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# Surface Roughness and Characteristics of High Hardness Zirconia Coating by Means of Gas Tunnel Type Plasma Spraying<sup>†</sup>

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## Abstract

*By means of gas tunnel type plasma spraying, a high hardness zirconia coating can be formed at a short spraying distance. This high hardness coating had a hardness layer near the surface.*

*The Vickers hardness of this high hardness layer became lower after thermal process. The crystal form of the tetragonal phase  $ZrO_2$  was not transformed by the thermal process, but the angular separation between the two tetragonal peaks was broader and clearer.*

*In this paper, on the surface layer of such a high hardness zirconia coating, the roughness was investigated, and the effect of spraying condition on the characteristic of roughness was clarified. The relation between the surface roughness of these zirconia coatings and deposit phenomena and the structure of the coating were discussed.*

**KEY WORDS** : (Surface Roughness), (High Hardness Coating), (Zirconia Coating), (Gas Tunnel Type Plasma Spraying), (Short Distance Spraying), (Roughness Parameter), (Mean Wavelength), (Coating Thickness)

## 1. Introduction

The zirconia coating by means of the gas tunnel type plasma spraying was investigated in the previous studies<sup>1,2,3</sup>). Especially, the characteristics of Vickers hardness on the cross sections of zirconia sprayed coatings were clarified<sup>4</sup>).

It was found that the Vickers hardness of the cross section of this coating was increased with decreasing spraying distance<sup>5</sup>), and a high hardness zirconia coating could be obtained at a short spraying distance less than a critical spraying distance  $L_p$ <sup>6</sup>).

This high hardness coating had a hardness layer of  $Hv \approx 1000$  near the surface. In this high hardness layer the pore was a little and the microstructure was very dense. When the spraying pass number was larger, the Vickers hardness of this layer was higher.

The effect of thermal process (plasma processing and heat treatment) on the characteristic of such a high hardness zirconia coating by means of gas tunnel type plasma spraying has been clarified.

After thermal process of the zirconia coating, the hardness of this high hardness layer:  $Hv = 1000$  becomes to the lower value of about  $Hv = 650$ . This was almost the same value for both heat treatment and plasma processing. Then, the clear change did not appear after the thermal process in the microstructure of this high hardness layer.

Now, the color of this coating was gray under a

certain spraying condition due to the deoxidation of  $ZrO_2$  by the heat of plasma. After thermal process of this coating, the oxidation proceeded and the color changed to white yellow which was color of zirconia powder. Corresponding to the change of color of this coating from gray to yellow after thermal process, the reflectivity of as sprayed coating:  $R = 22\%$  was increased to  $R = 50-65\%$ . This characteristic of reflectivity was similar to that of Vickers hardness of high hardness layer.

The crystal form of the cubic  $ZrO_2$  of powder was transformed into a tetragonal phase<sup>8,9</sup>) by this plasma spraying. It was found that this crystal form of the high hardness zirconia coating which is tetragonal  $ZrO_2$  was not changed by this thermal processing.

However, after heat treatment the angular separation between two tetragonal peaks was broader than that of as sprayed coating. On the other hand, through plasma processing, the angular separation was a little, because of the shortage of processing time.

In this way, the surface layer is very important for the property of high hardness zirconia coating by means of the gas tunnel type plasma spraying. In this paper, the effects of spraying condition on the roughness characteristic of the surface of such a high hardness zirconia coating had been investigated in detail: the distribution of surface roughness was measured and the characteristic of roughness parameter of such a high hardness zirconia coatings were clarified. And the influence of coating thickness on the roughness of the

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zirconia coating was clarified and the relation to the deposit phenomena and the structure of the zirconia coating was discussed.

### 2. Experimentals

The gas tunnel type plasma spraying apparatus used in this study and the experimental method to produce high hardness zirconia coatings by means of the gas tunnel type plasma spraying have been described in the previous papers<sup>4,5,6</sup>.

In this study, the ZrO<sub>2</sub> coatings were produced under spraying conditions which are shown in Table 1. In this case, the gas divertor nozzle diameter was  $d = 20\text{mm}$ <sup>7</sup>. Four pass spraying (4 times traverse) was mainly carried out to the substrate (blasted SUS304).

