Dielectric breakdown of silicon nitride substrates with various thicknesses

Chika MATSUNAGA¹,³, You ZHOU¹, Dai KUSANO², Hideki HYUGA¹ and Kiyoshi HIRAO¹

¹National Institute of Advanced Industrial Science and Technology (AIST), 2266–98 Shimo-Shidami, Moriyama-ku, Nagoya 463-8560, Japan
²Japan Fine Ceramics Co., Ltd., Sendai 981–3203, Japan

Dielectric breakdown of silicon nitride substrates was evaluated using alternating current voltage. Two different kinds of Si₃N₄ ceramics with mainly small and large grains were used. Test specimens were prepared from both Si₃N₄ ceramics with thicknesses of 0.25, 0.32, and 0.64 mm. Average breakdown strength of 0.25, 0.32 and 0.64-mm-thick Si₃N₄ specimens with small grains were 36.8, 35.1 and 24.9 kV/mm, and those of Si₃N₄ specimens with large grains were 29.5, 27.1, and 21.4 kV/mm, respectively. At all thicknesses, average breakdown strengths of Si₃N₄ specimens with small grains were higher than those of Si₃N₄ specimens with large grains. Average breakdown strength of both Si₃N₄ ceramics increased with decreasing thickness. Both the top and bottom surfaces of the Si₃N₄ ceramics with small and large grains had holes of 50 to 250 μm in diameter after breakdown test. Concentric cracks were observed around the holes. Both Si₃N₄ ceramics with small and large grains showed a tortuous breakdown channel in the direction of thickness after breakdown test. One or both edges of the tortuous breakdown channel had a crater-like structure where the opening was larger than the channel. The channel openings showed two different types of morphologies. One channel opening had concentric deposits around the channel, and other opening showed bare grains and no deposits.

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1. Introduction

In recent years, power electronic devices that convert and control electrical power with high efficiency have become popular as a key technology for energy conservation, and they have been increasingly used in the fields such as industrial robots, hybrid motor vehicles, and advanced electric trains.¹ Furthermore, the development of power device technology is expected toward higher voltage, larger current, higher power density, and smaller size, due to the replacement of Si with the wide-bandgap semiconductors such as SiC and GaN.² The increase of power density and output of the power devices causes increasing amount of heat generated in the power devices, therefore heat dissipation technology has become extremely important for the high-power devices. For such a purpose, ceramic insulating substrates with high thermal conductivity are required. AIN with thermal conductivity above 200 W/(m·K) has been used as a major ceramic substrate material. However, the mechanical properties of AIN are insufficient for such applications (typical bending strength of 300–400 MPa and fracture toughness of 3–4 MPa·m¹/²), which would result in substrates with low reliability.³ The electronics industry is eager to seek alternative high-thermal-conductivity substrate materials with good mechanical properties; therefore, attention has been turned to Si₃N₄ ceramics.

Over the last decades, Si₃N₄ ceramics have been investigated as high temperature structural materials, and now Si₃N₄ with a bending strength of over 1 GPa can be prepared by the selection of appropriate sintering additives and full development of a bimodal microstructure composed of interlocked rod-like grains.⁴ Recently, our group has been conducting research of fabrication of Si₃N₄ with both high thermal conductivity and high strength by using a reaction-bonding and post-sintering method⁵-⁷ through evaluation of a variety of parameters, such as the oxygen content in starting Si powder,⁸,⁹ aluminum content,⁹,¹⁰ iron content¹¹ and sintering additives employed.⁹,¹²,¹³ The reaction-bonding and post-sintering method is a fabrication process used to obtain dense silicon nitride sintered bodies by heat-treatment of a Si powder compact in a nitrogen atmosphere to achieve transformation into Si₃N₄, followed by post-sintering for densification.¹⁴ From the starting material consisting of a high-purity silicon powder and Y₂O₃ and MgO sintering aids, Zhou et al.¹⁵ successfully fabricated Si₃N₄ ceramics with a record-high thermal

Corresponding author: C. Matsunaga; E-mail: chika.matsunaga@aist.go.jp

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conductivity of 177 W/(m·K) and fracture toughness of 11.2 MPa·m²/3 by the reaction-bonding and post-sintering method. These results demonstrated that Si₃N₄ ceramics can be considered potential substrates for power devices.

