# Influence of Substrate Surface Roughness on Synthesized Diamond Films by Flame Combustion on Ti Substrate for Dental Implants

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Abstract: The flame combustion method enables the synthesis of diamonds via acetylene-oxygen gas flame combustion in ambient air, and this method has various advantages over other methods. However, most diamond films synthesized by this method delaminate because of thermal stress during cooling. Titanium (Ti) has recently been utilized as a dental implant in the dental industry. In this study, to improve the strength, wear resistance, and biocompatibility of dental implant surfaces, diamond films were synthesized on a Ti substrate, a dental implant material, by the flame combustion method. Moreover, to obtain high-quality diamond films and achieve good adhesion on the Ti substrate, as a pretreatment of the substrate to prevent delamination, scratch processing, in which a substrate is ground with emery paper in one direction, was performed to roughen the surface. The surface roughness of the Ti substrates was varied by scratching with emery paper of #180, #400, and #1500 grain sizes. According to these results, diamond films were synthesized on the Ti substrate surface by flame combustion. The surface morphology of the synthesized films could be altered by varying the scratching process using emery paper. Delamination of the synthesized films during the scratching process with emery paper #180 (Case A) and #400 (Case B) grain size was completely prevented. However, delamination occurred during the scratching process with a grain size of emery paper #1500 (Case C). To investigate the reason for this result, the surface roughness of the pretreated Ti substrate was observed, and it affected the surface roughness of pretreated Ti substrate affected the surface morphology and delamination of the synthesized diamond films.

**Keywords:** Diamond films; Flame combustion; Titanium substrate for dental implant; Pretreatment of substrate; Surface roughness; Delamination.

## 1. Introduction

Owing to its excellent properties, i.e., high thermal conductivity, high hardness, and high wear resistance, diamond is utilized in the industry for the manufacture of cutting and polishing tools. Recently, diamond films have been considered as coating materials for dental cutting tools [1-3]. Thus, diamond has been extensively studied for use as a coating material in medical devices [4-6].

The flame combustion method enables the synthesis of diamond using acetylene-oxygen gas  $(C_2H_2/O_2)$  flame combustion in ambient air [7][8]. It has various advantages over other methods, such as high synthesis speed, safety, and low cost of equipment used, which are desirable in the industrial market. However, to date, the factors affecting diamond synthesis are unknown, and no means of precisely controlling this method has been established. Moreover, during cooling, most diamond films synthesized by the flame combustion method delaminate because of thermal stress. We previously synthesized diamond films on molybdenum (Mo) and tungsten carbide (WC) substrates using the flame combustion method [9–15]. Therefore, we focused on the synthesis of diamond films for medical devices using the flame combustion method, which has such an advantageous feature.

In the medical community, owing to the excellent properties of diamond, further application of diamond films in medical devices is required. Currently, the demand for dental implants is increasing owing to accidents, diseases, and the aging of the population [16][17]. Metals are typically used as dental implants. Titanium (Ti) has recently been used as a dental implant in the dental industry. However, there is an issue that dental implants corrode during use and are fractured by various loads. The failed implants must be discarded. In addition, implant materials have been reported to cause metal allergies, and the compatibility of metal materials with the human body is an issue.

In recent years, diamond films have been studied as Ti substrate [18-20]. The study of diamond films on metals for dental implants has garnered significant attention to address these challenges [21]. However, the experimental equipment utilized in the CVD method was very large, and the synthesis speed was very slow. We focused synthesis of diamond films on a Ti substrate using the flame combustion method [22].