**Table 1** Spraying conditions for zirconia coating, gas divertor nozzle diameter:  $d = 20\text{ mm}$ .

Power input	$P = 26\text{ kW}$
Working gas (Ar)	$Q = 220\text{ l / min}$
Powder carrier gas	$10\text{ l / min}$
Powder feed rate	$w = 25\sim 35\text{ g / min}$
Traverse speed	$v = 96\text{ cm / min}$
Spraying distance	$L = 30\sim 50\text{ mm}$

Table 2 shows the chemical composition and the size of zirconia powder used in this study. Its mean size was about  $30\text{ }\mu\text{m}$ . This zirconia powder was commercially prepared type of K-90.

**Table 2** Chemical composition and particle size of zirconia powder used.

Composition (wt%)					Size ( $\mu\text{m}$ )
ZrO <sub>2</sub>	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	
90.78	8.15	0.38	0.20	0.11	10~44

For the obtained ZrO<sub>2</sub> coating, the roughness of the coating was measured by the following method. Original roughness curve  $f(x)$  on the surface of the ZrO<sub>2</sub> coating was obtained by means of surface roughness measurement machine. Typical roughness curve is shown in Fig.1(a). This measurement was carried out under the following conditions: the cutoff value was  $0.80\text{mm}$  and measurement length was  $4\text{mm}$ .

From this roughness curve, the surface roughness  $Ra$  is given by

$$Ra = \frac{1}{\ell} \int_0^{\ell} |f(x)| dx$$

where  $f(x)$  is the function which indicate the hight of the surface and  $\ell$  is the measurement length.

This original curve was not smooth as shown in Fig.1 (a). Then, the smoothing treatment was carried out by means of computer to remove small noise. Fig.1 (b) shows the roughness curve after smoothing (5 times).

Now, we introduce another roughness parameter  $\lambda$ . The wavelength  $\lambda_k$  is defined as the  $k$ th length between the two peaks shown in Fig.1(b). then, the mean wavelength  $\lambda$  is indicated by next equation,

$$\lambda = \frac{\sum_{k=1}^N \lambda_k}{N}$$

where  $N$  is the number of the wave in the roughness curve.

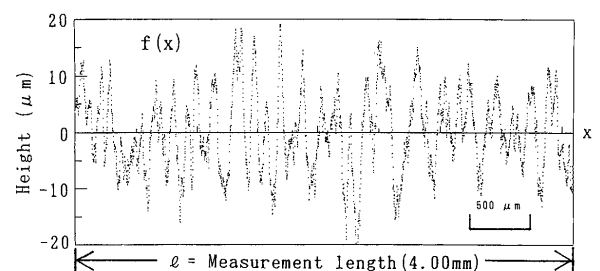
Surface roughness  $Ra$  and mean wavelength  $\lambda$  were calculated as a mean value of 3 time measurements.

### 3. Results and Discussion

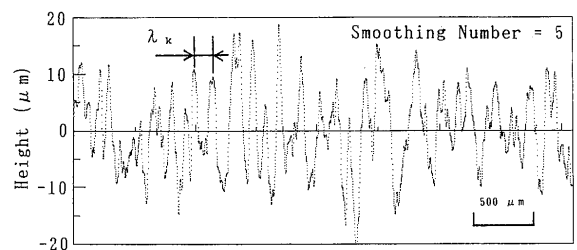
#### 3.1 Surface roughness of the zirconia coating

Figure 2 shows the schematic illustration of formation process of zirconia coating produced by the plasma spraying.

