Annealing Effects on Breakdown Threshold of Radio-frequency Windows

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The breakdown threshold of alumina rf windows depends not only on the bulk characteristics, but also on surface treatments such as polishing and annealing. Sapphire and alumina-ceramic disks, polished and/or annealed, were examined by high-power tests. The polished disk exhibited a lower threshold value than the annealed one, probably due to its surface charging at defects mechanically introduced by polishing. The elimination of the charged sites present on the surface is considered to be important.

1. Introduction

The breakdown of alumina rf windows is one of the most serious problems in the development of high-power klystrons. Alumina ceramics have been used as rf-window materials^{1,2}, and several extensive studies of breakdown phenomena have been carried out³⁻⁵. The results are summarized below.

(1) As a pre-breakdown phenomenon, multipactoring due to a high secondary-electron-emission (SEE) coefficient (δ) took place together with optical emission (luminescence of alumina) and X-ray radiation. The multipactoring electron has a kinetic energy of about 7 keV at a power of 30 MW for a S-band klystron.

(2) Multipactoring induced F-center defects in alumina, which caused rf losses leading to breakdowns such as melting.

(3) Alumina ceramics having low dielectric losses were superior for rf-window materials because they were confirmed not to be liable for an F-center.

(4) Thin-film coatings having a low δ , such as TiN, on alumina ceramics, were confirmed to be effective for the suppression of multipactoring. However, occasional selective flashovers on a TiN-coated surface were observed, accompanied by tree like patterns of alumina luminescence.

A flashover is probably related to surface charging

during rf operation. These charges are generally considered to exist in (1) impurities and point defects, such as F⁺- and F-centers and, (2) other defects, such as dislocation and/or micro cracks⁶. The former depends mainly on the materials, and the latter on the surface treatment, such as machining (lapping or polishing)⁷ and annealing.

In this study, the mechanism of the flashover was investigated from the view points of surface treatments. The surface characterizations of charging and defects were performed by the SEE and cathodoluminescence (CL) measurements, respectively.

2. Results and discussion

2.1 Alumina material and surface treatment

Two kinds of alumina ceramic (UHA-99⁸) and HA-997⁸) and single-crystal-alumina (sapphire) disks³ (92 mm in diameter and 3.5 mm thick) were examined. UHA-99 comprises small grains having diameters of about 1 μ m, and has a highly dense structure. HA-997 exhibits lower dielectric losses (tan δ) (Table 1).

The disks were lapped with silicon carbide (SiC) abrasives of 400 and 600 grit for alumina ceramics and sapphire, respectively. Some disks were subjected to polishing using diamond abrasives ($\sim 2 \mu$ m in diameter) to attain a mirror finish. A sapphire disk, whose surface was chemically etched, was also prepared. Annealing at 1500°C for one hour in air⁹) was carried out for some disks. Photographs of unpolished, polished and annealed surfaces are shown in Fig. 1. The grain boundary of

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Table 1 Surface charging and point defects of the samples

	material				Surface			before high-power tests	
	alumina	purity (%)	ϵ	$\tan \delta$ ($\times 10^{-3}$)	finish	Roughness (μm)	treatment	charging type	CL spectra
#1	UHA-99	99.0	9.81	9.4	unpolished	0.6	none	(a)	F^+ , Cr^{3+}
#2					polished	0.1	annealed	(a)	F^+ , Cr^{3+}
#3							none	(c) high	F^+ , Cr^{3+}
#4	HA-997	99.7	9.95	4.2	unpolished	1.4	none	(a)	F^+ , Cr^{3+}
#5					polished	0.1	annealed	(a)	F^+ , Cr^{3+}
#6							none	(c) high	F^+ , Cr^{3+}
#7	sapphire	100	10.2	2.3	unpolished	0.4	chem.etched	(c) low	mainly F
#8						0.25	none	(c) low	mainly F
#9					polished	0.05	annealed	(a)	F^+
#10							none	(c) high	mainly F

Fig. 1 Microscopic structure of the window material: a) unpolished HA-997 (#4), (b) polished HA-997 (#6), and c) annealed HA997 (#5).

the ceramic after annealing was clearly visible. Rearrangements in the grain probably occurred due to annealing, and the dislocations of the ceramic were considered to be relaxed.

Secondary electron emission (SEE) and cathodoluminescence (CL) were measured in order to investigate the surface conditions before and after a high-power transmission examination using a resonant ring³.