In this study, it is considered that if diamond films can be directly synthesized on the Ti surface by flame combustion and good adhesion can be achieved, surface improvement in terms of high hardness, corrosion resistance, and ultimately in terms of the compatibility of metal materials with the human body can be realized. Therefore, to improve the strength, corrosion resistance, and biocompatibility of dental implant surfaces, diamond films were synthesized on a Ti substrate which is a dental implant material, by the flame combustion method using a mixture of acetylene and oxygen gas, and the possibility of synthesizing a diamond film on a Ti substrate was examined. In addition, to obtain high-quality diamond films and achieve good adhesion on the Ti substrate, we focused on the effect of pretreatments on the substrate surface. As a pretreatment of the substrate to prevent delamination, scratch processing, in which a substrate is ground with emery paper in one direction, was performed to roughen the surface. The surface roughness of the Ti substrate was varied by scratching with #180, #400, and #1500 emery papers. According to these results, films were synthesized on the Ti substrate surface by flame combustion. The films synthesized using this method were analyzed, and the results were discussed. The results proved that diamond films were deposited on the Ti substrate surfaces using this method. The surface morphology of the synthesized films could be altered by varying the scratching process using emery paper. Delamination of the synthesized films during the scratching process with emery paper of #180 and #400 grain sizes was completely prevented. However, delamination occurred owing to the scratching process with emery paper of #1500 grain size. Moreover, the effect of pretreatment of the substrate on the synthesized diamond films on the Ti substrate by flame combustion was investigated. To investigate the reason for this result, the surface roughness of a scratched Ti substrate was observed. The surface roughness of the scratched Ti substrate affected the surface morphology and delamination of the synthesized diamond films. The optimal pretreatment conditions were investigated and determined.

### 2. Experimental details

#### 2.1 Substrate

Recently, Titanium (Ti) and Ti alloys (Ti-6Al-4V) have been used as dental implants in the dental industry [16][21]. However, the toxicity of Vanadium (V) in Ti alloys (Ti-6Al-4V) has also been reported to affect the human body [23][24]. Therefore, in this study, Ti was used as a substrate for dental implant materials. Titanium (Ti) with 99.5% purity for dental implants was used as the substrate for synthesis diamond. The Ti substrate had a disk shape with a diameter of 10 mm and thickness of 3 mm. As a pretreatment to prevent delamination, scratch processing, in which the substrate surface is ground with emery paper in one direction, was used to roughen the surface. Furthermore, as growth nuclei for the diamond synthesis, diamond seed particles of approximately 0.25  $\mu$ m in diameter were dispersed in acetone, the Ti substrate was added, and seed attachment processing was performed for 30 min with an ultrasonic syringe. Many diamond seed particles were attached to the substrate surface by pretreatment.

#### 2.2 Experimental equipment

The experimental setup is illustrated in Figure 1. A  $100 \times 100 \times 55 \text{ mm}^3$  rectangular copper box was utilized for cooling. Cooling water was poured into the box and the film surface temperature was kept constant at 1203 K. A noncontact infrared radiation thermometer was utilized to measure the film surface temperature during synthesis. As a support for cooling, a copper (Cu) rod with a diameter of 10 mm was set vertically at the center of the box and fixed to a table using a flange. The Ti substrate was then attached to this Cu rod. For efficient cooling, thermally conductive Ag paste was applied between the Ti substrate and Cu rod. They were then glued to a furnace at 473 K.

A cooling box was placed on the stage. Because it was capable of moving vertically, the distance from the cooling water side to the film surface was changed, and the film surface temperature was controlled. A stepping motor was set on the stage and controlled using a stage controller.

Acetylene and oxygen gas were used as the fuels for the synthesis. A burner was utilized for welding. A mixed gas was introduced into the burner and combusted. The burner exit diameter was 1 mm. In addition, a mass flow controller was utilized as the gas flow meter, which could precisely control the gas flow rate and digitally display the flow quantity.

### **2.3 Synthetic conditions**

The synthetic conditions are presented in Table 1. These conditions have been determined and reported previously [7–10], and are believed to be the optimum conditions for preventing delamination during the synthesis

of diamond films. The ratio of the oxygen flow rate  $F_0$  to acetylene flow rate  $F_a$  was set to  $R_f = F_0/F_a = 0.90$ , because delamination-free crystallite growth could be realized at  $R_f = 0.90$  [10].