As shown in this figure, the sprayed particles are melted and become sphere in diameter of  $20\text{-}30\text{ }\mu\text{m}$  during the flight. And the spraying particles flight towards the coating surface in the vertical (thickness) direction. Then those particles collide as flat circles to deposited on



(a)



(b)

Fig.1 Roughness curve.  
(a) Original curve  
(b) Smoothing curve

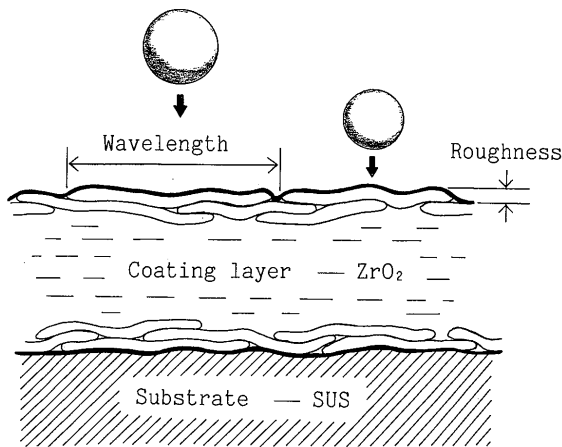


Fig.2 Schematic illustration of cross section of sprayed coating.

the substrate like a layer, which consist of sprayed coating. The coatings are actually formed by a few time traverse of the spraying powder. The observation of this sprayed coatings by optical microscope showed that small pores were spreadened all over the cross section of the coating.

As the result, the coating is formed by many deposit particles, and has a rough surface. The surface layer of the coating, namely, final pass of the traverse is most important for the roughness.

From the comparison of thus deposit phenomena with the roughness curve, we estimated the surface roughness:  $R_a$  as the thickness of one deposit particle and the wavelength:  $\lambda$  as a diameter of the deposit particle.

Figure 3 shows the typical roughness curve on the surface of zirconia coatings: (a) and substrate:(b) respectively.

Here Fig.3 (a) is the roughness curve of 4 pass sprayed zirconia coating whose thickness is about  $100 \mu\text{m}$ . The measurement was carried out at the center of the coating surface in the traverse direction.

In this case, the sprayed coating was produced at the spraying distance:  $L = 40\text{mm}$ , and at the power input:  $P = 26 \text{ kW}$ . Other spraying conditions were shown in Table 1.

And Fig.3 (b) is the roughness curve on the surface of the substrate (SUS304) used in this study, which was shot blasted. In this case, the roughness of surface was  $R_a = 1.70 \mu\text{m}$ . and the frequency is very large.

In this way, there was a large difference between the coating surface and the substrate as shown in this figure. Namely, after spraying, the coating surface becomes rough as compared with the substrate. In these zirconia coatings, the mean value was  $R_a = 5.0 \mu\text{m}$ . And it was found that the mean wavelength of the coating surface became larger value ( $\lambda = 110 \mu\text{m}$ ) than that of the

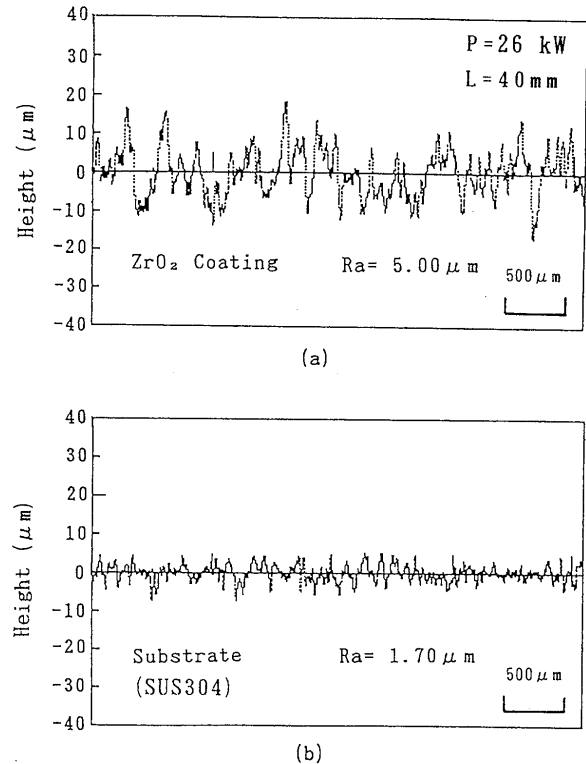


Fig.3 Comparison of roughness curve.

