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# Structure Analysis of High Hardness Alumina Sprayed Coating by Polarizing Microscope†

Akira KOBAYASHI\*, Naoto HASEGAWA\*\* and Hirokazu NAMIKAWA\*\*\*

## Abstract

By means of gas tunnel type plasma spraying, a high hardness alumina coating can be formed at a short spraying distance. The microstructure of such a hardness coating was observed by a polarizing microscope, and the mechanism of the formation of such hardness coating and the relation between the microstructure and Vickers hardness of this high hardness coating was discussed.

**KEY WORDS:** (High Hardness Coating) (Alumina Coating) (High Hardness Layer) (Gas Tunnel Type Plasma Spraying) (Short Distance Spraying) (Polarizing Microscope) (Microstructure) ( $\alpha$ -alumina Rich Layer)

## 1. Introduction

In a gas tunnel type plasma spraying, the deposit characteristics of sprayed ceramic powder has been studied at various spraying conditions<sup>1)</sup>, and it was clarified that the sprayed particles which are in good melting state deposit onto the substrate.

As the results, the porosity is decreased, and the mechanical properties such as Vickers hardness are improved better. Therefore, it has been obtained high quality ceramic coatings with a gas tunnel type plasma spraying, as compared to the conventional ceramic coatings<sup>2,3)</sup>. And in order to know the mechanism of the formation of such ceramic coatings, the characteristics of those Vickers hardness and porosity of the power input to plasma jet and spraying distance has been investigated.

Moreover, it has been reported in the previous paper<sup>4)</sup> that by means of gas tunnel type plasma spraying can be obtained a high hardness coating (in the case of alumina coating, more than  $H_V = 1300$ ) at a short spraying distance.

Namely, the Vickers hardness of the ceramic coating is increased with decreasing spraying distance, and the characteristics of the Vickers hardness change dramatically at the spraying distance  $L_p$ , in the gas tunnel type plasma spraying. A higher Vickers hardness can be obtained in the case of short distance spraying ( $L < L_p$ ) at each power input.

For example, Vickers hardness of alumina coating was  $H_V = 1500$  at  $L = 30\text{mm}$ , when  $P = 30\text{kW}$ . In this case, the value of  $L_p$  was from 40mm to 50mm, which related to

the plasma jet length.

According to the measurement of the distribution of the Vickers hardness on the cross section of the coating, these high hardness coating has a hardness layer near the surface. This high hardness layer is increased in the width (thickness) and has a higher value of Vickers hardness in the case of larger power input.

Observing the microstructure of alumina coating in this high hardness layer, it is found that the cell size is very fine compared with other parts of coating.

Therefore, in this paper, the microstructure of high hardness alumina coatings formed by the gas tunnel type plasma spraying has been analyzed by using a polarizing microscope<sup>5)</sup>, and the effects of spraying conditions on the structure of the sprayed coating was investigated.

And the relation between the structure and characteristics of the hardness in the alumina coatings with gas tunnel type plasma spraying was discussed.

## 2. Experimentals

The gas tunnel type plasma spraying apparatus used in this study, and the experimental method in order to form high hardness ceramic coatings by means of the gas tunnel type plasma spraying has been described in the previous papers<sup>1,2,3)</sup>.

The experiment of this plasma spraying was carried out mainly at short spraying distances ( $L = 20 \sim 50\text{mm}$ ) using alumina powder, and the effects of spraying conditions on the hardness of the sprayed coating were clarified.

