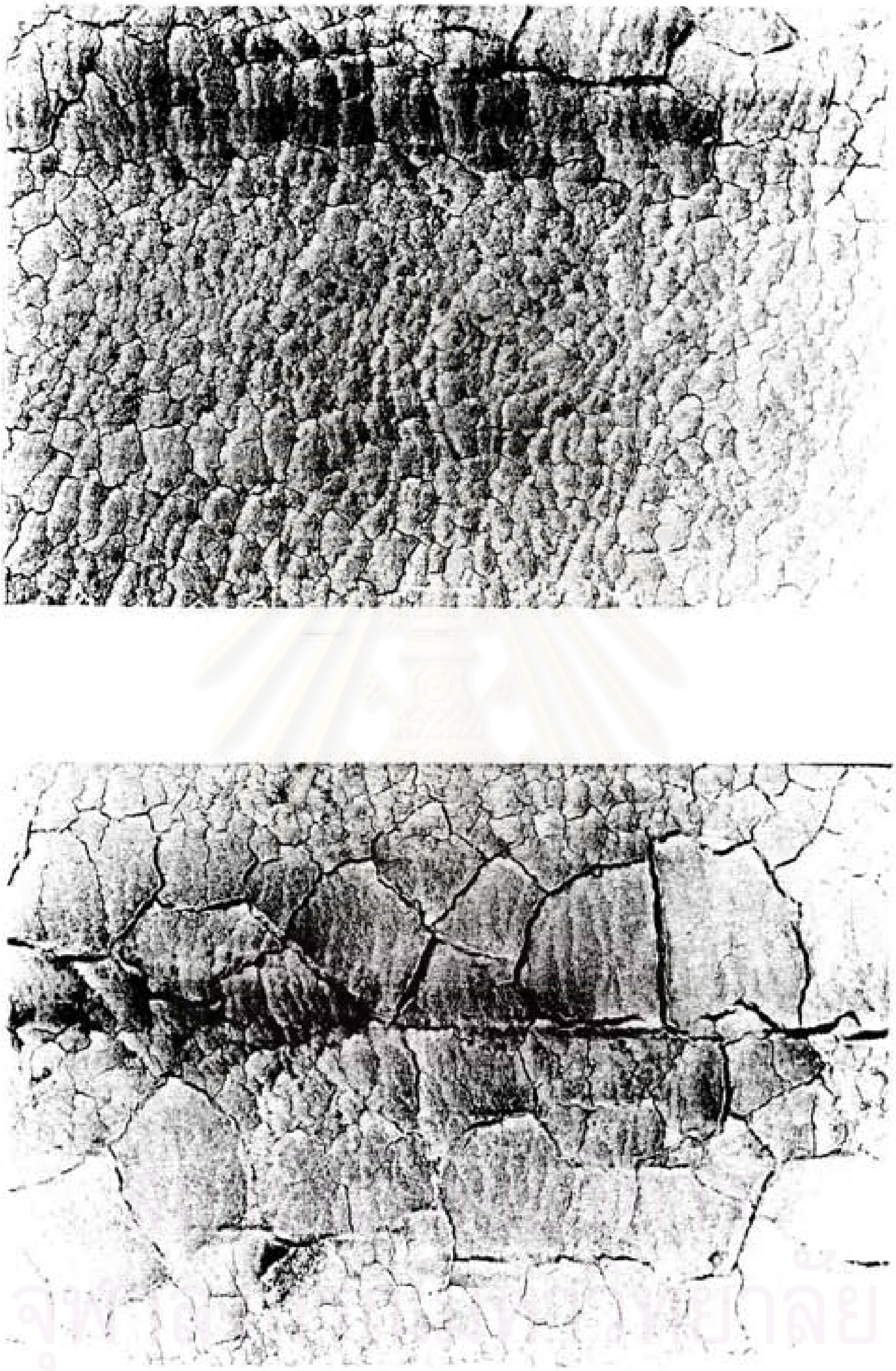
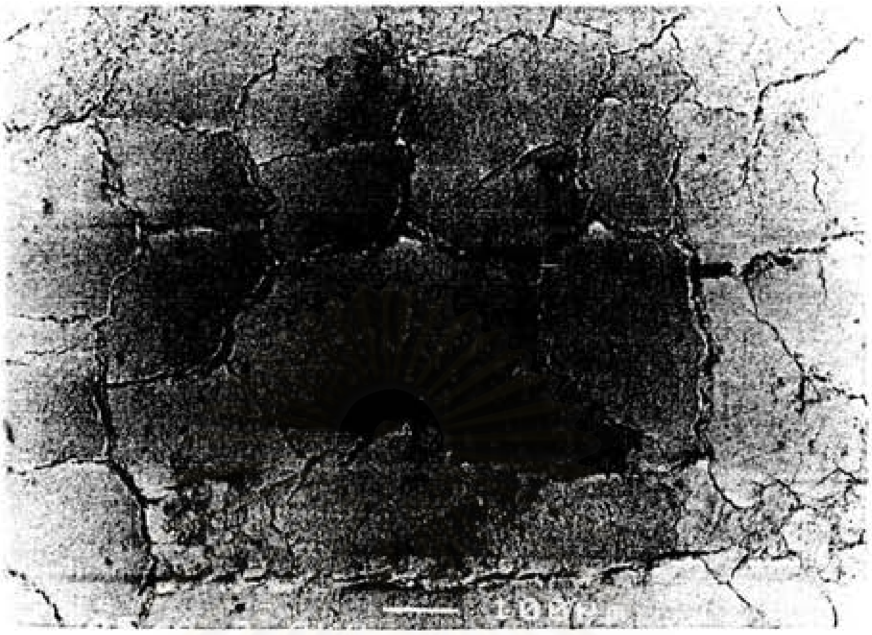


Figure 5.46 The surface of washcoat deposited on monolith dipped for two times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom : Magnification 100 times



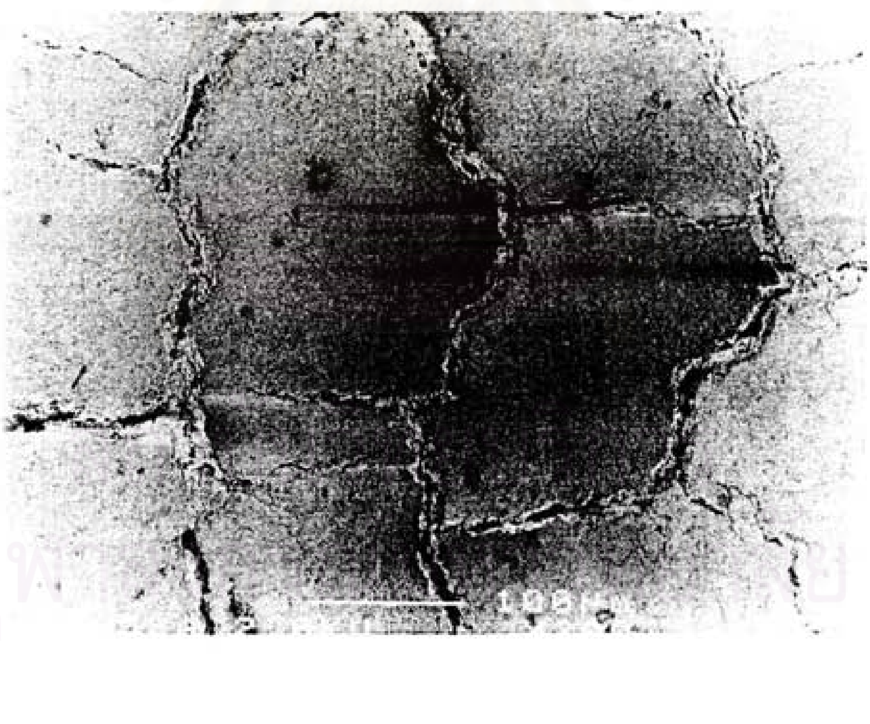


Figure 5.47 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times

Figure 5.48 The surface of washcoat deposited on monolith dipped for two times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 20 times

Bottom: Magnification 100 times

5.3.3.3 Comparison the effect of calcination temperature between 500 and 600 °C for three times of washcoat coating

The similar trend as above experimental results can be observed on monoliths that were dipped in alumina washcoat slurry for three times which was shown in figures 5.49, 5.50, 5.51 and 5.52.

Figures 5.49 and 5.50 show the washcoat surface on the monolith that were calcined at 500 and 600 °C for 2 hr and thermalshock resistance tested at 600 °C, respectively.

Figures 5.51 and 5.52 show the washcoat on the monolith that were calcined at 500 and 600 °C for 2 hr and thermalshock resistance tested at 800 °C, respectively.

It was found that the higher the calcination temperature, 600 °C, caused the washcoat cracked into small grains and will released from the monolith surface. Hence, the calcination temperature at 500°C is an appropriate temperature .