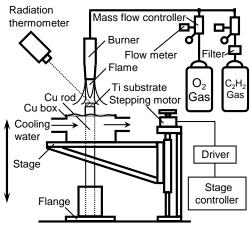


Figure 1. Experimental setup for synthesizing diamond by acetylene-oxygen flame combustion.

An outline of the flame combustion is illustrated in Figure 2. Flame combustion comprises a flame inner cone, acetylene feather, and an outer luminous layer. Diamond was synthesized using an acetylene feather. The distance, d, of the flame inner cone from the Ti substrate surface is illustrated in the figure. During the synthesis, when the distance was altered, diamond film synthesis and delamination were affected because the acetylene feather area changed. When the distance was short, the diamond growth rate was high, and when the distance was farther, the diamond growth rate was low. We confirmed that delamination at d = 1.5 mm during diamond film synthesis could be effectively prevented [10][11]. Therefore, the diamond films were synthesized at d = 1.5 mm. The total synthesis time was set to 3600 s [10][11]. The average thickness of synthesized films was 30 µm/h in this method. In this method, the film surface temperature was set to 1203 K during synthesis.

We focused on the effect of surface roughness caused by pretreatments on the substrate surface. Therefore, diamond films should be synthesized on the pretreated substrate surfaces.

As a pretreatment to prevent delamination, scratch processing, in which the substrate surface is ground with emery paper in one direction, was performed. The Ti substrate was pretreated by scratching to roughen its surface. The surface roughness of the Ti substrate was varied by scratching with #180, #400, and #1500 emery papers. In this study, the synthesized films were deposited on pretreated Ti substrates of the scratching process with emery paper of grain sizes #180, #400, and #1500. The pretreatment conditions are presented in Table 2.

Table 1. Conditions for diamond syntheses.				
Reaction gas	$C_2H_2 + O_2$			
Film surface temperature	1203 [K]			
Inner cone-to-substrate distances	1.5 [mm]			
Pure $C_2H_2$ Flow rate, $F_a$	70.9 [cm <sup>3</sup> /s]			
$O_2$ Flow rate, $F_0$	63.8 [cm <sup>3</sup> /s]			
Flow ratio, $R_{\rm f} = F_{\rm o} / F_{\rm a}$	0.90			

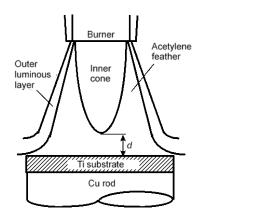


Figure 2. Outline figure of flame combustion.

### 2.4 Pretreatment of the Ti substrate

We observed that surface roughness caused by pretreatments on the substrate surface affects delamination. Therefore, diamond films should be synthesized on pretreated substrate surfaces.

As a pretreatment to prevent delamination, scratch processing, in which the substrate surface is ground with emery paper in one direction, was performed. The Ti substrate was pretreated by scratching to roughen its surface. The surface roughness of the Ti substrate was varied by scratching with #180, #400, and #1500 emery papers. In this study, the synthesized films were deposited on pretreated Ti substrates of the scratching process with emery paper of grain sizes #180, #400, and #1500. The pretreatment conditions are presented in Table 2.

Table 2. Conditions for pretreatment of Ti substrate surface.					
	Case A	Case B	Case C		
Emery paper grain size level	#180	#400	#1500		

# 3. Results and discussion

#### 3.1 Delamination of synthesized films

Delamination of the synthesized films during the scratching process with emery paper #180 (Case A) and #400 (Case B) grain sizes was completely prevented. In this regard, the following reasons were given. The substrate surfaces were scratched using emery papers, which effectively prevented delamination. As a pretreatment to prevent delamination, a scratching treatment in which the substrate is scratched with emery paper, was used to roughen the surface, and the substrate surface conditions were optimal for preventing delamination. However, delamination occurred during the scratching process with emery paper #1500 (Case C). We considered that the pretreatment affected the delamination of the synthesized films.