2.2 Secondary electron emission (SEE) and surface charging

The SEE was measured using a single-pulsed electron beam (1 ms, 100 pA) with a scanning electron microscope (SEM)²⁻⁴, as shown in **Fig. 2**. The secondary electrons were captured by a biased (+40

V) Faraday cup. The relation of the measured currents is

$$I_p = I_F + dQ/dt, \quad (1)$$

where I_p is the primary current, I_F is the current captured by the Faraday cup and Q is the amount of charge in the alumina disk. Furthermore,

$$I_{ab} = I_p - I_F = dQ/dt, \quad (2)$$

where I_{ab} is the absorption current. The SEE coefficient (δ) is given by

$$\delta = I_F/I_p \quad (t=0). \quad (3)$$

SEE measurements were good diagnostics for surface charging. Four typical SEE signals in the measurement are shown in **Fig. 3**.

(a) $\delta > 1$. When surface charging is negligible, secondary emitted electrons are captured by the

Fig. 2 Schematic of SEE measurements. I_p is the primary current, I_F the secondary electron current captured by Faraday-cup and I_{ab} is the absorption current.

Faraday cup ($I_F > I_p$). Positive charging develops with time ($dQ/dt > 0$) which recoils secondary electrons to the surface. Thus, δ decreases rapidly. A small positive charge (< 10 eV) is sufficient to draw the electrons, because they have a low kinetic

energy of about $1 \sim 5$ eV^{10,11}). In fact, the shift of the AES peak was less than 10 eV during an AES analysis of the alumina ceramics. I_F decreases with time, and finally reaches the value in case (b).

(b) $\delta = 1$. The number of secondary electrons captured by the Faraday cup equals that of the primary electrons ($I_F = I_p$). Q does not change ($I_{ab} = 0$), and this case is the steady state.

(c) $\delta = 0$. When the surface is positively charged (> 50 eV), all of the secondary electrons released from the surface return to the sample ($I_F = 0$). If electron irradiation continues, Q decreases with time and finally also reaches the steady state of case (b).

(d) $\delta = 1$. When the surface is negatively charged, the primary electrons are repelled before reaching the sample. This case is also the steady state ($\delta = 1$), and is attained only after electron irradiation to the surface, where δ is less than unity (the incident energy is > 30 keV for alumina and > 2 keV for TiN coatings, respectively); negative charging (< -100 eV) during an AES analysis of TiN coated alumina was observed at an incident energy of 3 keV, which was also reported for Cu coatings on MgO¹²).

Fig. 3 SEE signals obtained by a pulsed beam (1 ms, 100 pA) at an incident energy of 1 keV together with the schematic of surface charging. Time scale: 1 ms/div. (a) Normal surface, (b) slight positive charging, (c) positive charging. (d) negative charging, this signal was obtained after 8 keV electron irradiation on TiN-coated area.

The measured surface charging is summarized in **Table 1**. All of the polished disks are positively charged. Although surface charging was also observed on unpolished sapphire disks, the charged area was one order of magnitude smaller than that of the polished disks. Polishing is considered to induce charged sites, such as dislocations and/or microcracks, where electrons are removed (thus positive charging).

It is found that the charging due to polishing was removed by annealing. This is caused by the occurrence of electric conduction at high temperatures¹³. The vanishing of surface charges was also interpreted as being caused by a decrease in the number of charged sites due to the relaxation of mechanically introduced dislocations and/or microcracks upon annealing.

2.3 Cathodoluminescence (CL) and point defects

The spectra of cathodoluminescence (CL) were observed using a SEM with a 5 keV and a 10 keV electron beam (20 nA)²⁻⁴. It is reported that the F-center defects (oxygen vacancy having two trapped electrons) contribute to electron emission sites³. On the other hand, the F⁺-centers (oxygen vacancy having one trapped electron) and impurity levels of Cr³⁺ (ruby) were not considered to be related to electron emission.

The defects in the sample disks obtained from

CL are also summarized in Table 1. These pre-existing F-centers depended not on the mechanical treatment, but rather on the type of material.

The sapphire disks have F-center defects which disappeared after annealing. This indicates that electron detrapping due to thermal activation took place upon annealing (the conversion from F-center to F⁺- or α -center)¹⁴.

2.4 High-power rf tests and flashover threshold

High-power transmission tests were performed using a resonant ring³. The surfaces of a dielectric disk were coated with 1-nm-thick TiN films in three quarters⁴. An rf window was examined at a maximum power of 230 MW (2856 MHz, 2 μ s, 50 pps) in this experiment.