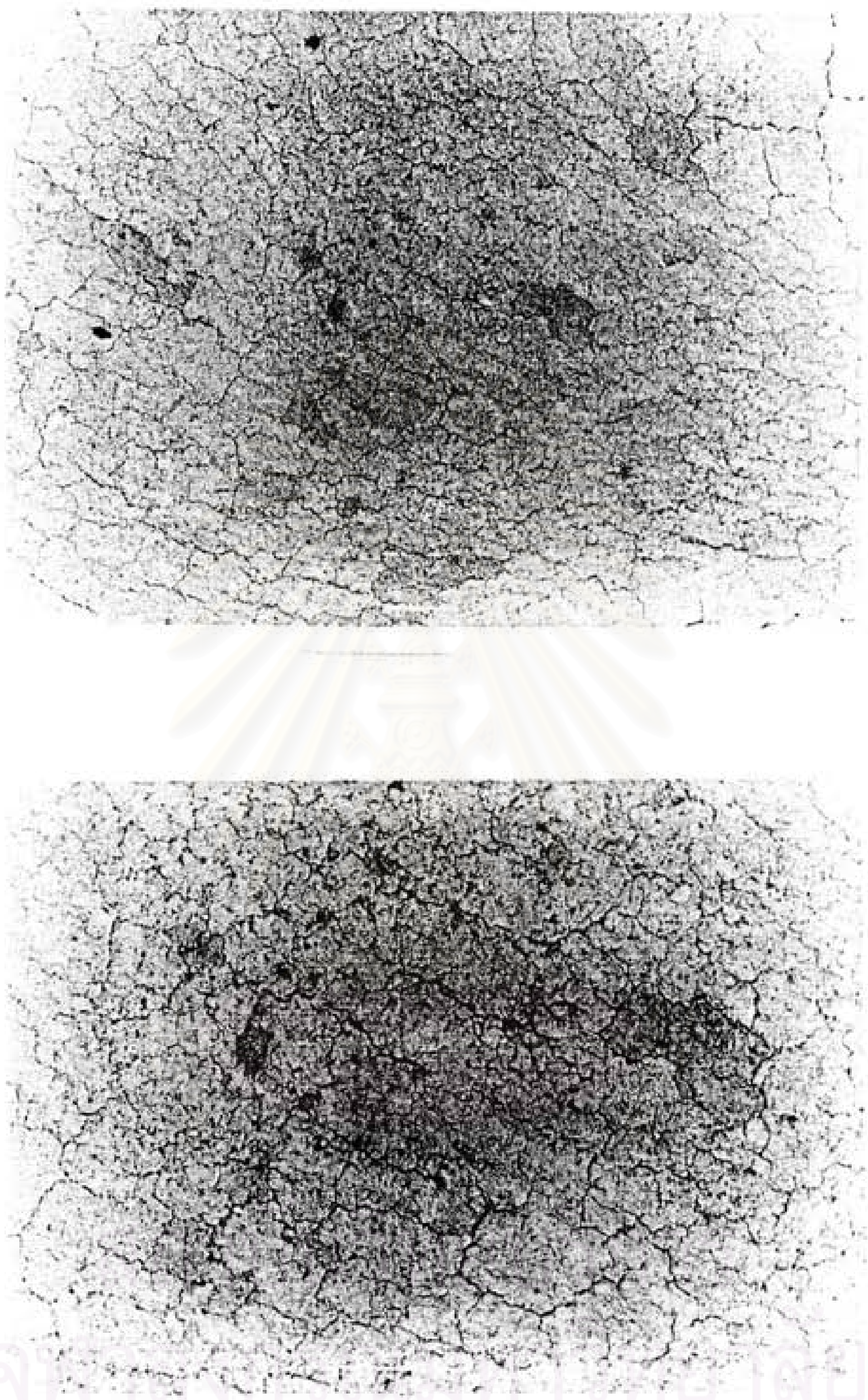


Figure 5.49 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom: Magnification 200 times

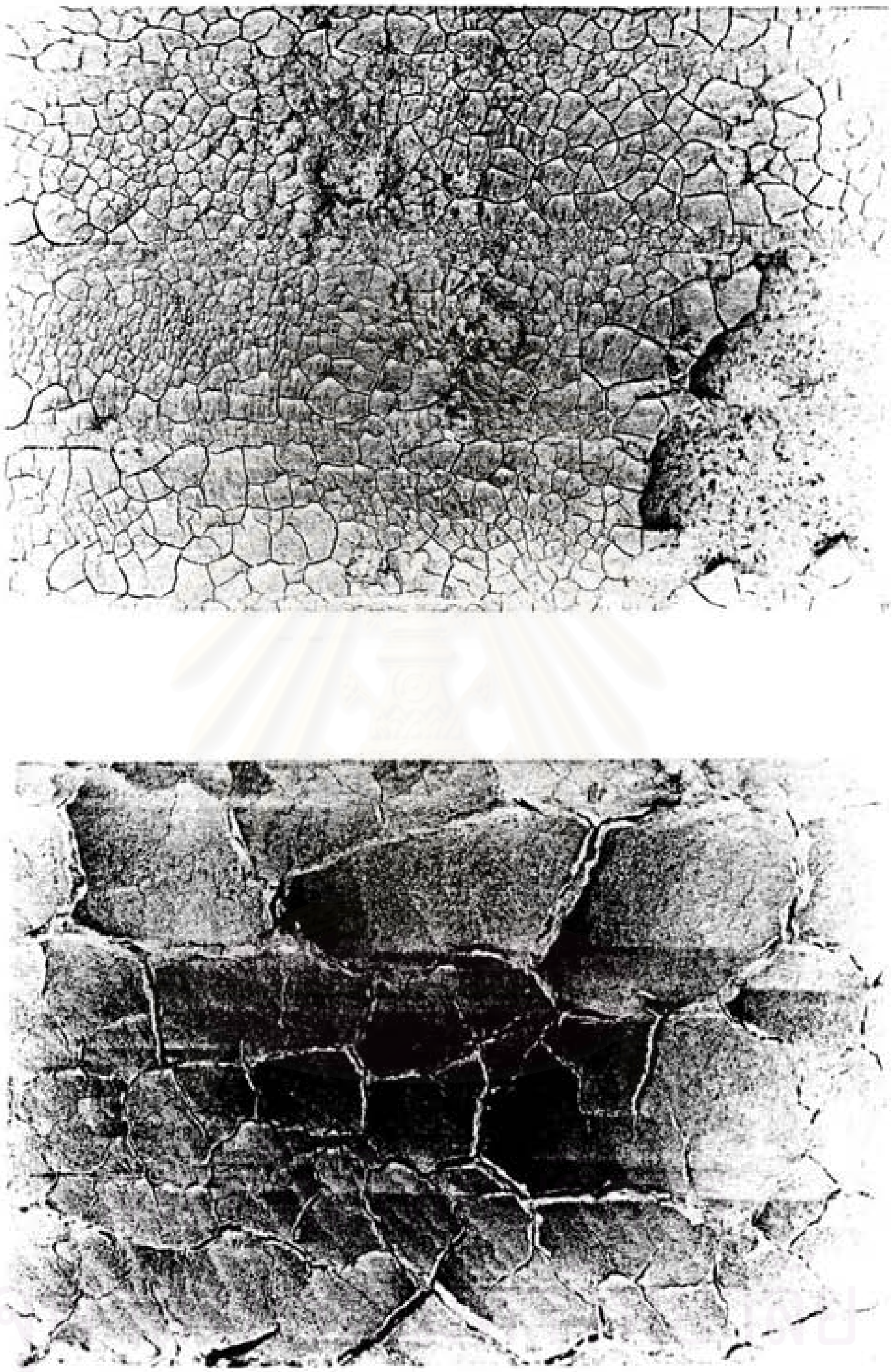


Figure 5.50 The surface of washcoat deposited on monolith dipped for three times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

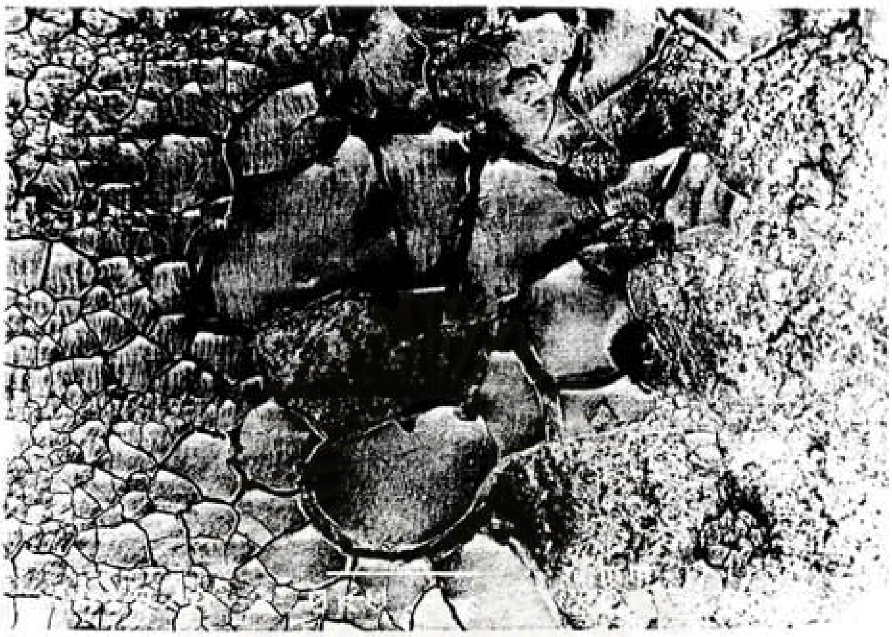
Top : Magnification 20 times

Bottom : Magnification 100 times

Figure 5.51 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom : Magnification 200 times