#### 3.2 Investigation of synthesized films

The films synthesized under various pretreatment conditions were analyzed by scanning electron microscopy (SEM, JEOL JSM-7800F) and X-ray diffraction (XRD, Rigaku RINT-2200V) analysis. SEM images of the films synthesized in Case A (Emery paper #180) are illustrated in Figure 3. In the SEM image, the synthesized crystallites indicate no uniform size or low density. The crystallites of the synthesized film on the scratched surface were directed by the scratching direction of the white arrow. The XRD patterns of Case A are illustrated in Figure 4, where the peaks indicating the existence of diamond and the Ti substrate were confirmed. Peaks indicating the existence of diamond (111) and (220) surfaces were confirmed. The peak of the diamond (111) surface exhibit a crystal growth direction at the top of the octahedral morphology. SEM images of the films synthesized in Case B (Emery paper #400) is illustrated in Figure 5. In the SEM images, the synthesized crystallites indicated a nearly uniform size and high density. Thus, we considered that the pretreatment conditions affected the diamond synthesis. The XRD patterns of Case B are illustrated in Figure 6, where the peaks indicating the existence of diamond and the Ti substrate were confirmed. Peaks indicating the existence of diamond (111) and (220) surfaces were confirmed as well. The SEM images of the films synthesized in Case C (Emery paper #1500) are presented in Figure 7. Delamination of the synthesized films occurred in Case C. However, the synthesized film remained on the Ti substrate after synthesis; therefore, a residual synthesized film was observed. In the SEM image, the synthesized crystallites indicate no uniform size or low density. Thus, we considered that the pretreatment conditions affected the diamond synthesis. The XRD patterns of Case C are illustrated in Figure 8, where the peaks indicating the existence of diamond and the Ti substrate were confirmed. Peaks indicating the existence of diamond (111) and (220) surfaces were confirmed as well. From the SEM images and XRD patterns of Cases A, B, and C, diamond crystallites were synthesized, and diamond films were synthesized on scratched substrates. In the XRD patterns of Cases A, B, and C, the peak of the diamond (111) surface was very distinct. The synthesized diamond crystallites exhibited an approximately octahedral morphology and thus were considered good quality diamond crystallites. In the SEM images of Cases A, B, and C, the average sizes of the synthesized diamond crystallites were 25.5, 19.4, and 17.4 µm in diameter, in Cases A, B, and C, respectively. The surface morphology of the synthesized films could be altered by varying the scratching process with emery paper of grain sizes #180, #400, and #1500. It can be suggested that the use of EDAX analysis in the characterization of synthesized films should be considered in future investigations.

In this study, delamination of the synthesized films during the scratching process in Cases A and B was prevented. However, delamination of the synthesized films during the scratching process in Case C occurred. To discuss the delamination of the synthesized films, the residual area rates of the synthesized films on the substrate were measured. We considered that the optimal synthesis occurred in the central area of the synthesized films on the Ti substrate. Therefore, a circular area with 7 mm of diameter of synthesized films were the evaluation area. The measured residual area rates of the synthesized films on the substrates are presented in Table 3. In Cases A

and B, measured residual area rates of synthesized films were 100%. Therefore, delamination of the synthesized films during the scratching process in Cases A and B is completely prevented. However, in Case C, the measured residual area rate of synthesized films was 27.6%. More than half of the synthesized films were delaminated in this region. The pretreatment of the scratching process affects the delamination of diamond synthesis on the substrate.

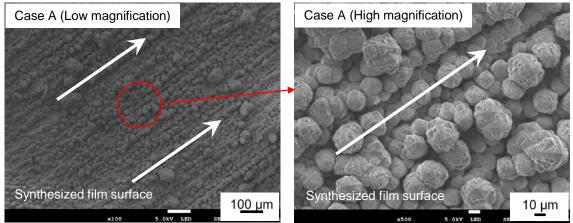


Figure 3. SEM image of the synthesized diamond film in Case A.

