ISBN: 978-3-95450-233-2 ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-THPFDV008

RESEARCH ON CERAMIC FOR RF WINDOW

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Abstract

Kyocera and KEK have started joint research on the development of materials that satisfy the required characteristics as RF window material. In this report, the characteristics of the new material AO479U were evaluated by comparing it with other materials, including the presence or absence of Titanium-Nitride (TiN) coating. In order to clarify the influence of materials or its manufacturing processes on heat generation and multipactor discharge generated in RF windows, we measured important characteristics as RF window material (relative permittivity, dielectric loss tangent, surface resistivity, volume resistivity, secondary electron emission coefficient, and TiN thickness), and investigated their correlation.

INTRODUCTION

When accelerating charged particles in an accelerator, an alumina ceramic window is used as a partition of a waveguide to put microwaves generated in klystron into accelerating cavity. In previous studies, the alumina material AO479B had been developed for RF window material, and it has been applied to some products, however, AO479B has a size limitation. Recently, large RF windows is required from the market. Therefore, we have developed a new material AO479U which can be designed regardless of the product size. As required characteristics of the RF window, there are dielectric loss tangent (tan δ) and secondary electron emission coefficient (δ_{SEE}). Low tanδ is required to suppress heat generation in high power RF operation. Low δ_{SEE} is also required to suppress multipactor discharge on the ceramic surface. In addition, relative permittivity (ε), surface resistivity (ρ_s), and volume resistivity (ρ_v) were also measured.

RESEARCH ON SECONDARY ELECTRON EMISSION COEFFICIENT

The secondary electron emission coefficient on ceramic surface is the most important parameter to be evaluated by the effect of multipactor discharge in high power RF operation. A scanning electron microscope (SEM) with beam blanking system which can generate a pulse beam was installed at KEK for δ_{SEE} measurement in 2018. Since Alumina is an insulator material, it essentially uses a pulse beam to avoid charge-up on the ceramic surface. The specifications of this measurement system are described in these references [1, 2]. Table 1 shows four types of ceramic samples with different manufacturing processes. The coating was conducted by Company A (TiN A) and Company B (TiN B), respectively. The heat treatment (HT) was carried out under the same conditions as the brazing process for the accelerator manufacturing conducting at 1000 °C and 800 °C in the furnace of Canon Electron Tubes & Devices Co., Ltd. (CETD). Figure 1 shows the ceramic samples with diameter of 19 mm, without coating samples and with two different TiN coating samples. The samples for δ_{SEE} evaluation were prepared by a process equivalent to the accelerator cavity manufacturing process conditions, and the effects of δ_{SEE} were investigated under various additional process conditions. The charge of ceramic samples were measured using an electrometer, and all the samples were charged to several volts after measured the δ_{SEE}. Figure 2 shows summary of secondary electron emission coefficient for ceramic samples without TiN coatings, and Fig. 3 shows that with TiN coatings. Figures 2 and 3 show the following results:

- The TiN coating significantly reduced the δ_{SEE}.
- The measured values tended to be unstable without ethanol ultrasonic rinsing (USR).
- The δ_{SEE} tended to increase in the samples with and without TiN coating after heat treatment.
- There was a difference in the δ_{SEE} of the sample by the TiN coating company.
- There was no significant difference between ethanol ultrasonic rinsing and ozonized (O_3) water rinsing.
- The δ_{SEE} varied depending on the material, however, was almost same regardless of the TiN coated material.

Table 1: Ceramic Sample List for δSEE Measurement

Material	Coating	Heat treatment (°C)	Rinsing	#
AO479U	Free	No	No/USR	1/2
AO479U	Free	1000	USR	3
AO479U	Free	800	USR	3
AO479U	Free	1000→800	USR	3
AO479U	Free	1000→800	O_3	3
AO479U	TiN A	No	USR	3
AO479U	TiN A	1000	No/USR	1/2
AO479U	TiN A	1000→800	No/USR	1/1
AO479U	TiN A	1000→800	O_3	1
AO479U	TiN B	No	USR	3
AO479B	Free	No	No/USR	1/2
AO479B	TiN A	No	No/USR	1/2
AO473A	Free	No	No/USR	1/1
AO473A	TiN A	No	USR	3
HA95	Free	No	USR	1
HA95	TiN A	No	USR	2