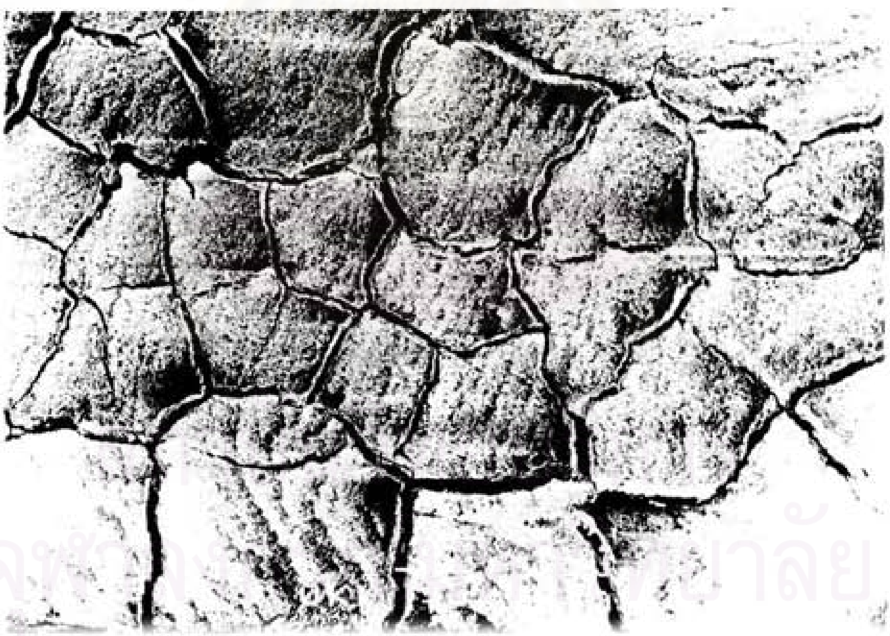


Figure 5.52 The surface of washcoat deposited on monolith dipped for three times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 50 times

Bottom : Magnification 100 times

5.3.4 Effect of thermalshock temperature on washcoated monolith

The monoliths were coated with alumina washcoat, then calcined at 500 - 600°C for 2 hr. and tested thermalshock resistance at 600-800 °C. The washcoat deposited on the monoliths were observed.

5.3.4.1 Comparison the effect of thermalshock temperature between 600 - 800 °C for one time of washcoat coating

Figures 5.53 and 5.54 show the surface of washcoat on monoliths that were calcined at 500°C for 2 hr. and thermalshock resistance tested at 600 and 800°C, respectively. It was found that the washcoat cracked to the small grains about 20-100 μm. for thermalshock temperature at 600°C. There was no washcoat grain released from the monolith surface.

For thermalshock temperature at 800°C, the washcoat cracked to large grain, 100 - 200 μm., when compared with figure 5.53 but the washcoat grains still adhere on the monolith surface.

Figures 5.55 and 5.56 show the washcoat surface deposited on monoliths that were calcined at 600 °C for 2 hr and thermalshock resistance tested at 600 and 800°C, respectively. The similar trend can be seen on the washcoat surface which shows that the washcoat cracked to small grains, 50-100 μm., at 600°C thermalshock temperature. However, at 800°C thermalshock temperature, the washcoat grains were 200-500 μm. and there were large crack lines between each grain. Some of the washcoat grains released from the monolith surface.

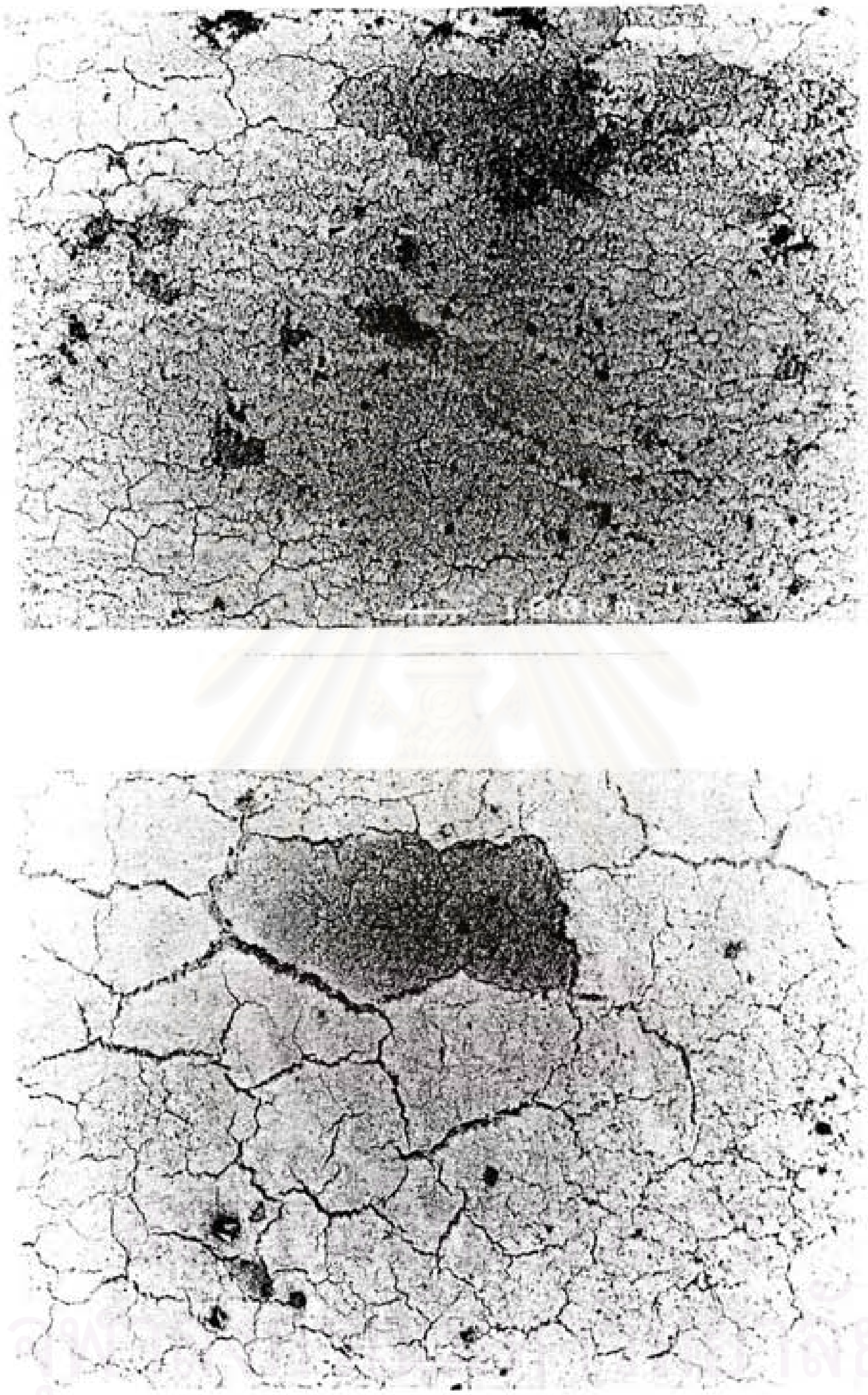


Figure 5.53 The surface of washcoat deposited on monolith dipped for one time, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom: Magnification 200 times