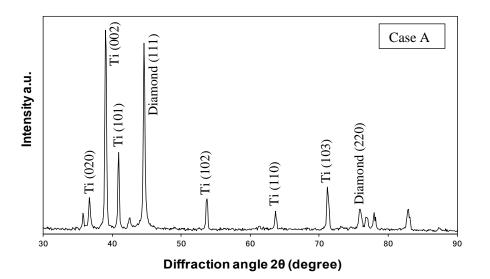


Figure 4. XRD patterns of the synthesized diamond film in Case A.

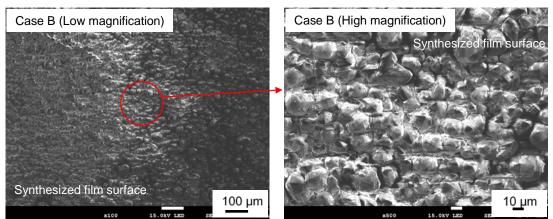


Figure 5. SEM image of the synthesized diamond film in Case B.

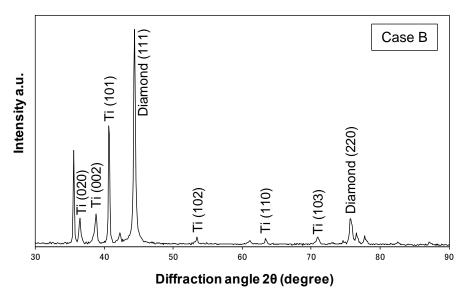


Figure 6. XRD patterns of the synthesized diamond film in Case B.

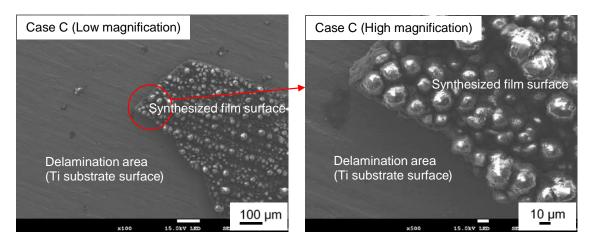


Figure 7. SEM image of the synthesized diamond film in Case C.

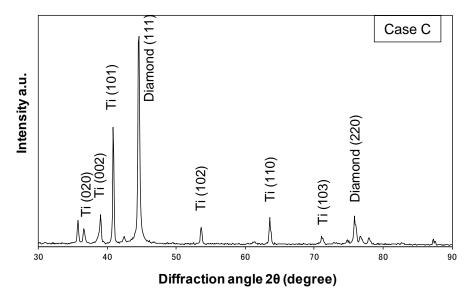


Figure 8. XRD patterns of the synthesized diamond film in Case C.

Table 3.	Residual	area ra	ates of	synthesized	l films o	n Ti sut	ostrate.
				Case	A C	ase B	Case C

Case A	Case B	Case C
#180	#400	#1500
100%	100%	27.6%
	#180	#180 #400

Diamond films on the Ti substrate were obtained using a mixture of acetylene and oxygen gas. The Ti substrate was pretreated by the scratching process with emery paper to roughen the surface. Pretreatment affected the delamination and morphology of diamond synthesis on the substrate. We considered that the reason for this result is the change in surface roughness with the scratching process. The surface roughness for each scratching process was measured using a scanning white-light interferometer (SWLI, Zygo New View6K). The measured surface conditions of the pretreated Ti substrates are illustrated in Figure 9. The relationship between the measured surface roughness Ra (arithmetical mean deviation of the assessed profile) and the emery paper grain size level is illustrated in Figure 10. The error bars in Figure 10 are illustrated because of the slight variation in the surface roughness, and the standard deviation is indicated. The mean values of the measured surface roughness for Cases A, B, and C were 0.370, 0.298, and 0.277 µm, respectively. From the results, it can be confirmed that the mean value of the measured surface roughness changes in each Case, and the measured surface roughness decreases as the emery paper grain size decreases from Cases A to C. The morphology and delamination of the synthesized diamond films were affected by varying the surface roughness of the Ti substrate. Delamination of the synthesized films during the scratching process in Cases A and B was completely prevented. In Case B, the synthesized crystallites indicated a nearly uniform size and high density. Therefore, in Case B, the surface roughness of the Ti substrate was a good condition for diamond crystallites for synthesis and non-delamination. In this experiment, Case B was the optimal condition.