- (a)  $\text{ZrO}_2$  coating  
(b) Substrate

substrate.

### 3.2 Distributions of roughness parameters on the coating surface

The change of roughness curve on the surface of zirconia coating by means of the gas tunnel type plasma spraying was also measured to know the surface condition of the coating and the following results were obtained.

The distribution of roughness curve on the surface of zirconia coating is shown in Fig.4. The measurement of roughness curve was carried out in the same manner as Fig.1. The measured direction is shown the upper left of this figure, where the size of specimen is also indicated.  $x$  indicates the distance from the end of substrate.

This zirconia coating was produced as the following conditions: 4 pass spraying of the powder feed rate was about  $w = 30\text{g/min}$  at  $L = 30\text{mm}$  when  $P = 26 \text{ kW}$ . And the coating thickness was  $150 \mu\text{m}$  at the traverse axis:  $x = 30\text{mm}$ .)

It is found that for the roughness curve near the traverse axis ( $x = 30\text{mm}$ ), the roughness was a little smaller than that of both side of the traverse axis ( $x = 25\text{mm}$  and  $x = 40\text{mm}$ ). While, the roughness curve at the

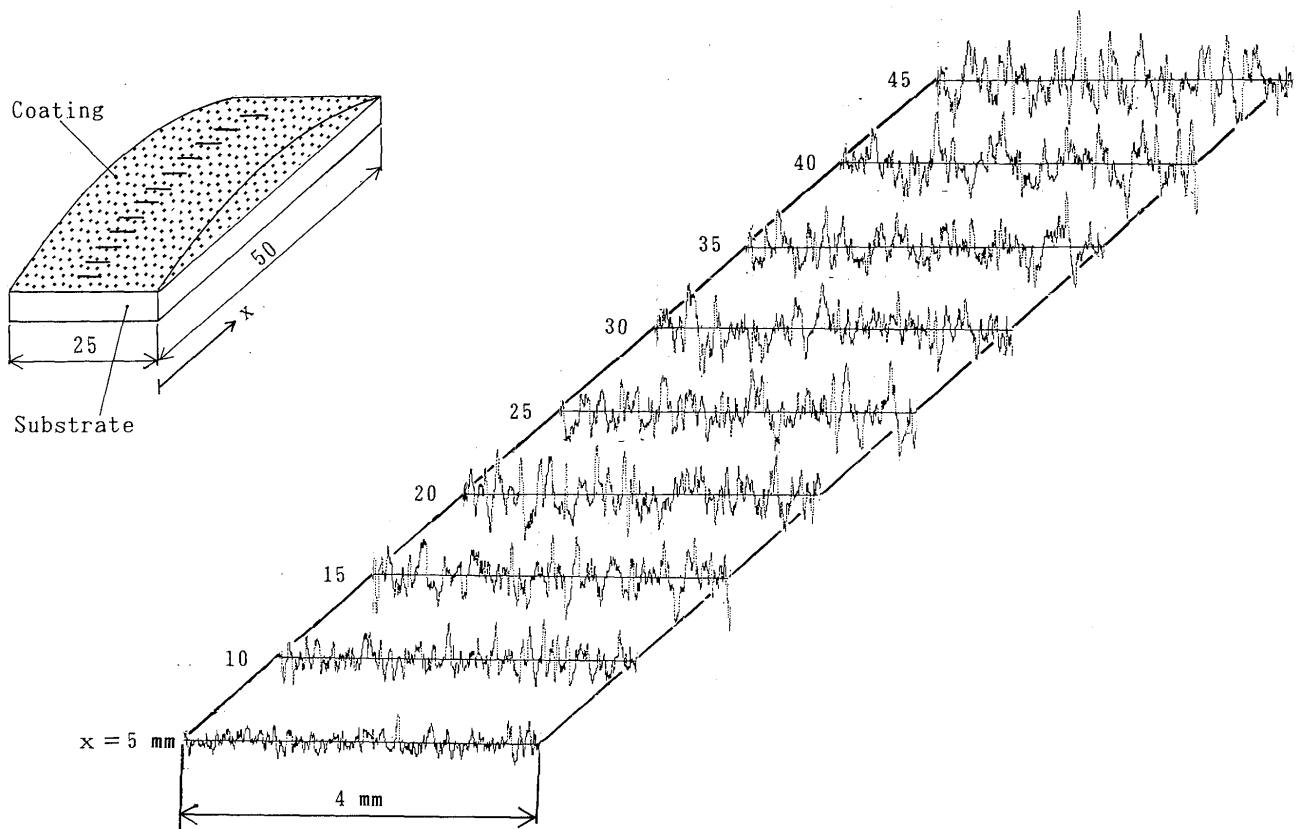


Fig.4 Distributions of roughness curve on surface of zirconia coating.  
Upper left: specimen and measurement direction

end of specimen was similar to that of the substrate, because the thickness of the coating was very thin.