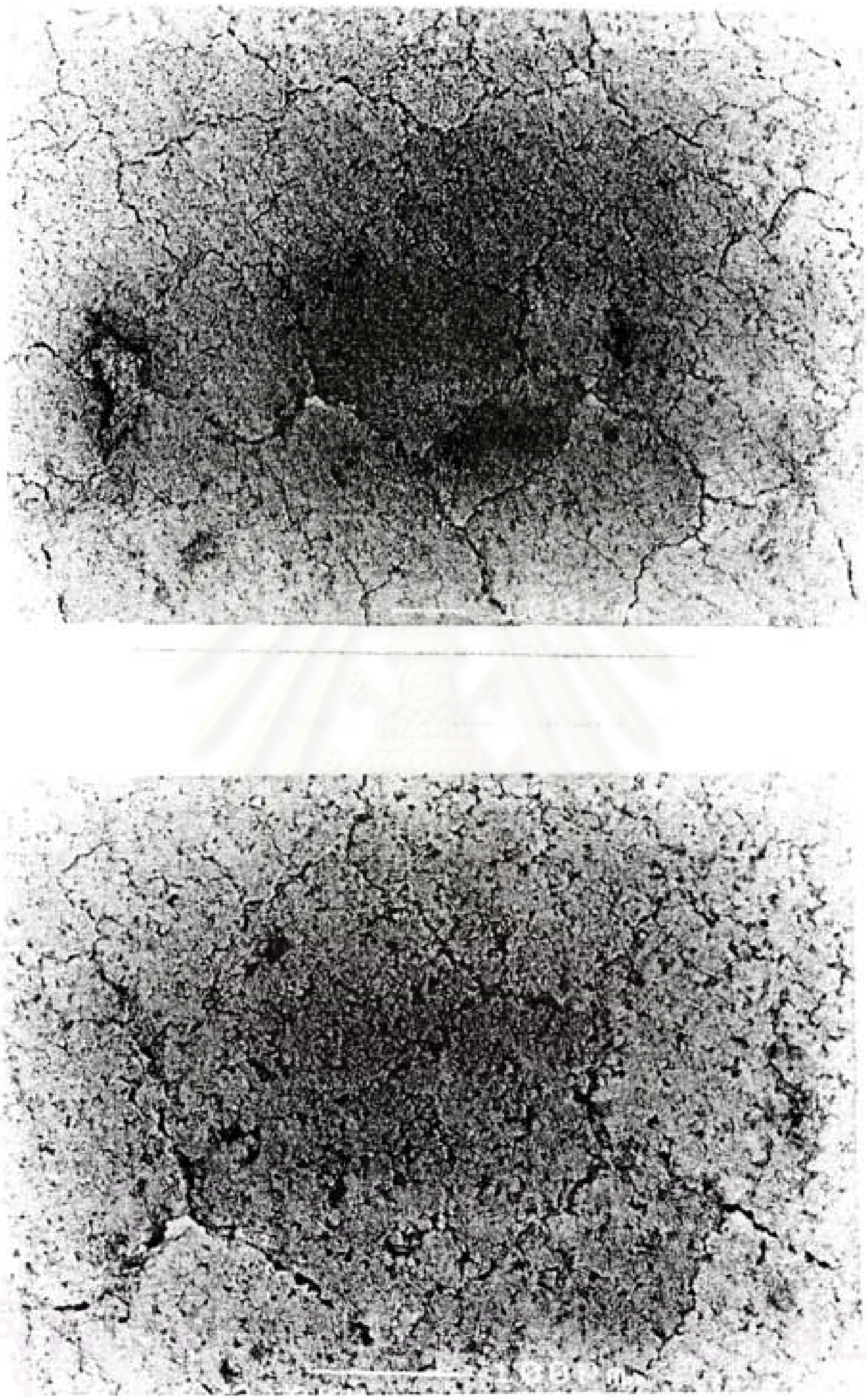


Figure 5.54 The surface of washcoat deposited on monolith dipped for one time, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom: Magnification 200 times

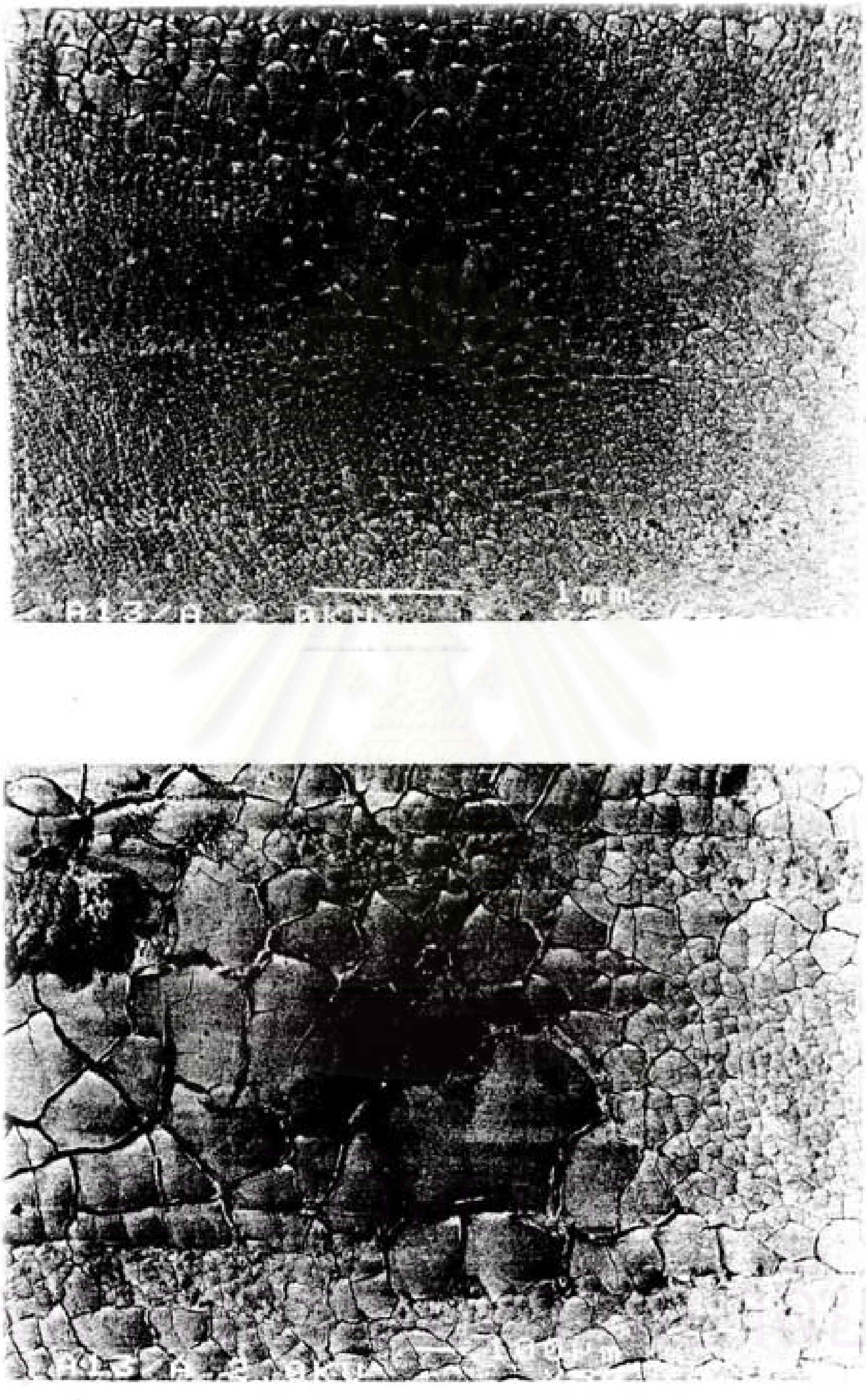


Figure 5.55 The surface of washcoat deposited on monolith dipped for one time, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 20 times

Bottom: Magnification 50 times

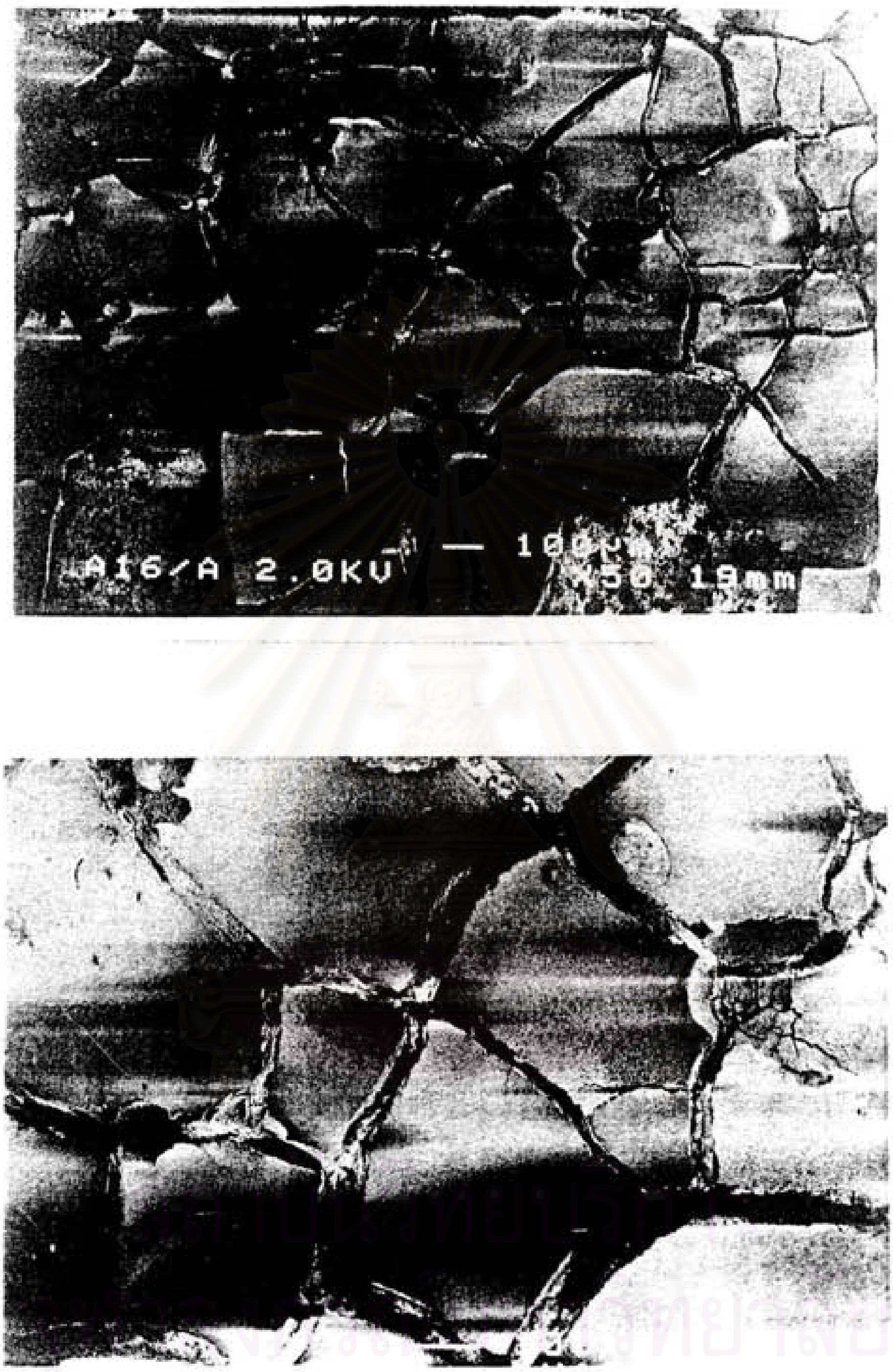


Figure 5.56 The surface of washcoat deposited on monolith dipped for one time, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 50 times