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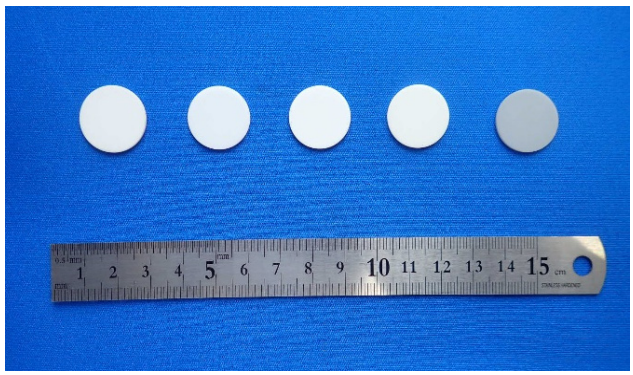

Figure 1: Ceramic samples (AO473A without coating, AO479B without coating, AO479U without coating, AO479U with TiN A coating, and AO479U with TiN B coating from left to right) for secondary electron emission coefficient measurement.

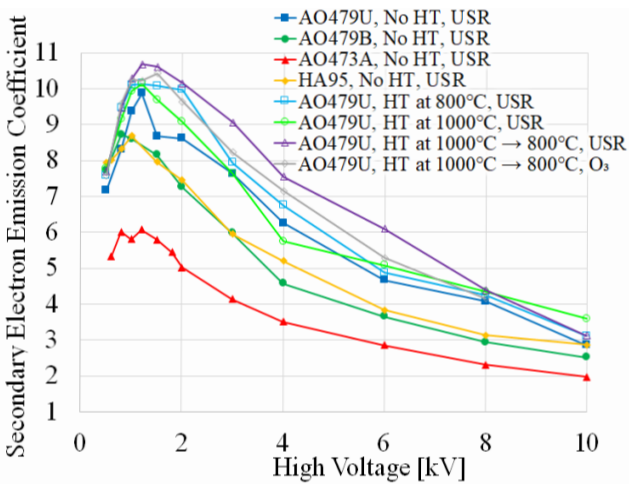

Figure 2: Summary of secondary electron emission coefficient for ceramic samples without TiN coatings.

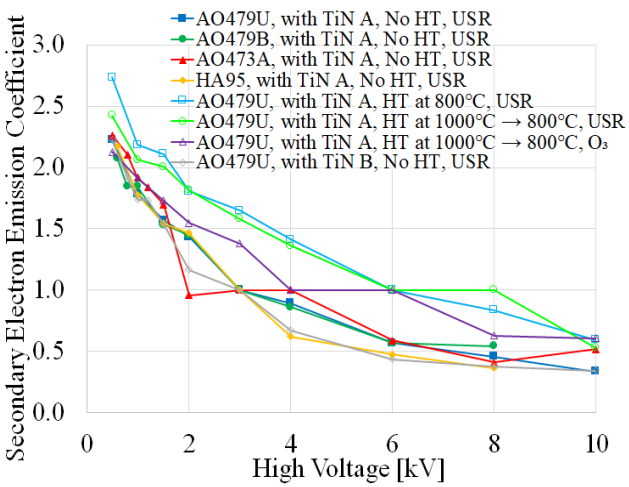

Figure 3: Summary of secondary electron emission coefficient for ceramic samples with TiN coatings.

RESEARCH ON RELATIVE PERMITTIVITY AND DIELECTRIC LOSS TANGENT

The relative permittivity (ε) and dielectric loss tangent (tanδ) of four kinds of ceramics were measured at AET [3], Kyocera, and KEK. Table 2 shows the ceramic sample list for ε and tanδ measurement at AET, and Fig. 4 shows the four samples with the size of 1 mm x 3 mm x 80 mm. AO479U and AO479B contain aluminium oxide more than 99%, and AO473A contains 93% aluminium oxide. The measurement principle is described in reference [3]. The measurement mode is TM_{010}, and the calculation included corrections for differences of sample size. The measurements were made at 1 GHz and 2 GHz, and the average value was calculated from three to five measured data per one sample. Figures 5 and 6 shows the results of this measurement values for each ceramic. The result of relative permittivity depended on a purity of each ceramic. There was no significant difference between no heat treatment and after heat treatment. The result of tanδ of AO479U was equivalent to that of AO479B used for RF window.