Figure 5 shows the distributions of roughness parameter (surface roughness and mean wavelength) obtained from roughness curve on the surface of zirconia coatings produced by 4 pass spraying. In the same figure, the thickness of sprayed zirconia coating was indicated by the square mark. Here,  $R_a$ ,  $\lambda$  and  $t_c$  were surface roughness, mean wavelength and coating thickness respectively. The distribution of those value of sprayed zirconia coating was measured in the  $x$  direction, which is indicated in Fig.4.

Fig.5 (a) shows the distributions for the same coating as Fig.4, produced at  $L = 30\text{mm}$  when  $P = 26\text{ kW}$ . As shown in this figure,  $t_c$  is most thick at the center region ( $x = 30\text{mm}$ ):  $t_c = 160\ \mu\text{m}$ .

In this zirconia coating, the distribution of surface roughness:  $R_a$  consists of two parabolic curves and have two peaks. The value of  $R_a = 4.5\text{-}5.5\ \mu\text{m}$  at the center region. The surface roughness of the coating was changed from  $R_a = 5.5$  to  $2.0\ \mu\text{m}$  in this distribution.

While, the wavelength is  $\lambda = 135\ \mu\text{m}$  at the center, and decreased on the both side. Here,  $R_a$  have a flatter

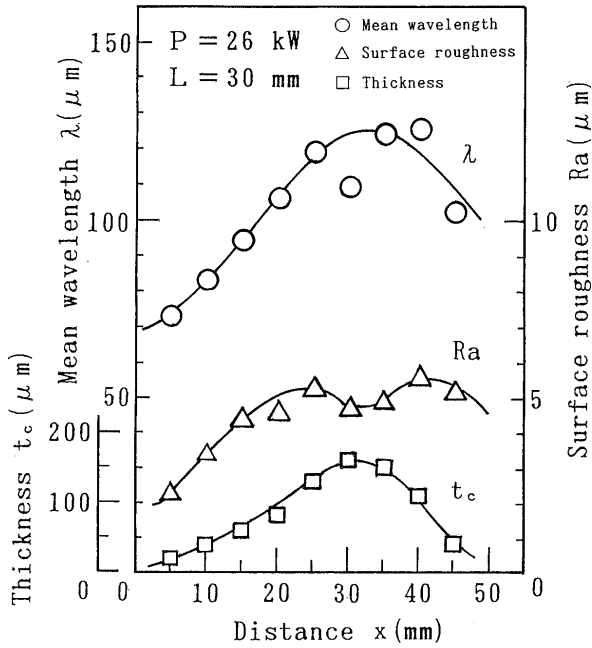
distribution as compared with  $\lambda$ .

In Fig.5 (b), is shown the distribution of roughness parameter for the coating at  $L = 50\text{mm}$ , when  $P = 26\text{ kW}$ . This is thin coating (about  $100\ \mu\text{m}$ ).