Bottom: Magnification 100 times

5.3.4.2 Comparison the effect of thermalshock temperature between 600 - 800 °C for two times of washcoat coating

Figures 5.57 and 5.58 show the washcoat surface deposited on monolith that were calcined at 500°C for 2 hr and thermalshock resistance tested at 600 and 800°C, respectively. There was a difference on the washcoat surface between the samples which were tested thermalshock resistance at 600 and 800°C that, for 600°C thermalshock temperature, the washcoat grainsize was about 100 μm. The grains sticked on the monolith surface. At 800°C thermalshock temperature, the washcoat cracked to the large grains about 100-200 μm. Each grain was separated by large crack lines.

Figures 5.59 and 5.60 show the surface of washcoat on monolith that were calcined at 600 °C for 2 hr. and thermalshock resistance tested at 600 and 800°C, respectively. It was observed at 600°C thermalshock temperature that the washcoat cracked to the grainsizes about 10-50 μm. There was no washcoat grain slip off the monolith surface. For 800°C thermalshock temperature, some of the washcoat grains released from the monolith and the washcoat grainsize were about 200-400 μm.

From the above results, it may be explained that the high thermalshock temperature, 800°C, caused the washcoat cracked to large grains and the edge of each grain was divided by the large cracked line, then the washcoat grains were released from the monolith surface.

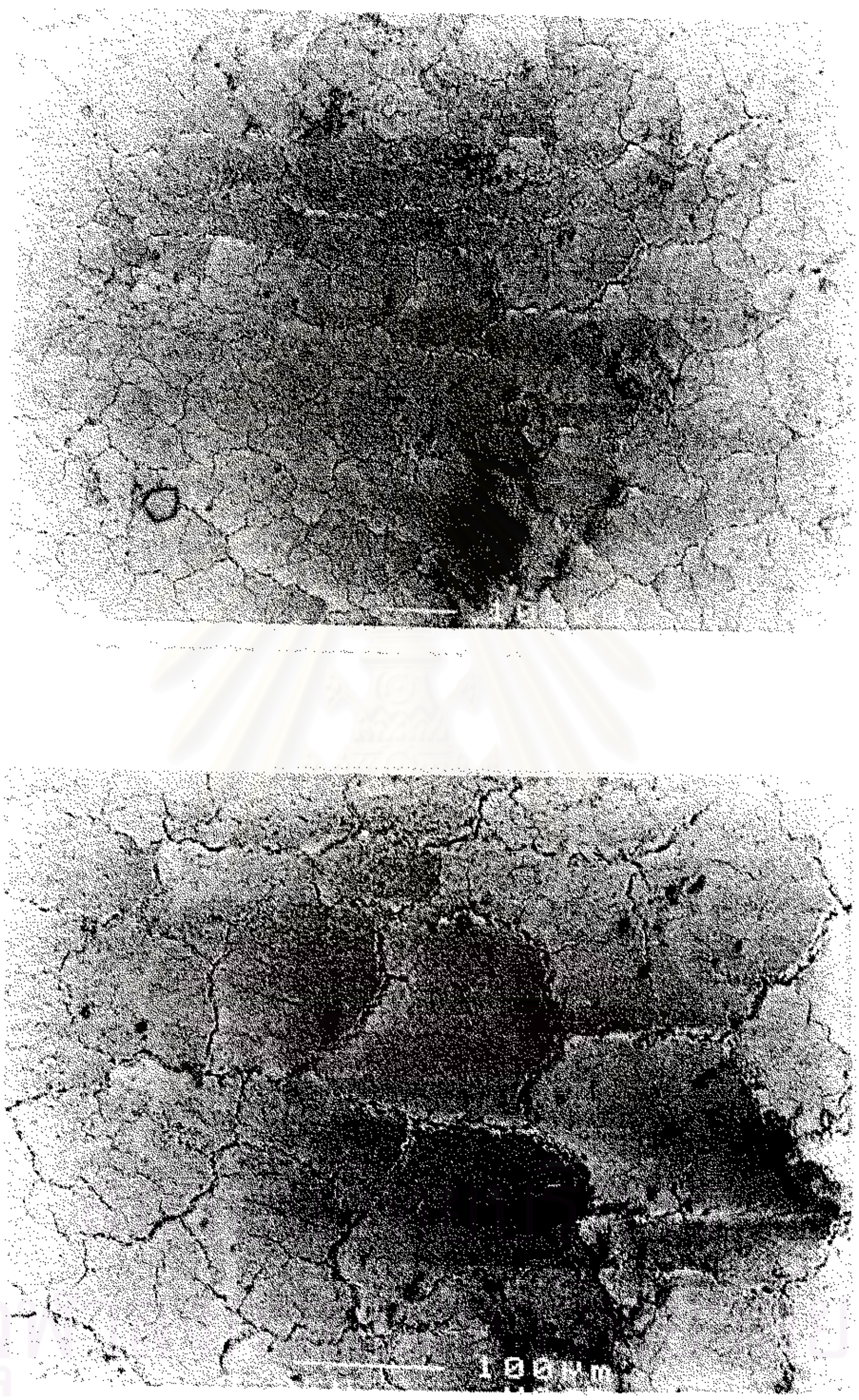


Figure 5.57 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom: Magnification 200 times