Table 2: Ceramic Sample List for ε and Tanδ Measurement

Material	Coating	Heat treatment (°C)	#
AO479B	Free	No	5
AO479U	Free	No	3
AO479U	Free	1000→800	3
AO473A	Free	No	1

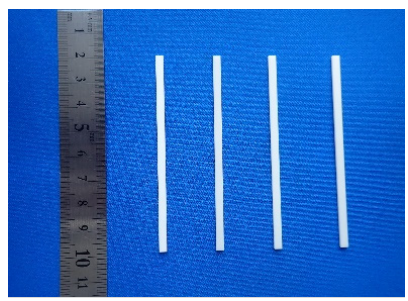

Figure 4: Ceramic samples (AO473A, AO479B, AO479U, and AO479U after heat treatment from left to right) for relative permittivity and dielectric loss tangent measurement.

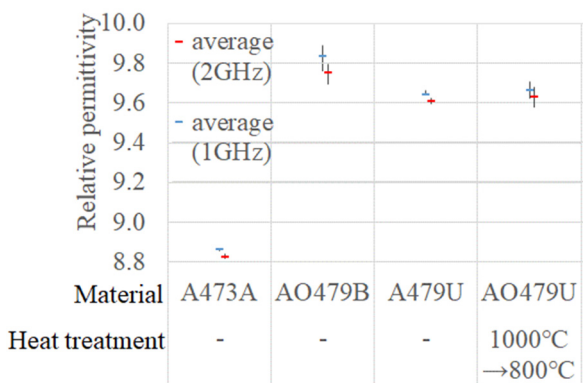

Figure 5: Relative permittivity for four ceramics.

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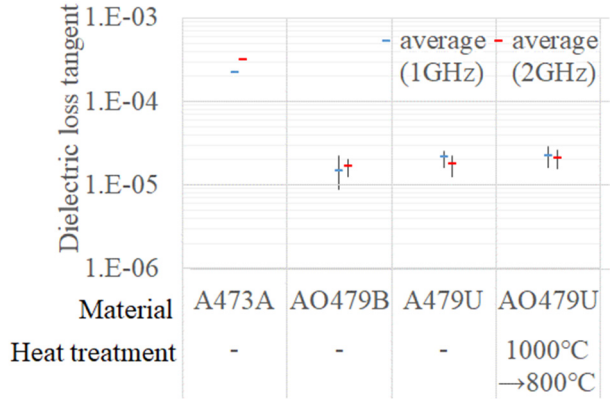

Figure 6: Dielectric loss tangent for four ceramics.

The relative permittivity and tanδ of AO479U at 1.3 GHz were measured at Kyocera, in order to be expected to use it for the RF window of ILC. The measurement is the dielectric resonator method. The measurement principle is described in reference [4]. Figure 7 shows the sample with the size of 102.5 mm (diameter) x 51.3 mm (thickness). The average value was calculated from two measured data per one sample. Figure 8 shows schematic diagram of measurement system. Figures 9 and 10 shows the results of this measurements including previous measurement results at 1 GHz and 2 GHz. The results of tanδ of AO479U at 1.3 GHz is equivalent to those at 1 GHz and 2 GHz.

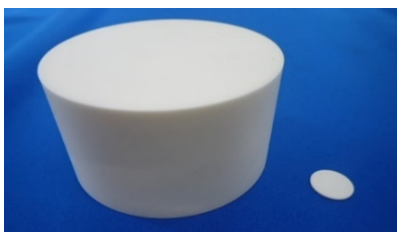

Figure 7: Ceramic samples of AO479U (The left side is the sample for relative permittivity and dielectric loss tangent measurement, and right side is a sample for secondary electron emission coefficient measurement).

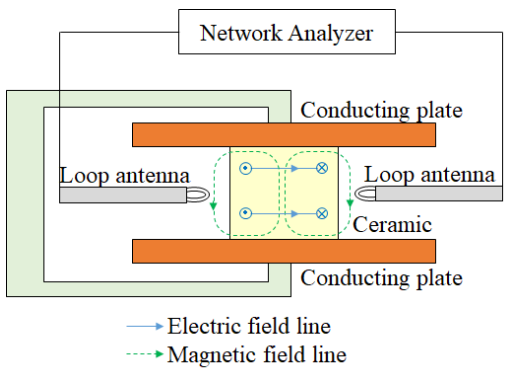

Figure 8: Schematic diagram of measurement system.

The relative permittivity and tanδ of AO479U at 3.4 GHz were measured with S-band Cavity at KEK. The results of tanδ of AO479U at 3.4 GHz is equivalent to those at 1 GHz and 2 GHz.