When the Si₃N₄ ceramics are used as substrates for power devices, electrical insulation property is also an important factor. Electrical insulation gets lost by dielectric breakdown which leads to formation of an electrically conductive channel through the insulator. In general, breakdown strength defined as the breakdown voltage per sample thickness is used for evaluation. Breakdown strength of alumina and glass can be found in many literatures, and they have been reported to be dependent on microstructure, sample thickness and loading condition.

Malec et al., reported that the breakdown strength increased with increasing purity in range of 92 to 99.5% of alumina when sample thickness was 635 µm and applied voltage was alternating current (AC) voltage. For the alumina substrates with average grain sizes in the range between 1.1 to 3.6 µm, breakdown strength increased with decreasing grain size. However, the electrical insulation property of Si₃N₄ bulk ceramics has not yet been reported. Si₃N₄ to be used as a circuit substrate is required to have high electrical insulation since power density of power devices is expected to increase with year. Moreover, the thickness of Si₃N₄ substrates is an important factor. The thickness of circuit substrates is typically in the range of several hundred micrometers to several millimeters. Therefore, in the present study, effects of microstructure and thickness on the breakdown strength of Si₃N₄ substrates were investigated. The thicknesses of the Si₃N₄ substrates were chosen to be several hundred micrometers in considering the measurable range of the dielectric breakdown measuring appliance used in this study.

2. Experimental procedure

Two kinds of Si₃N₄ ceramics which were distinguished by their microstructures, one with relatively small grains and the other with large grains, were employed in this study (Japan Fine Ceramics Co., Ltd., Miyagi, Japan). The Si₃N₄ ceramics with smaller and larger grains had thermal conductivities of 90 and 140 W/(m·K), respectively. Each kind of Si₃N₄ materials were machined to 3 types of substrates of different thicknesses (0.25, 0.32 and 0.64 mm). After mirror-surface polishing, specimens with a dimension of 10 mm × 10 mm were cut out from the substrates for dielectric breakdown measurement. For each type of specimen, 46 to 48 pieces were measured.

Dielectric breakdown strength was measured by an AC voltage tester (7473, Keisoku Giken Co., Ltd., Kanagawa, Japan). A specimen was sandwiched between electrodes and immersed in an insulating oil (FC-3283, 3M Japan Ltd., Tokyo, Japan), and then AC voltage of 50 Hz was applied at room temperature. AC voltage was increased by 1 kV in 0.4 s and was held for 1 s. Then the AC voltage continued to be increased at this pace and in this stepwise fashion up to a dielectric breakdown voltage or 20 kV. The electrodes had a circular column shape with rounded edges and the diameter was 6 mm according to Japan Industrial Standard C2210-2:2010. The dielectric breakdown voltage was determined at the value when the leakage current reached 9.99 mA.

X-ray diffraction (XRD; RINT-2500, Rigaku, Tokyo, Japan) with monochromatic CuKα radiation (40 kV/100 mA) was used to identify the phases of the substrates. The morphology of the specimens before and after dielectric breakdown test was observed using scanning electron microscopy (SEM; JSM-5600, Jeol, Tokyo, Japan). Average grain sizes were calculated by the intercept method with SEM images. The average grain sizes of the silicon nitride ceramics with small and large grains were calculated from over 850 grains. Channels in the specimens with 0.64 mm thickness after dielectric breakdown tests were analyzed by X-ray Microcomputed Tomography (Vera 520; Carl Zeiss X-ray Microscopy, Pleasanton, CA, USA). The emission source was set at 60 kV and 5 W. The resolution was 2.5 µm × 2.5 µm per voxel.

3. Results and discussions

Figure 1 shows SEM images of polished and plasma-etched surfaces of Si₃N₄ specimens with (a) small and (b) large grains. The Si₃N₄ specimens with small and large grain are hereafter referred to as S-SN and L-SN, respectively. S-SN has a bimodal microstructure with some large elongated grains dispersed in a matrix of fine grains. L-SN led to a very coarse microstructure, where some large elongated grains had widths over 10 µm and lengths around 100 µm. The calculated average grain sizes of the S-SN and L-SN were 1.28 and 2.84 µm, respectively. It seems that area fraction of grain boundary phase in L-SN (bright area) was smaller than that in S-SN.