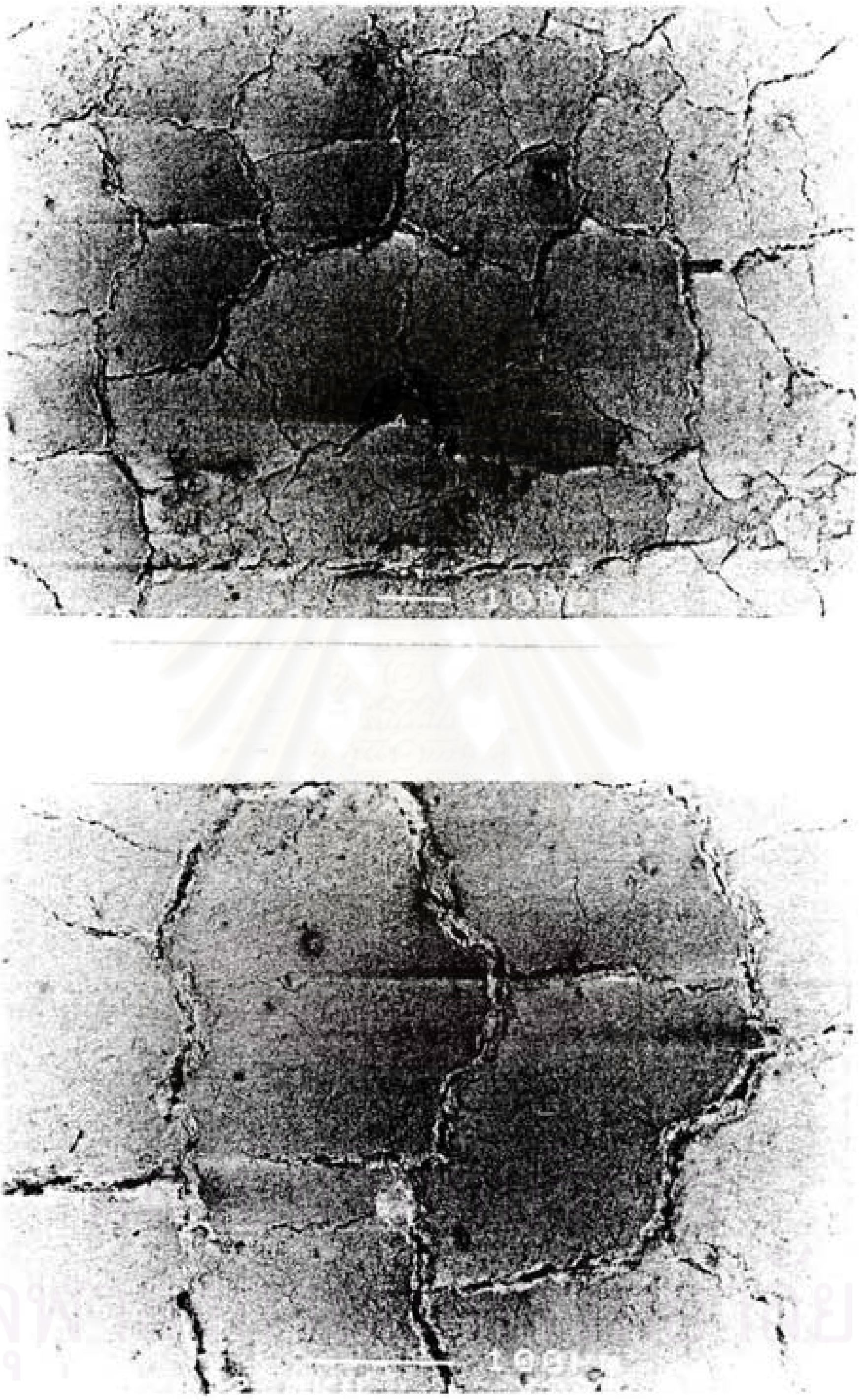
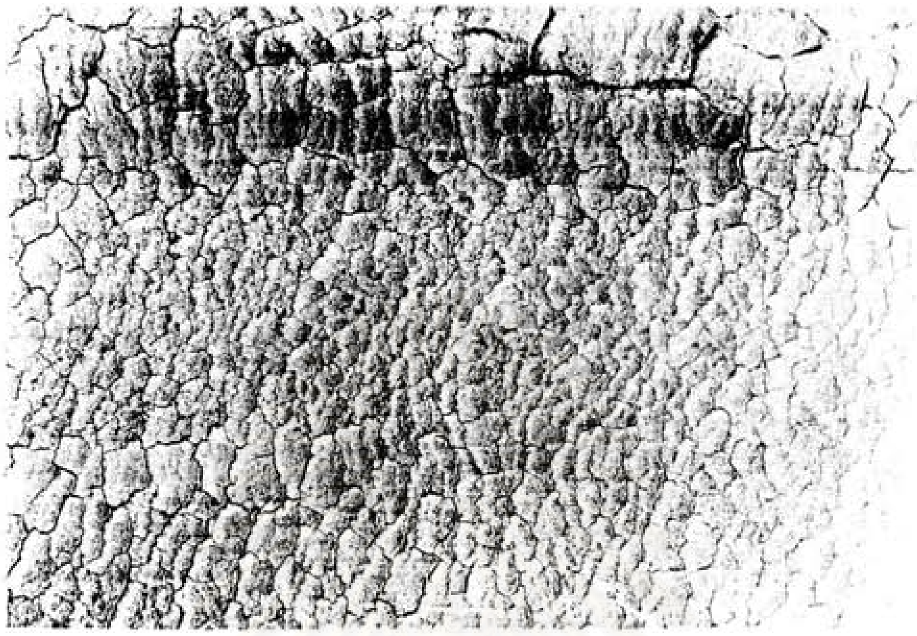


Figure 5.58 The surface of washcoat deposited on monolith dipped for two times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom: Magnification 200 times



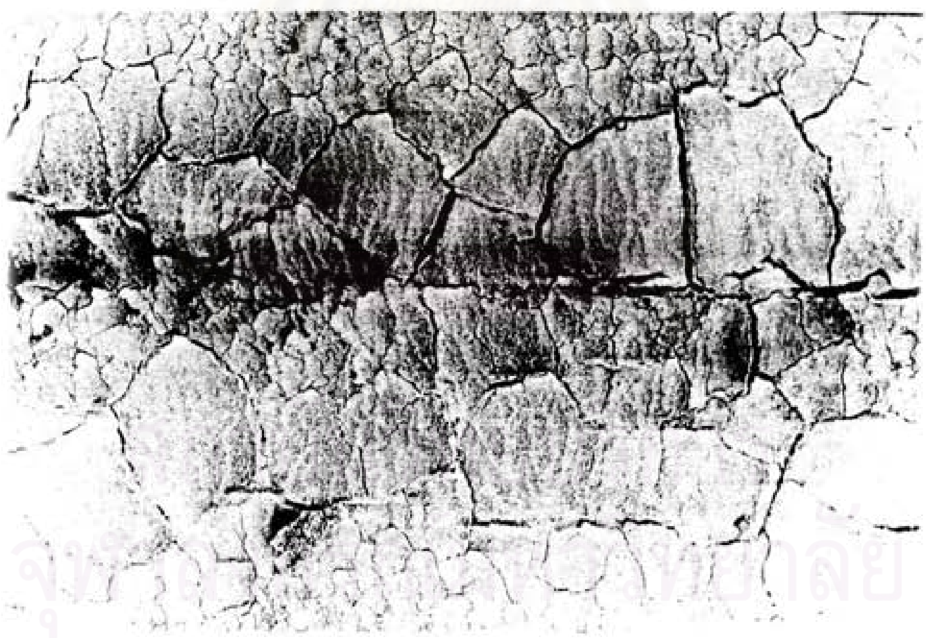
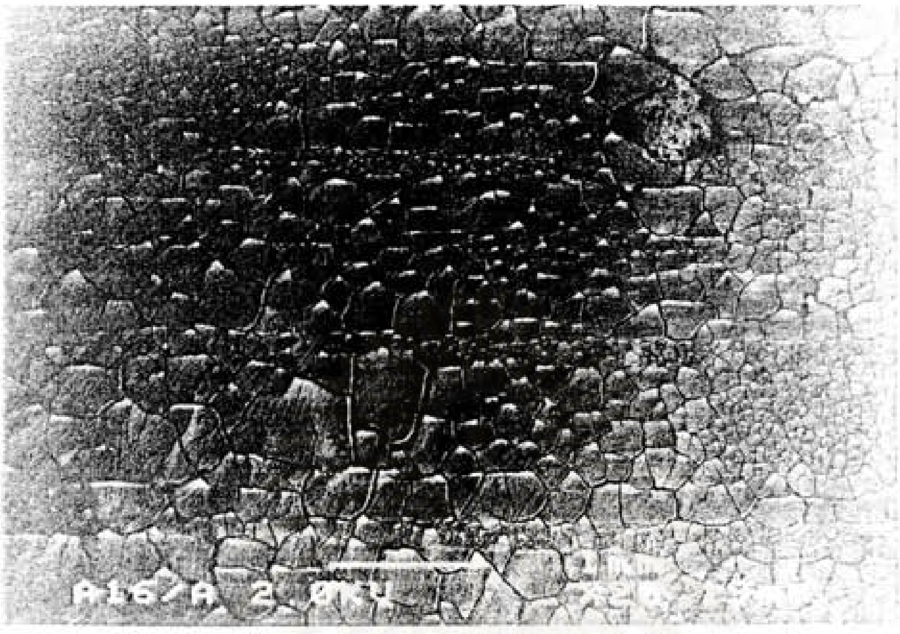


Figure 5.59 The surface of washcoat deposited on monolith dipped for two times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom: Magnification 100 times



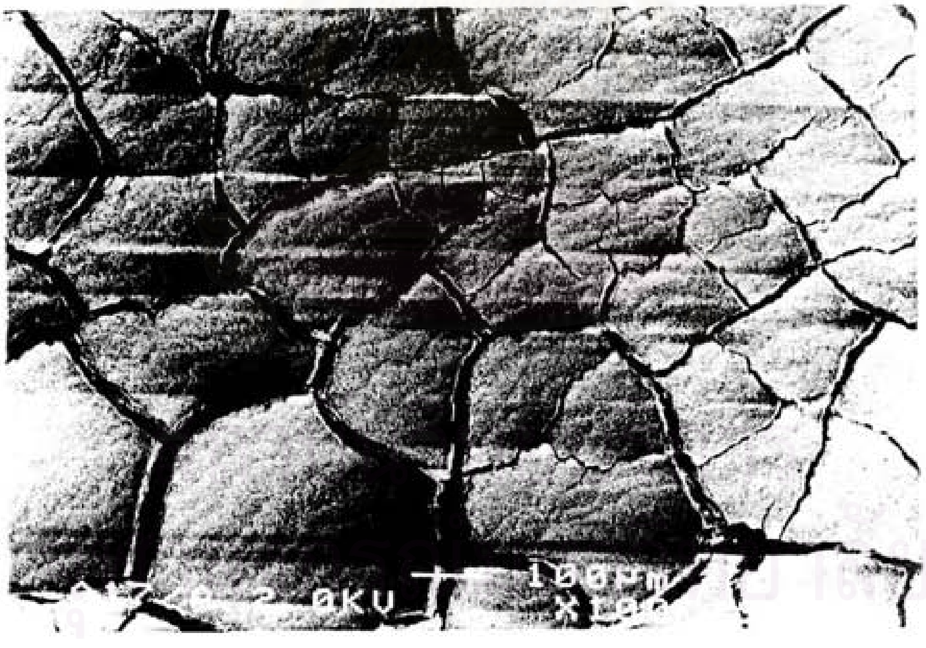


Figure 5.60 The surface of washcoat deposited on monolith dipped for two times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 20 times

Bottom: Magnification 100 times

5.3.4.3 Comparison the effect of thermalshock temperature between 600 - 800 °C for three times of washcoat coating

Figures 5.61 and 5.62 show the surface of washcoat on monoliths that were calcined at 500 °C for 2 hr and thermalshock resistance tested at 600 and 800°C, respectively. At 600°C thermalshock temperature, it was found that the washcoat cracked to the small grains in the range of 10-50 μm. and there was no grain came off the monolith surface. The result for 800°C thermalshock temperature is that the washcoat grainsizes was about 200 μm.