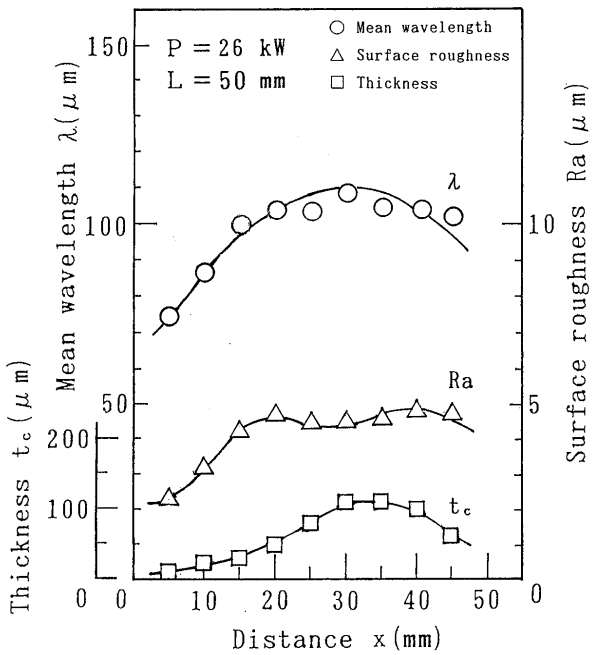
This zirconia coating has a flatter distribution of  $R_a$ . The surface roughness is decreased on the side of center axis. The value at the center region changed from  $R_a = 4.5\text{-}5.5\ \mu\text{m}$  to  $R_a = 4.5\ \mu\text{m}$ . The distribution of  $\lambda$  is also like parabolic curves, but a little broader. The maximum value is  $115\ \mu\text{m}$ , a smaller value than the case of  $L = 30\text{mm}$ .

In this way, the roughness of the surface layer of zirconia coating was lower at the center of traverse axis, and this coating have a rough surface at the both side of center axis. This is thought that the zirconia coating has a flatter deposite of at the center and large wavelength, but rough surface at the side by the spattering, which is highest value of about  $R_a = 5.5\ \mu\text{m}$ .

Observing the microstructure of those zirconia coatings, it was found that the coating structure was almost the same structure of all over the sprayed coating.



(a)



(b)

Fig.5 Distributions of mean wavelength, surface roughness and coating thickness on the coating surface.  
 (a) Zirconia coating at  $L = 30\text{mm}$   
 (b) Zirconia coating at  $L = 50\text{mm}$

**3.3 Characteristics of surface roughness of high hardness zirconia coating**

In case of the zirconia coating in this study, as mentioned above the Vickers hardness becomes lower as

the spraying distance is large. Then, the characteristics of the roughness parameter at the center of coating was studied. On this region of  $x = 25\text{-}40\text{ mm}$  in the case of Fig.4, the thickness of the coating was maximum and hardest,

Figure 6 shows the dependences of the surface roughness and mean wavelength of the zirconia coating on the spraying distance at  $P = 26\text{ kW}$ . It is found that the thickness of these coatings change from  $t_c = 180\text{ }\mu\text{m}$  at  $L = 30\text{mm}$  coating, to  $t_c = 110\text{ }\mu\text{m}$  at  $L = 50\text{mm}$ .

Here, the mean wavelength decreases as the spraying distance  $L$  increases, corresponding to the increase of coating thickness. It is thought that the deposit particle becomes flatter and increase its diameter on the coating surface. On the contrary, the surface roughness,  $R_a$  is the value between  $5.5\text{-}4.5\text{ }\mu\text{m}$  and become a little lower at large spraying distance. The dependence of  $R_a$  on the spraying distance is weak.

**Effect of coating thickness**

Figure 7 shows the dependence of roughness parameter of zirconia coating in the coating thickness. These coating were produced by 4 pass spraying at  $L = 30\text{mm}$  when  $P = 26\text{ kW}$ .

The surface roughness was nearly constant at any coating thickness,  $R_a = 5\text{ }\mu\text{m}$ . The dependence on  $t_c$  is weak as well as the case of Fig.7.