In the Vickers hardness measurement of the sprayed

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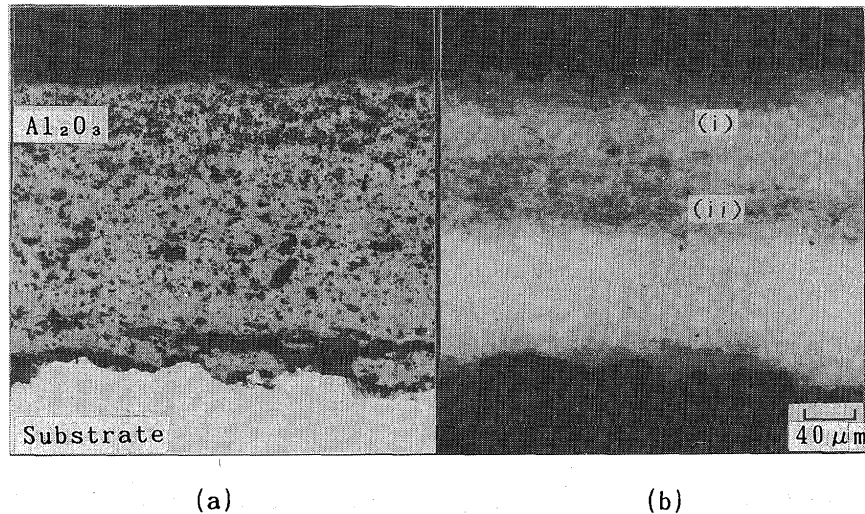


Fig.1 Photograph of cross section of alumina coating by reflected type polarizing microscope at spraying distance of  $L=30\text{mm}$ , when power input is  $P=20\text{kW}$ , (a) open Nicol, (b) cross Nicol.

coatings, the following conditions were adopted. The load weight was 100g and/or 300g, its load time was more than 15s. The measurement was carried out at each distance from the coating surface in the thickness direction.

And the structure of the hardness in the ceramic coatings with gas tunnel type plasma spraying was analyzed by the reflected type polarizing microscope. This observation was carried out on the cross section of the coating. And from the surface of the coating, it was analyzed by the X-ray diffractometer.

### 3. Results and Discussion

#### 3.1 Observation of alumina sprayed coating by means of polarizing microscope

Figure 1 shows the photograph of the cross section of an alumina coating by a reflected type polarizing microscope. In this case, the coating was formed when the power input was  $P=20\text{kW}$  and spraying distance was  $L=30\text{mm}$ . Fig.1(a) is the photograph by the polarizing microscope in open Nicol condition, and Fig.1(b) is in cross Nicol condition.

Generally, the alumina sprayed coating consists of  $\gamma$ -alumina and few  $\alpha$ -alumina<sup>6)</sup>. Here, as a crystal system,  $\alpha$ -alumina is hexagonal lattice and optical anisotropy. This is observed as bright color (white) by a polarizing microscope in cross Nicol condition because of the polarization of light. On the other hand,  $\gamma$ -alumina is cubic lattice and optical isotropy, which show dark color (black)<sup>7)</sup>.

In open Nicol condition, the photograph is almost the same as that by an optical microscope, as shown in Fig. 1(a).

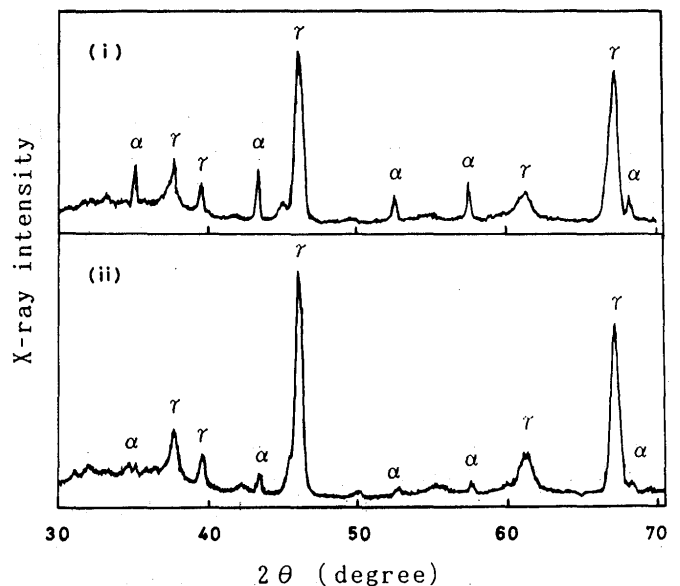


Fig.2 X-ray diffraction patterns of alumina coating at the location of (i) and (ii) in the photograph (b) of Fig.1.