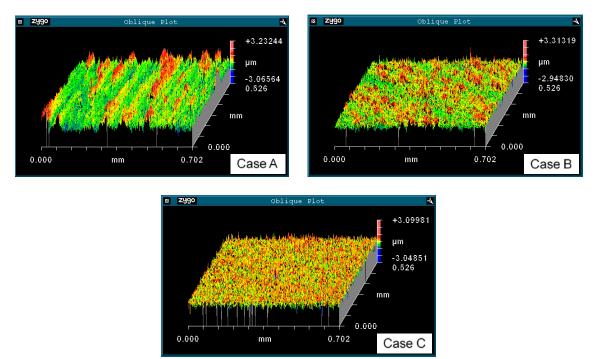


Figure 9. Surface condition of pretreated Ti substrates in Cases A, B, and C.

### 3.3 Discussion

The SEM images illustrate that the diamond morphology of the synthesized diamond films was affected by varying the surface roughness of the Ti substrate. Delamination of the synthesized films during the scratching process in Cases A and B was completely prevented. However, delamination of the synthesized films during the scratching process in Case C occurred. In Cases A and B, measured residual area rates of synthesized films on the substrate were 100%. However, in Case C, the measured residual area rate of the synthesized films was 27.6%. The pretreatment of the scratching process affects the delamination of diamond synthesis on the substrate. The measured surface roughness (arithmetic mean roughness:  $R_a$ ) values of Cases A, B, and C were 0.370, 0.298 and 0.277 µm, respectively. As the emery paper grain size decreased, the measured surface roughness ( $R_a$ ) decreased. We considered that the conditions for pretreatment and surface roughness affected the diamond synthesis on the

substrate and the delamination of the synthesized films. Therefore, from Figures 9 and 10, we discussed the cause of the diamond morphology difference and the delamination of the synthesized diamond films by the surface roughness of the Ti substrate during the scratching process in Cases A, B, and C.

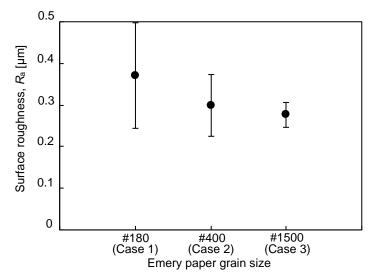


Figure 10. Surface roughness (Ra) of pretreated Ti substrate surface.

Here, as the emery paper grain size level increased, the height difference between the mountaintop and the bottom of the valley of the scratched substrate surface was expected to be larger, and the spacing between the mountaintops was expected to be wider. In addition, as the emery paper grain size level decreased, the height difference between the mountaintop and bottom of the valley was expected to be smaller, and the spacing between the mountaintops was expected to be narrower. The basic concept of the state of the scratched surface and state of nucleation for each case condition are illustrated in Figure 11. In Case A, the synthesized crystallites indicated no uniform size or low density. From Figure 3, the crystallites of the synthesized film on the scratched surface were directed by the scratching direction of the white arrow. This is because the surface roughness during the scratching process in Case A was the largest among all cases. As illustrated in Figure 11 (a), in Case A, because of the large difference in height between the mountaintop and the bottom of the valley created by the scratching process and the wide spacing between the mountaintops, new nuclei were generated at the mountaintop during the growth of the nuclei at the bottom of the valley during synthesis. In addition, for diamond synthesis, the difference in height between the mountaintop, the bottom of the valley, and the spacing between the mountaintops of the substrate surface were not appropriate; thus, the growth energy of the nuclei for the nuclei produced was not moderately dispersed over the nuclei, resulting in the synthesized crystallites showing no uniform size and low density. In Case B, the synthesized crystallites indicated an approximately uniform size and high density. As illustrated in Figure 11 (b), this is because the height difference between the mountaintop, bottom of the valley of the substrate, and the spacing between the mountaintops were appropriate, and the nucleation was homogeneous at the mountaintop and bottom of the valley of the substrate. The growth energy of the nuclei relative to the generative nuclei was moderately dispersed over several nuclei, resulting in synthesized crystallites exhibiting uniform size and high density. In Case C, the synthesized crystallites exhibited no uniform size or low density. This is because the surface roughness during the scratching process in Case C was the smallest among all cases. As illustrated in Figure 11 (c), in Case C, because the difference in height between the mountaintop and the bottom of the valley of the substrate surface becomes smaller, and the spacing between the mountaintops is narrower, the nuclei are less likely to be produced at the bottom of the valley, and more nuclei are produced at the mountaintop and grow there. In addition, because the difference in height between the mountaintop, bottom of the valley, and the spacing between the mountaintops of the substrate surface were not appropriate, the growth energy of the nuclei for the produced nuclei was not moderately dispersed over the nuclei, resulting in synthesized crystallites exhibiting no uniform size and low density.