On the other hand, the mean wavelength  $\lambda$  was

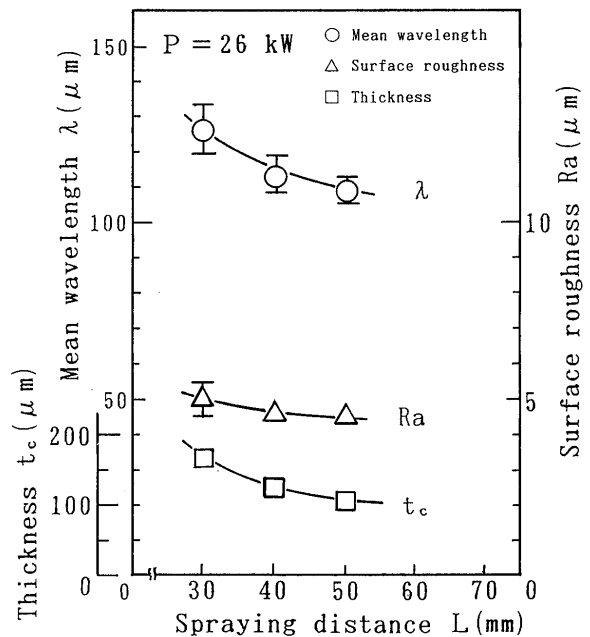


Fig.6 Dependence of mean wavelength, surface roughness and coating thickness on spraying distance, when  $P = 26\text{ kW}$ .

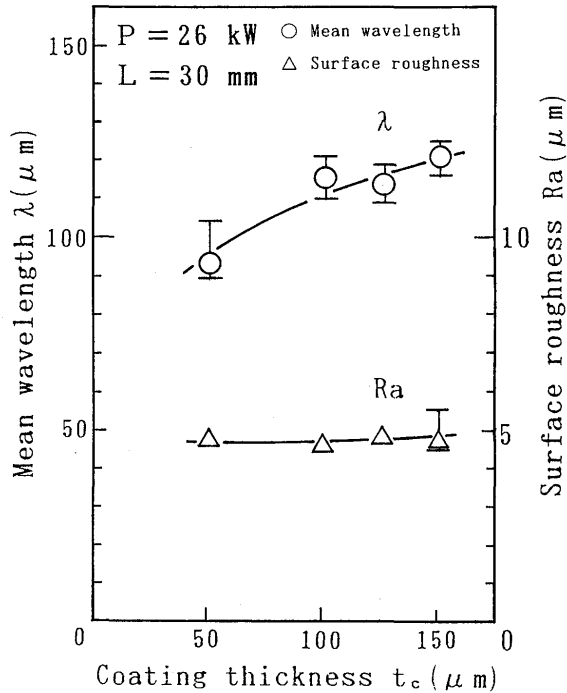


Fig.7 Dependence of mean wavelength and surface roughness on coating thickness at  $L = 30\text{mm}$  when  $P = 26\text{kW}$ .

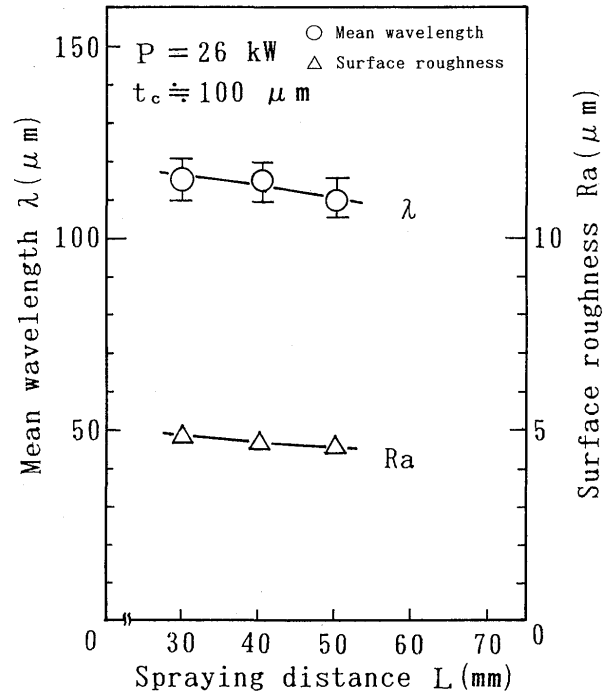


Fig.8 Dependence of mean wavelength and surface roughness for constant coating thickness on spraying distance.

increased largely as the thickness was increased. The value is  $\lambda = 95 \mu\text{m}$  for the coating of  $t_c = 50 \mu\text{m}$ , and  $\lambda = 125 \mu\text{m}$  for the coating of  $t_c = 150 \mu\text{m}$ . This characteristic of  $\lambda$  is similar to that of Vickers hardness described in the previous report.