Then, in cross Nicol condition, the photograph of Fig.1(b), the under part of the coating near the substrate shows white color. This means, namely, that there exists many  $\alpha$ -alumina. In the part (i) near the coating surface, there also exists a few  $\alpha$ -alumina. On the other hand, in part (ii), middle of coating the color is black, which means that this part mainly consists of  $\gamma$ -alumina.

This was proved by means of the X-ray diffraction method. Figure 2 shows the X-ray diffraction patterns at the location of (i) and (ii) in the photograph (b) of Fig.1. The condition of this measurement was as follows: the source of X-ray was copper, tube voltage was 40 kV and tube current was 20mA.

This result shows that in part (ii) there is mainly  $\gamma$ -alumina, very few  $\alpha$ -alumina. On the other hand, in part (i) the peak of  $\alpha$ -alumina is larger than that of part (ii). It is thought that this part consists of both  $\alpha$ -alumina and  $\gamma$ -alumina. Therefore it is found that  $\alpha$ -alumina is observed as a white color area in cross Nicol condition by a reflected type polarizing microscope. On the other hand,  $\gamma$ -alumina is observed as a black color area.

In this way, we can know easily the distribution of  $\alpha$ -alumina in the cross section of high hardness alumina coating by using a reflected type polarizing microscope.

### 3.2 Structure of alumina sprayed coating

The microstructure of high hardness alumina coatings formed by the gas tunnel type plasma spraying, was analyzed by means of the polarizing microscopic measurement of those cross sections of coatings.

Figure 3 shows the photograph of the cross section of the alumina coating by a polarizing microscope. The spraying conditions of this case were as follows: the power input was  $P = 20\text{kW}$  and the spraying distance was  $L = 30\text{mm}$ . (a) was in the case of one path scanning and (b) three path scanning sprayed coating. (In this case, three path was scanned not continuously.) The coating thickness per one path was about  $100\ \mu\text{m}$  respectively.

Observing the photograph (ii) of Fig.3(a), in the sprayed coating, the part near the substrate is bright. This

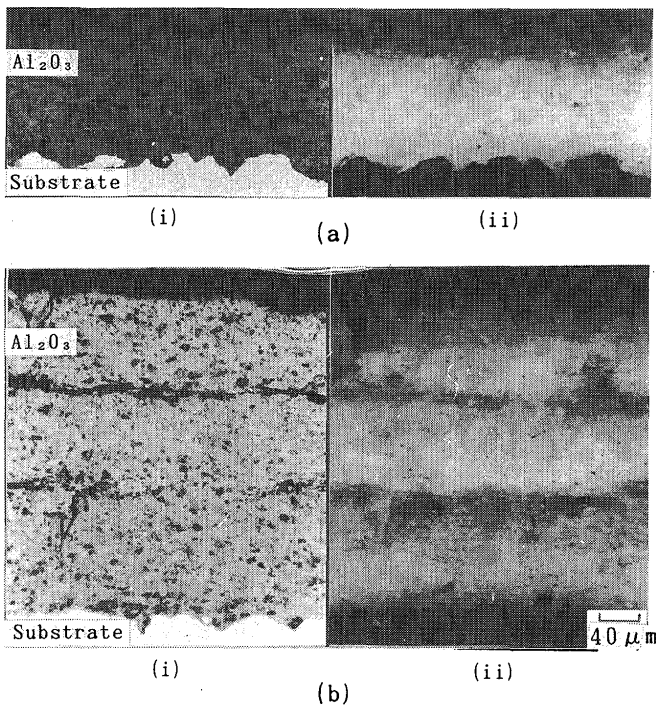


Fig.3 Photograph of cross section of alumina coating by a polarizing microscope at spraying distance of  $L = 30\text{mm}$ , when power input is  $P = 20\text{kW}$ . (a) one path scanning, (b) three path scanning, (i) open Nicol, (ii) cross Nicol

white layer seems to be an  $\alpha$ -alumina rich layer as a coating structure. On the other hand, in the part near the coating surface is formed a dark (black) layer. This shows that the coating surface is affected by the heat from plasma, so that  $\alpha$ -alumina sufficiently changes to  $\gamma$ -alumina.