Figures 5.63 and 5.64 show the washcoat surface deposited on monoliths that were calcined at 500 °C for 2 hr. and thermalshock resistance tested at 600 and 800°C, respectively. The results are similar for 600 and 800°C thermalshock temperature that the washcoat surfaces were not smooth. There are some of washcoat grains released from the monolith surface.

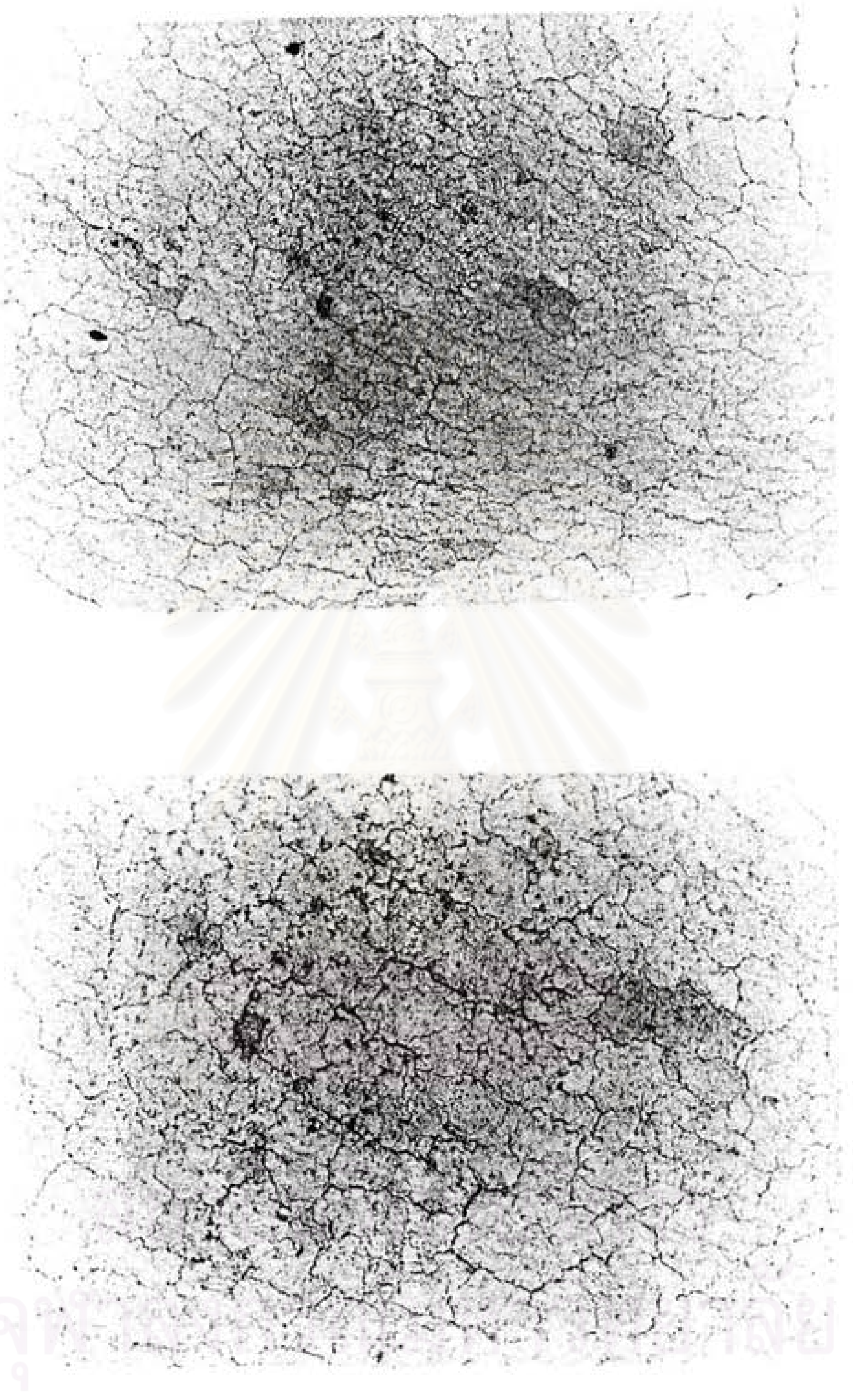


Figure 5.61 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 100 times

Bottom: Magnification 200 times

Figure 5.62 The surface of washcoat deposited on monolith dipped for three times, calcined at 500°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 100 times

Bottom: Magnification 200 times

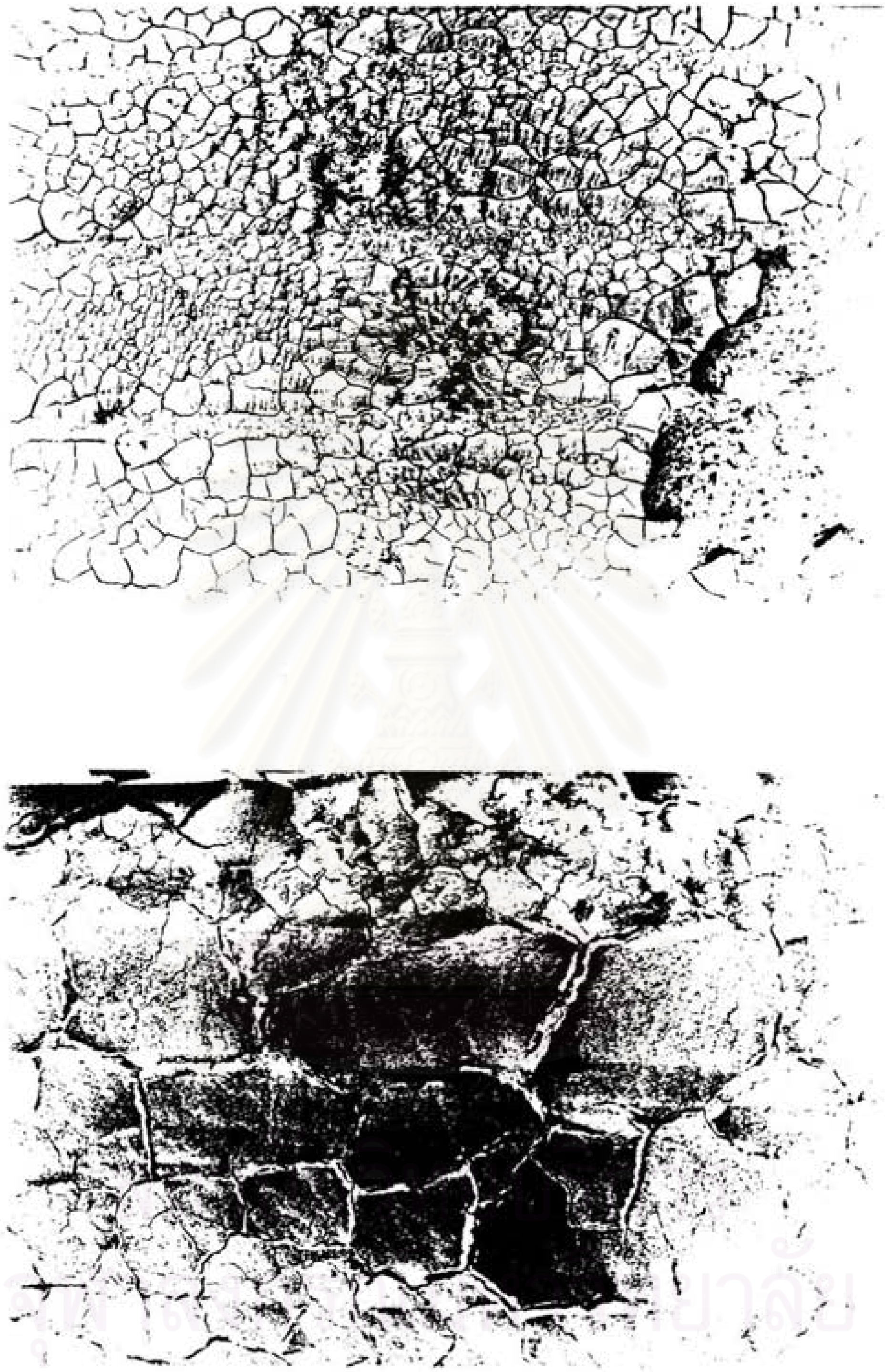
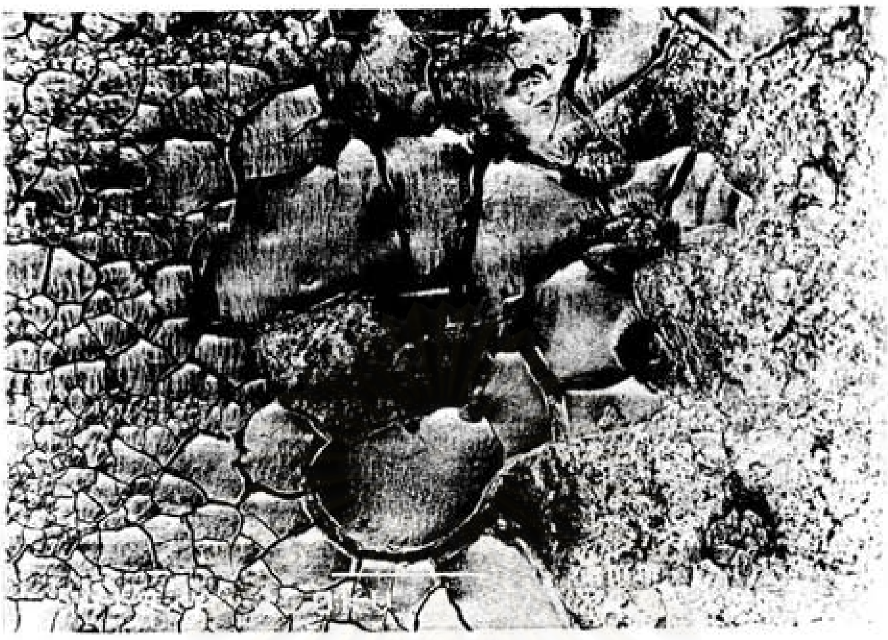