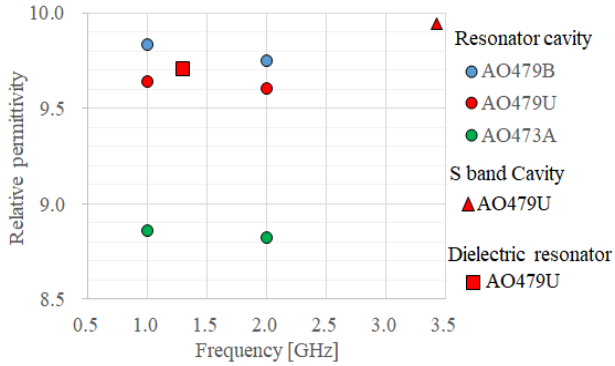

Figure 9: Relative permittivity of A479U.

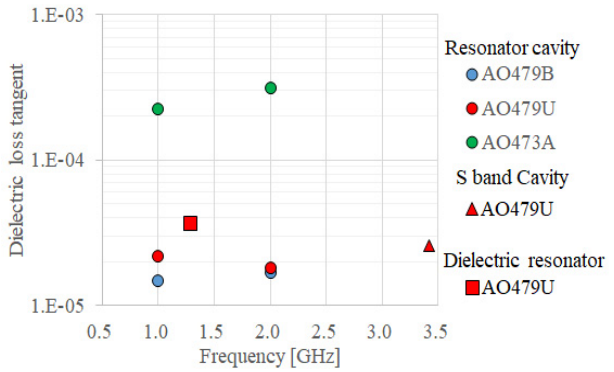

Figure 10: Dielectric loss tangent of A479U.

RESEARCH ON SURFACE AND VOLUME RESISTIVITY

The surface and volume resistivity were measured at JFCC [5], using five ceramic samples with diameter of 19 mm as shown in Fig. 11. Table 3 shows the ceramic sample list for ρ_s and ρ_v measurement. At JFCC, the electrodes were burn-in on the ceramic surface. The measurement principle is described in reference [2]. The applied voltage is 1 kV, and the applied stress is 10kgf. The baking process was carried out at 120°C for 2hours before and after each measurement process. Each measurement time is 1 hour. Figures 12 and 13 shows the results the surface and volume resistivity. The volume resistivity of each ceramic was from 10^{17} to 10^{18} Ω•cm. The surface resistivity measured in 2020 was 10^{15} Ω/□. On the other hand, the surface resistivity measured in 2018 was from 10^{15} to 10^{18} Ω/□. It is necessary to improve the measurement method of surface resistivity.

Table 3: Ceramic Sample List for ρ_s and ρ_v Measurement

Material	Coating	Heat treatment (°C)	#
HA95	Free/ TiN A	No	1/1
AO479B	Free	No	1
AO473A	Free/ TiN A	No	1/1
AO479U	Free/ TiN A	No	1/1
AO479U	Free	1000→800	1

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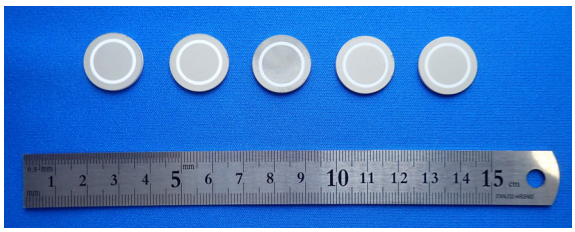

Figure 11: Ceramic samples (AO473A without coating, AO473A with TiN A coating, AO479U without coating, AO479U with TiN A coating, and AO479U without coating with heat treatment from left to right) for surface and volume resistivity measurement.

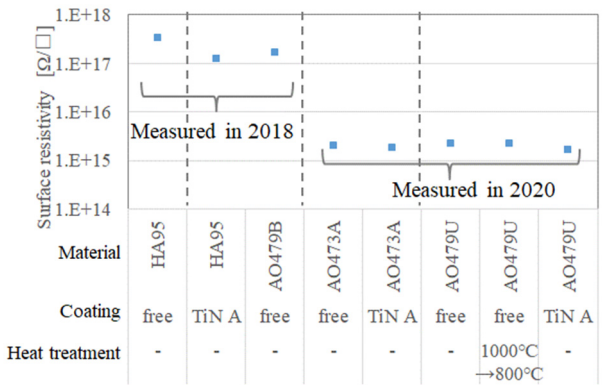

Figure 12: Surface resistivity measurement value of eight ceramics.