The photograph (ii) of Fig.3(b) has three patterns which is shown in the sprayed coating Fig.3(a). Each pattern corresponds to each one path. The total thickness of this coating is about  $300\ \mu\text{m}$ . In this way, the coating per one path scanning, has such a pattern.

On the other hand, the alumina coating which was formed at a normal spraying distance such as  $L = 80\text{mm}$  when  $P = 20\text{kW}$  is shown in Fig.4. In this case, such pattern as the case of short distance spraying is not

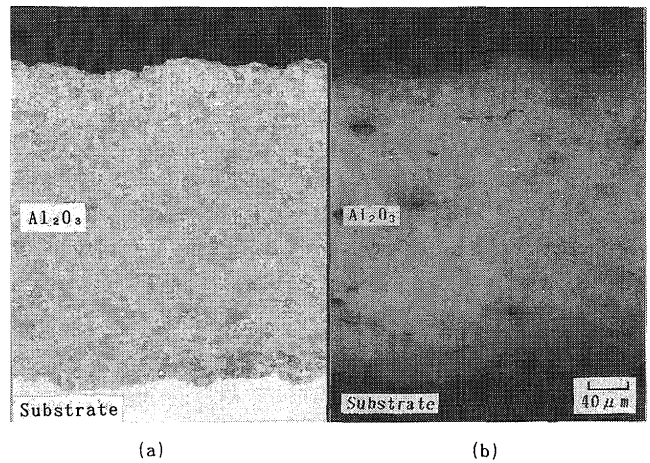


Fig.4 Photograph of cross section of alumina coating by a polarizing microscope at  $L = 80\text{mm}$ , when  $P = 20\text{kW}$ , (a) open Nicol, (b) cross Nicol.

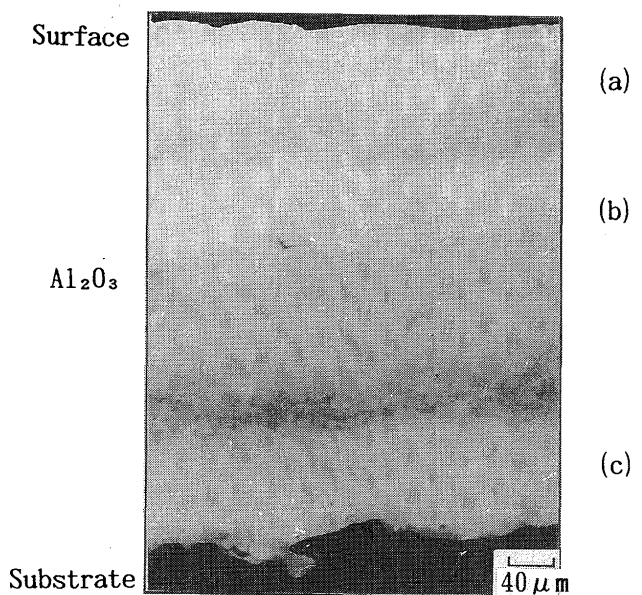


Fig.5 Photograph of cross section of alumina coating by polarizing microscope (cross Nicol) at  $L = 30\text{mm}$ , when  $P = 20\text{kW}$ .

observed in Fig.4(b) (cross Nicol). The brightness of this photo is uniform over all area of the coating. Therefore, it seems that the ratio of  $\alpha$ -alumina to  $\gamma$ -alumina is constant. This corresponds to the fact that it have a flat distribution of Vickers hardness on the cross section in the case of normal spraying distance. A high hardness layer does not exist in the alumina coating at  $L = 80\text{mm}$  when  $P = 20\text{kW}$ .