Figure 5.63 The surface of washcoat deposited on monolith dipped for three times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 600°C

Top : Magnification 20 times

Bottom: Magnification 100 times



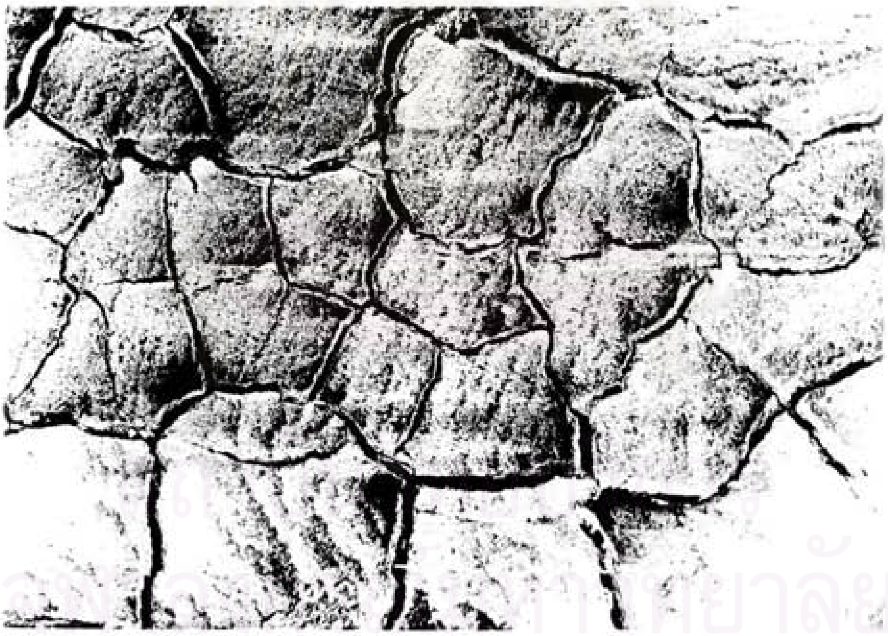


Figure 5.64 The surface of washcoat deposited on monolith dipped for three times, calcined at 600°C for 2 hr.and tested thermalshock resistance at 800°C

Top : Magnification 50 times

Bottom: Magnification 100 times

5.4 Arasive strength of washcoated monolith

The purpose of this section is to study the effect of washcoat grainsize on the abrasive strength of washcoated monolith by observing the weightloss of alumina washcoat. The washcoat grainsizes which were studied in the previous section can be devided into two types, small and large grains. The small washcoat grains deposited on monolith were obtained by dipping the monolith in washcoat slurry three times, then calcined at 500°C for 2 hr. and tested thermalshock resistance at 600°C. SEM photograph of this monolith is illustrated in figure 5.37. To obtain the large washcoat grains deposited on monolith, the ceramic monolith was coated in alumina washcoat slurry for three times and calcined at 500°C for 2 hr. and thermalshock resistance tested at 800°C .Figure 5.39 shows the surface of large washcoat grains on this monolith.

Washcoated monolith samples were heated at 800°C for 24-72 hr. The weightloss of alumina washcoat at various times were observed

Figure 5.65 shows the effect of washcoat grainsizes on the weightloss of alumina washcoat. The percent weight of large washcoat grains that released from the monolith was higher than the small washcoat grains. The weightloss of alumina washcoat of the large washcoat grains monolith was 5.26% by weight at the first time,24 hr., then the weightloss of washcoat was nearly constant for 48 hr. and 72 hr., that were 5.69 and 6.37 % by weight,respectively. This is because most of the washcoat had been released at the first time when the monolith was used. After that only the small amount of washcoat was slip off. The similar trend for small washcoat grainsize can be seen in the weightloss of alumina washcoat which showed that the weightloss of washcoat was 3.12% by weight at 24 hr. and it was little changed when heated for a long time at 48 hr. and 72 hr. The percent weightlosses of washcoat were 3.78 and 3.94 % by weight, respectively.

It can be concluded that the most suitable procedures for preparing the washcoated monlith is dipping the monolith in washcoat slurry for three time and calcined at 500°C for 2 hr. This procedure will give the minimized weightloss of washcoat.

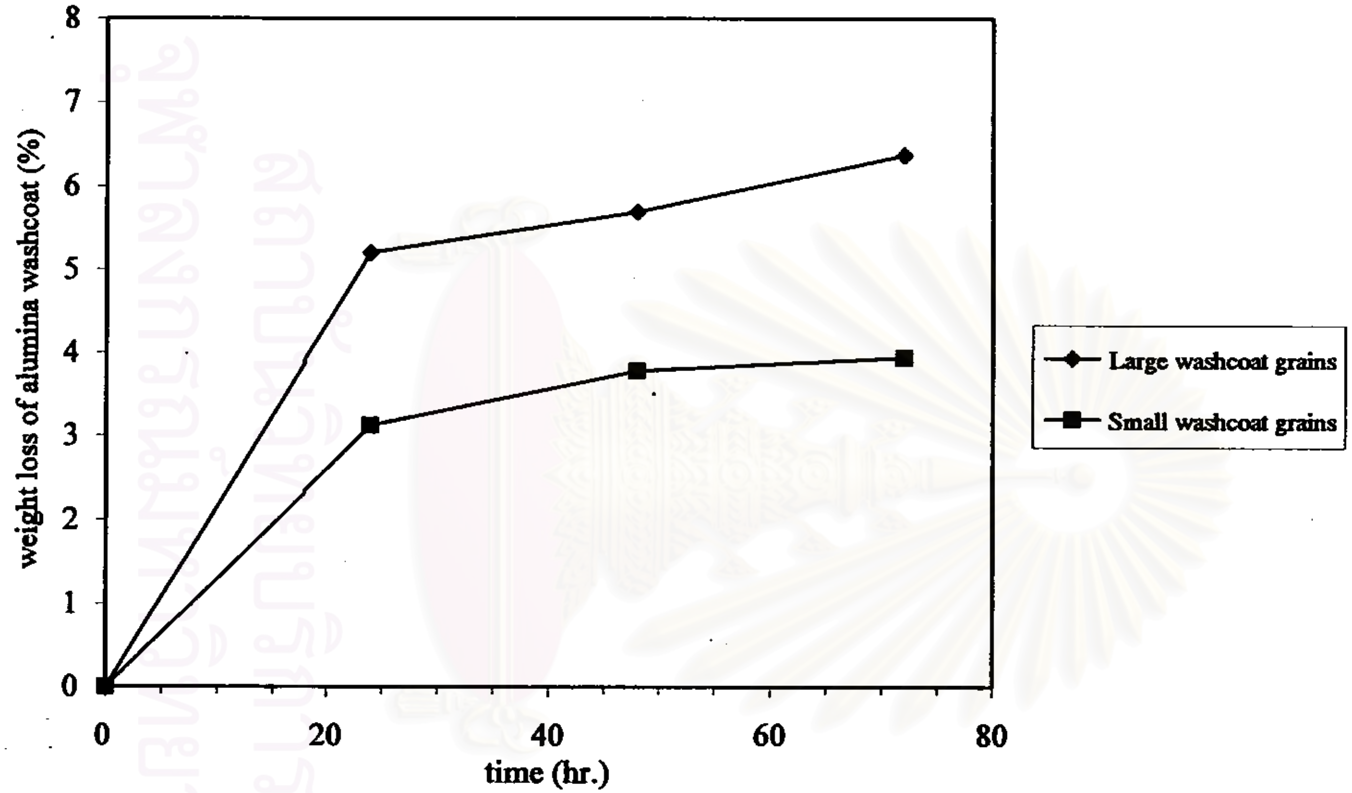


Figure 5.65 Weight loss of washcoated monolith in abrasive strength testing