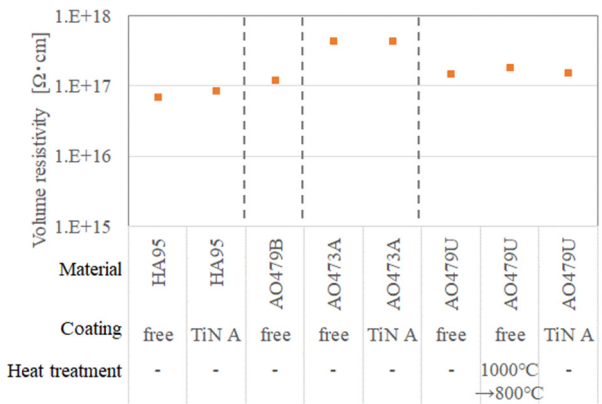

Figure 13: Volume resistivity measurement value of eight ceramics.

RESEARCH ON THE THICKNESS OF TIN

The thickness of TiN was measured by X-ray photoelectron spectroscopy (XPS) and argon ion (Ar^+) sputtering at Kyocera. Figure 14 shows schematic diagram of measurement system. XPS is a method for analysing the elemental composition in a few nm depth from the surface and the depth profile analysis can be analysed by XPS with ion sputtering method. The ion species is Ar^+, the sputter etching rate is 9 nm/min, and the thickness is converted into the SiO_2 thermal oxide film thickness. Table 4 shows the ceramic sample list for depth profile measurement, and Figures 15, 16, 17, and 18 shows the results for the depth profile of ceramic with TiN coating at company A, B, C, and D. The Titanium (Ti) atom concentration gradually decreases and the Aluminium (Al) atom concentration gradually increases as the sputtering depth increases. Since the ceramic samples having surface roughness of about 0.5 µm Ra are coated with TiN having a thickness of several tens nm, there are areas which are difficult to be shaved by sputtering due to the unevenness of ceramic surface. Therefore, atomic concentrations of Ti and Al changed gradually, the boundary between TiN and ceramic was not clear. When a thickness at half of the maximum concentration of Ti was used as the thickness of TiN coating, the thickness of Company A was 9 µm and that of Company B was 21 µm and that of Company C was 27 µm and that of Company D was 3 µm. TiN coating made by Company A is considered to contain a very low amount of Nitride (N). Figure 19 shows the thickness of TiN coating and δ_{SEE}. The thicker the TiN coating, the lower the δ_{SEE}.

Table 4: Ceramic Sample List for Depth Profile Measurement

Material	Coating	#
AO479U	Company A	1
AO479U	Company B	1
Ceramic C	Company C	1
Ceramic D	Company D	1

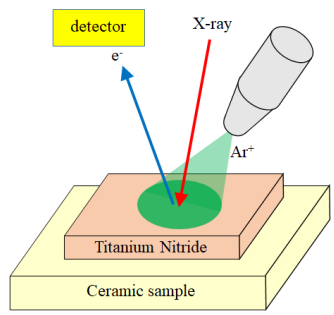

Figure 14: Schematic diagram of measurement system.

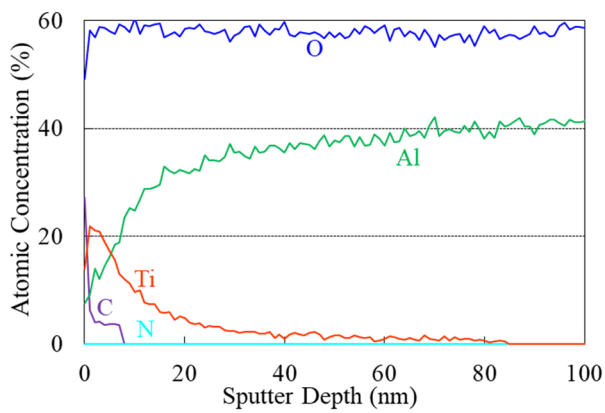

Figure 15: The depth profile of AO479U with TiN coating at company A.