Thus in this high hardness zirconia coating, the dependence of mean wavelength:  $\lambda$  is very large, as compared with the surface roughness:  $R_a$ . The  $\lambda$  became higher as the thickness increased. Such an effect is similar to that of Fig.6. This is thought that on the surface of a high hardness layer the temperature is higher as the increasing thickness.

**Effect of spraying distance**

From the results of Fig.7, for the surface of high hardness zirconia coating formed by the gas tunnel type plasma spraying, it is found that the effect of  $t_c$  was very large. Then in the case of same thickness, the effect of spraying distance was studied.

The dependences of mean wavelength and surface roughness on the spraying distance are shown in Fig.8. These coatings were produced at  $P = 26 \text{ kW}$  and those thickness were constant; about  $100 \mu\text{m}$ , respectively.

As compared with the result of Fig.6, the dependence of roughness parameter on the spraying distance is a little. The  $\lambda$  of sprayed coating becomes lower value through the increase of spraying distance.

This shows that the deformation of deposit particles depend upon the thickness, and structure of the deposit particle of zirconia coating did not change largely through spraying distance.

On the other hand, the  $R_a$  of the coating is almost constant as compared with the wavelength.

Thus, the dependences was weak on the spraying distance. It is thought that the thickness of zirconia which increase the surface temperature was dominant for the surface condition on the thermal process.

**4. Conclusion**

In this study, the roughness of the high hardness zirconia coating by means of gas tunnel type plasma spraying was investigated and the following results were obtained.

- 1) After plasma spraying the coating surface became rough as compared with the substrate. The surface roughness of the coating was about  $R_a = 5.0 \mu\text{m}$ , while the substrate,  $R_a = 1.7 \mu\text{m}$ .
- 2) The measurement of the distribution of roughness curve showed that the zirconia coating had a rough surface at the both side of traverse axis. This is thought that the zirconia coating has a flatter deposit at the center and that the surface become rough at the side by the spattering.

3) From the distributions of roughness parameters on the surface, it was found that the wavelength  $\lambda$  of the coating was largest value of about  $\lambda = 125 \mu\text{m}$  at the center. The surface roughness  $R_a$  of zirconia coating was a minimum of  $R_a = 4.5 \mu\text{m}$  at the center. This distribution of  $R_a$  was flatter than that of  $\lambda$ .

When the spraying distance was increased, the distributions of  $R_a$  and  $\lambda$  became broader respectively. For  $R_a$ , the deviation between center value and those peaks were decreased.

As the results, the smooth surface was obtained at the center of surface layer of the coating. Then the roughness parameter at the center was measured.

4) The surface roughness became a little smaller value, as the spraying distance was increased.  $R_a$  of zirconia coating was about  $R_a = 5.0 \mu\text{m}$  at  $L = 30\text{mm}$ , when  $P = 26 \text{ kW}$  and became a lower value of about  $4.5 \mu\text{m}$  at  $L = 50\text{mm}$ .

On the other hand, the mean wavelength of the coating surface was decreased largely, when the spraying distance was increased:  $\lambda$  was about  $125 \mu\text{m}$  at  $L = 30\text{mm}$  and  $\lambda = 110 \mu\text{m}$  at  $L = 50\text{mm}$ .

5) Corresponding to the increase of coating thickness, the wavelength of roughness curve of sprayed coating was changed largely: for thin coating  $\lambda = 95 \mu\text{m}$  and for thick coating  $\lambda = 125 \mu\text{m}$ . Thus, it was found that the coating thickness  $t_c$  had a large effect on the condition of coating surface.

6) The results obtained by means of the modification for the coating thickness showed that the effect of spraying distance on the roughness parameter was not so large as compared to that of coating thickness.

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