Figure 5 shows the photograph of the cross section of high hardness alumina coating by a polarizing microscope (cross Nicol), which was formed in the way of three path scanning. In this case, second pass and third pass scanning were carried out continuously, different from the case of Fig.3(b).

This coating was formed in the following conditions, the power input to plasma jet was  $P = 20\text{kW}$  and the spraying distance was  $L = 30\text{mm}$ . Ar gas flow rate was  $Q = 2001/\text{min}$ , and the powder feed rate was  $80\text{g}/\text{min}$ . The coating thickness was about  $450\ \mu\text{m}$ .

Observing this photograph of sprayed coating by the polarizing microscope, it is found that the part (c) near the substrate (first path) is the same coating structure as that of Fig.3(a). On the other hand, in the part (a) near the coating surface (third path) affected by the heat of plasma, and the sprayed powder was remelted. the cell size becomes bigger. Moreover, in the part (b) (second path), the brightness is the highest in the coating, and a white layer, which is  $\alpha$ -alumina rich, is formed.

Figure 6 shows the distribution of Vickers hardness on the cross section of this high hardness alumina coating.

As shown in this figure, a high hardness layer which have the Vickers hardness of more than  $H_v = 1300$  appears at the middle of the coating thickness. The width of this layer is about  $60\ \mu\text{m}$ . In this part, the sell size is very fine as compared with that in other parts. This layer corresponds to  $\alpha$ -alumina rich layer (white part) (b) in

Fig.5.

In the case of a large power input, as mentioned formerly<sup>4)</sup>, the Vickers hardness becomes higher and the hardness layer becomes wider, in the gas tunnel type plasma spraying. Figure 7 shows the photograph of cross section of high hardness alumina coating by a polarizing microscope (cross Nicol).

In this case, the power input was  $P = 30\text{kW}$  and two path scanning. and other spraying conditions were the same as Fig.4: the spraying distance was  $L = 30\text{mm}$ . Ar gas flow rate was  $Q = 2001/\text{min}$ , and the powder feed rate was  $80\text{g}/\text{min}$ . The coating thickness was about  $450\ \mu\text{m}$ .

Analyzing the photo of this sprayed coating, it is found that the part (c) near the substrate is the same coating structure as the part (c) in Fig.4. On the other hand, in

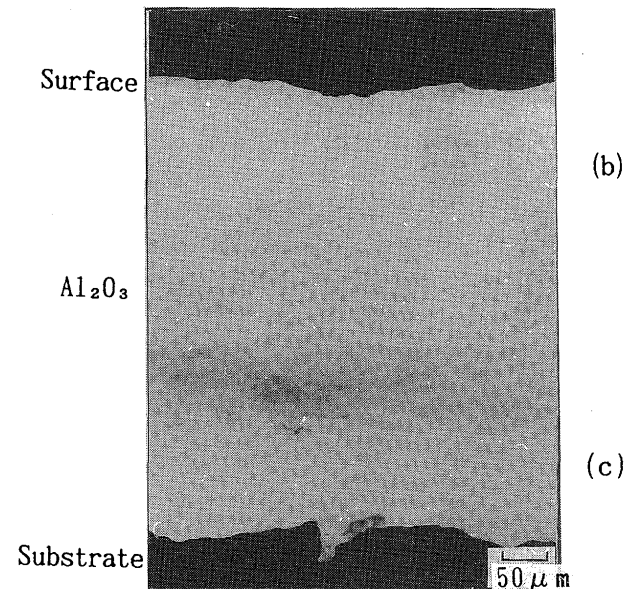


Fig.7 Photograph of cross section of alumina coating by polarizing microscope (cross Nicol) at  $L = 30\text{mm}$ , when  $P = 30\text{kW}$ .