The results are summarized in **Table 2**.

The flashover thresholds of unpolished disks were higher than those of the polished disks. Although the threshold increased upon annealing, the value did not completely match to that of an unpolished one. This is probably because annealing is insufficient for eliminating all of the charged sites. Sapphire disks showed a lower threshold value than did the ceramic disks due to their preexisting F-centers³. For annealed sapphire (#9), the F-center easily reappeared again during rf operation at a power of 30 MW or less. Since oxygen defects were possibly converted from F⁺-centers to F-centers¹⁴

Table 2 Polishing and annealing effects on flashover threshold

	alumina	finish	treatment	threshold power (MW)	after high-power tests		
					charging type	CL spectra	remarks
#1	UHA-99	unpolished	none	> 230	(c) high	F ⁺ , Cr ³⁺ , (F)*	
#2		polished	annealed	180	(c) strong	(F ⁺ , Cr ³⁺)**	crack
#3			none	145	(c) strong	(F ⁺ , Cr ³⁺)**	local melting
#4	HA-997	unpolished	none	> 230	(c) high	F ⁺ , Cr ³⁺	
#5		polished	annealed	230	(c) high	F ⁺ , Cr ³⁺	local melting
#6			none	230	(c) high	F ⁺ , Cr ³⁺	pin-hole
#7	sapphire	unpolished	chem. etched	230	(c) high	F ⁺ , F	local melting
#8			none	160	***	F ⁺ , F	local melting
#9		polished	annealed	100	***	F ⁺ , F	local melting
#10			none	80	(c) high	F ⁺ , F	local melting

*F-center appeared at the uncoated area.

**There were highly charged areas where CL spectra were difficult to obtain.

***not measured.

by trapping of electrons, annealing in air in this experiment is not sufficient to reduce the number of oxygen defects.

Flashovers, which limited the maximum transmission power, were observed on the TiN-coated area on all of the disks, except for #1 and #4. Surface melting occurred at the center of the disks after flashovers (Fig. 4).

All of the disks acquired positive charges (case (c)) after the high-power tests on both TiN-coated and noncoated areas, although the intensity of charges depended on the type of disk. It is found that multipactoring resulted in positive charges on the noncoated area. Positive charging on the TiN-coated area is discussed later.

On the highly charged areas generated in high-power tests (#2 and #3), the intensity of CL was two orders of magnitude lower than before high-power tests. This implies that charging not only occurred around dislocations and/or microcracks, but also affected the oxygen vacancy.

Surface charging took place after high-power tests, and point defects, in addition to dislocations and/or microcracks, were related to the strong surface charging.

2.5 Surface charging and flashover

A model of flashover on a TiN-coated surface can be proposed. TiN coatings suppress multipactoring by absorbing the secondary electrons emitted from alumina. Although the measured δ is less than unity for the TiN coatings at an incident energy of more than 2 keV, the electrons are accumulated in the coatings (case (d) in Fig. 3). The electrons emitted by the applied rf electric field (8 MV/m under the rf operation at the power of 200 MW⁵⁾) induce flashover, after which positive charges remain in the disk.

In this model, the threshold value depends on the amount of charges in TiN and the electric field. The amount of charges in TiN was considered to be more when the alumina had a greater number of charged sites. Thus, those polished disks with charged sites introduced by mechanical polishing resulted in a lower threshold. The lower threshold of sapphire was also considered to be due to the fact that the F-center defects contributed to electron emission sites and that more charges were accumulated in TiN.

It is concluded that the elimination of surface charging is important to increase the threshold of flashover.

It is desirable to measure not only the surface but also depth distribution of charges and the electric potential, which is probably related to

Fig. 4 An alumina disk (#6) with pinhole after high-power test. Surface melting is observed at the center of the disk.

flashover.

3. Summary

The breakdown of alumina rf windows was investigated. Various surface treatments were examined by secondary electron emission (SEE) and cathodo-luminescence (CL). SEE measurements were effective for estimating surface charges, and surface treatments such as polishing and annealing were suggested to affect the charged sites.

The breakdown (flashover) thresholds of the polished disks were lower than those of annealed or unpolished disks due to the accumulation of more charges on the TiN-coated surface, probably released from dislocations and/or microcracks of alumina caused by polishing.

It is concluded that the elimination of surface charging that is due to mechanical polishing was effective in increasing the threshold of flashover. Measurements of the charge distribution and the electric potential suggest the need for further investigations of surface charging and flashover.

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