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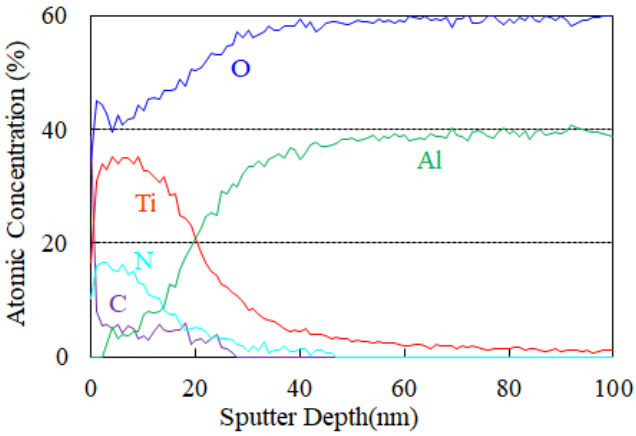

Figure 16: The depth profile of AO479U with TiN coating at company B.

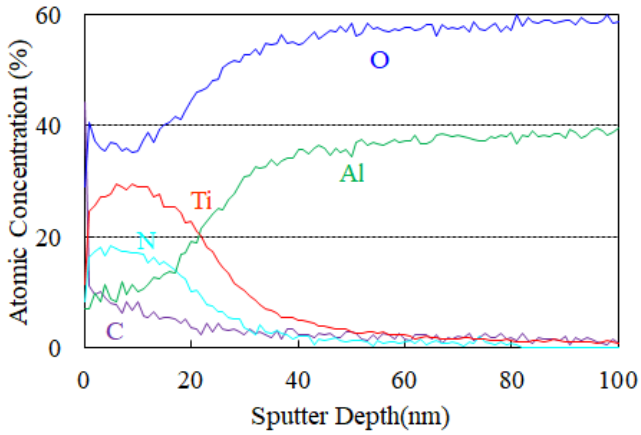

Figure 17: The depth profile of ceramic with TiN coating at company C.

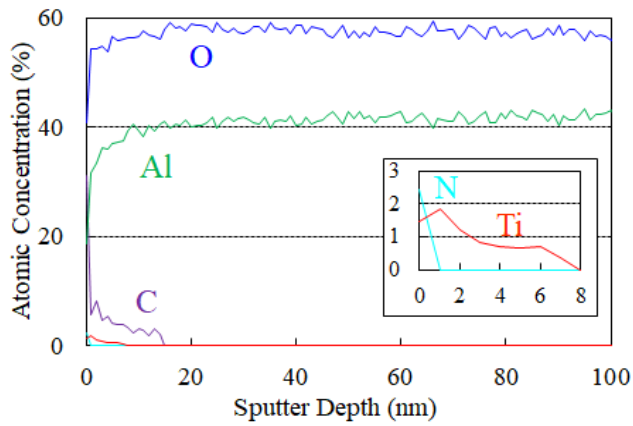

Figure 18: The depth profile of ceramic with TiN coating at company D.

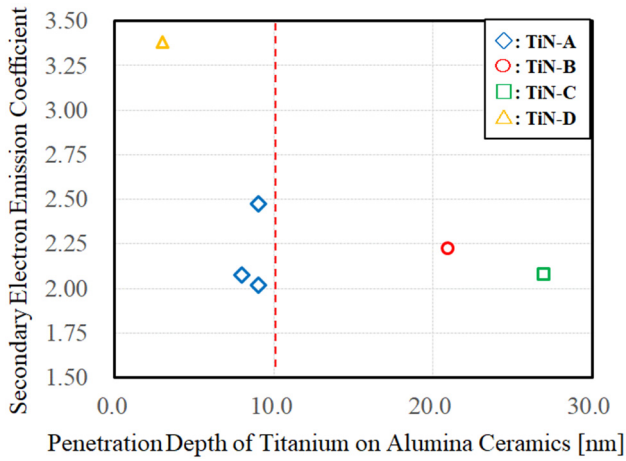

Figure 19: Thickness of TiN coating and δ_{SEE}.

CONCLUSION

The secondary electron emission coefficient decreased by TiN coating and increased by heat treatment equivalent to brazing. The relative permittivity was affected by the purity of the aluminium oxide. The dielectric loss tangent of AO479B was equivalent to that of AO479U. In the future, joint research with KEK is scheduled to investigate the cause of the high secondary electron emission coefficient due to the brazing process.

ACKNOWLEDGEMENTS

The authors would like to thank H. Sakai (KEK) for rinsing ceramic samples with ozonized (O_3) water; K. Yoshino (KEK) for providing data of relative permittivity and tanδ of AO479U at 3.4 GHz; H. Takahashi, K. Tetsuka (Canon Electron Tube & Devices Co., Led.) for heat treatment ceramic samples.

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