Delamination of the synthesized films during the scratching process in Cases A and B was completely prevented. For delamination, in Cases A and B, the height difference between the mountaintop, bottom of the valley of the substrate, and the spacing between the mountaintops were appropriate. The contact area with the substrate increased because of the combination of each nucleus after the growth of the generated nuclei; therefore, the bond strength increased. Delamination of the synthesized films during the scratching process in Case C occurred. For delamination, in Case C, the height difference between the mountaintop, bottom of the valley of the substrate, and the spacing between the mountaintops were not appropriate. The difference in height between the mountaintop

and bottom of the valley of the substrate surface was negligible, and the spacing between the mountaintops was narrow. Therefore, nuclei are less likely to be produced at the bottom of the valley, and more nuclei are produced at the mountaintops and grow there. It is considered that the number of nuclei in contact with the bottom of the valley of the substrate surface was negligible, and the films were delaminated in Case C.

Therefore, it was determined that the change in the roughness of the substrate surface by the scratching treatment during the pretreatment affected the diamond synthesis on the substrate and the delamination of the synthesized films. In Case B, the surface roughness of the Ti substrate was a good condition for diamond crystallites for synthesis and non-delamination. In this experiment, Case B was the optimal condition.

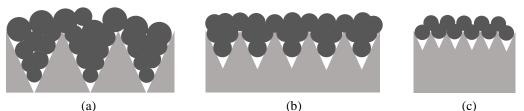


Figure 11. Cross-section morphologies of nucleation of diamond in Cases A, B, and C.

# 4. Conclusions

In this study, diamond films were synthesized on a Ti substrate, which is a dental implant material, to improve the strength, wear resistance, and biocompatibility of dental implant surfaces, by the flame combustion method using a mixture of acetylene and oxygen gas. The possibility of synthesizing a diamond film on a Ti substrate was examined. The aim was to obtain diamond films and achieve good adhesion on Ti substrates for dental implants. The Ti substrate was pretreated using a scratching process with emery paper to roughen the surface. The effect of pretreatment of the substrate on synthesized diamond films on the Ti substrate by flame combustion was investigated.

The diamond films on the pretreated Ti substrate were obtained using a mixture of acetylene and oxygen gas.
The surface morphology of synthesized films could be altered by varying scratching process with emery paper.

3) Delamination of the synthesized films during the scratching process with emery paper of #180 and #400 grain sizes (Cases A and B) was completely prevented.

4) Pretreatment of the scratching process affected diamond synthesis on the substrate. The delamination and diamond morphology of the synthesized diamond films were affected by varying the surface roughness of the Ti substrates.

5) Therefore, in Case B, the surface roughness of the Ti substrate was a good condition for diamond crystallites for synthesis and non-delamination. In this experiment, Case B was the optimal condition.

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