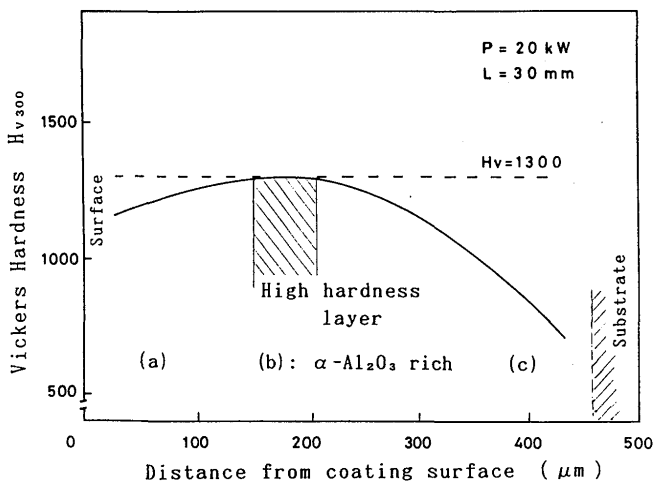


Fig.6 Distribution of Vickers hardness on cross section of alumina coating at  $L = 30\text{mm}$ , when  $P = 20\text{kW}$ .

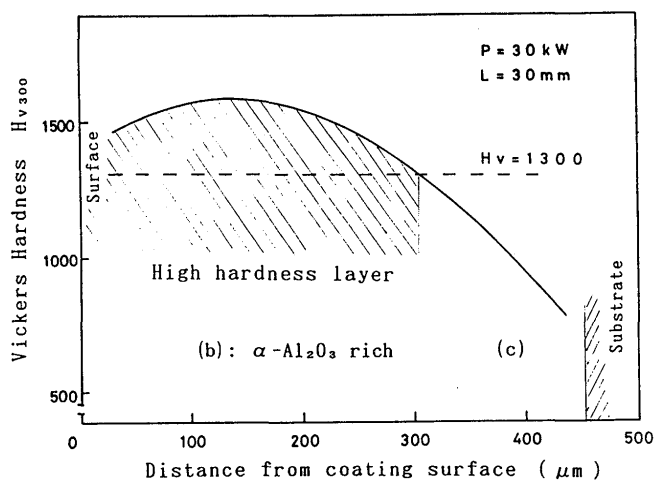


Fig.8 Distribution of Vickers hardness on cross section of alumina coating at  $L = 30\text{mm}$ , when  $P = 30\text{kW}$ .

the part (b) from the coating surface to  $2/3t_c$  ( $t_c$ : coating thickness), where the color is white. This shows that a wider  $\alpha$ -alumina rich layer was formed. It has been reported that in this part the cell size becomes finer.

Figure 8 shows the distribution of Vickers hardness on the cross section of this high hardness alumina coating. The distribution of Vickers hardness is also parabolic curve. However, the Vickers hardness near the coating surface is more higher than that at  $P=20\text{kW}$  (The highest value of Vickers hardness is  $H_V=1600$ ). And the width of the high hardness layer is more wider than that at  $P=20\text{kW}$ . This high hardness layer corresponds to  $\alpha$ -alumina rich layer (b) of Fig.7.

#### 4. Conclusion

It has been reported in the previous papers that by means of gas tunnel type plasma spraying, a high hardness alumina coating was formed at a short spraying distance. In this paper, the microstructure of this high hardness coating was investigated, and the relation to the Vickers hardness and so on were discussed.

The structure of high hardness alumina coatings formed by the gas tunnel type plasma spraying can be easily observed by using a reflected type polarizing optical microscope.

In the case of a short distance spraying, the  $\alpha$ -alumina rich layer was appeared at the surface side of the coating. And in this  $\alpha$ -alumina rich layer the cell size was very

fine by the heat from plasma.

These  $\alpha$ -alumina rich layer was increased in the width in the case of large power input. Comparing to the results of measurement of the distribution of the Vickers hardness, these  $\alpha$ -alumina rich layer corresponded to the high hardness layer near the surface.

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