Figure 2 shows the XRD patterns of (a) S-SN and (b) L-SN. Both of the XRD patterns had β-Si₃N₄ as the main phase and Y₂Si₃O₅N₄ as the secondary phase. No α-Si₃N₄ peaks were observed in both of them. It is considered that the secondary phase was formed by the reaction between Si₃N₄ and the sintering additives. The relative intensity ratio of β-Si₃N₄ to Y₂Si₃O₅N₄ phase in S-SN was smaller than that in L-SN, meaning that the amount of secondary phase in L-SN was smaller than that in S-SN. This is in accordance with the SEM results mentioned above.

Figure 3 shows Weibull plots of the breakdown strength for the specimens of (a) S-SN and (b) L-SN with 0.25, 0.32, and 0.64 µm thicknesses. The values of minimum breakdown strength of S-SN with 0.25, 0.32, and 0.64 mm thicknesses were 31.4, 29.7, and 20.2 kV/mm, and those of maximum breakdown strength were 43.1, 45.5, and 30.1 kV/mm, respectively. For the L-SN with 0.25, 0.32, and 0.64 µm thicknesses, the values of minimum breakdown strength were 27.3, 24.2, and 19.1 kV/mm and those of maximum breakdown strength were 35.1, 33.4, and 24.3 kV/mm, respectively. The minimum and maximum breakdown strength of S-SN were higher
than those of L-SN at all thicknesses. Though the L-SN had slightly lower breakdown strength than that of the S-SN, it showed narrower distribution of the breakdown strength.

In order to examine the effect of specimen thickness on breakdown strength these values were logarithmically plotted. Figure 4 shows relationship between arithmetic average breakdown strength and thickness of the S-SN and L-SN. The average breakdown strengths of S-SN with 0.25, 0.32, and 0.64 mm thicknesses were 36.8, 35.1, and 24.9 kV/mm, respectively. And those of L-SN with 0.25, 0.32, and 0.64 mm thicknesses were 29.5, 27.1, and 21.4 kV/mm.
All average breakdown strengths of S-SN were higher than those of L-SN at all thicknesses. Both average breakdown strengths of S-SN and L-SN increased with decreasing thickness. The result showed a similar tendency as the relationship between fracture strength of ceramic and thickness. The probability for a large defect initiating mechanical fracture increases with increasing sample thickness and thus its volume because mechanical failure of ceramic materials occurs due to microstructural defects.

It has already been reported that the electrical breakdown strength $E_{bd}$ of a ceramic sample has an inverse relation to the specimen thickness $d$.\textsuperscript{19,23}

$$E_{bd} = \frac{A}{d^n}$$

The equation is an experimental formula and $n$ assumes values from 0.1 to 0.5 and $A$ is a constant value depending on materials and microstructure. Yoshimura and Bowen reported that electrical breakdown occurs at the electrically weakest point in the specimens, which might correspond to microstructural or defects (scratches, grain boundaries, second phases, or pores), therefore a larger thickness has a statistically higher probability of such defects.\textsuperscript{19} In the results, fitting analysis of Fig. 4 leads to the following expressions:

$$E_{bd(S-SN)} = \frac{20}{d^{0.44}}$$
$$E_{bd(L-SN)} = \frac{18}{d^{0.35}}$$

The obtained $n$ values of S-SN and L-SN were 0.44 and 0.35, respectively. Therefore, the results were reasonable because $n$ satisfied the above condition. The $n$ values were determined by calculating the slopes of the lines depicting the correlation between average breakdown strength and thickness of specimens shown in Fig. 4. A large $n$ value, which is close to 0.5, means a large influence of thickness on breakdown strength; and a small $n$ value, which is close to 0.1, means a small effect of thickness.

Figure 5 shows 3D reconstruction from X-ray tomography of (a) S-SN and (b) L-SN with 0.64 mm thickness after dielectric breakdown tests. Each specimen has a tortuous breakdown channel in the direction of thickness after breakdown tests for S-SN and L-SN. One or both edges of the tortuous breakdown channel has a crater-like structure with a larger diameter than that of the channel. Similar observations has already been reported in literature of alumina.\textsuperscript{24,25} The diameter of the channel of S-SN was larger than that of L-SN. The channels of L-SN was more tortuously and longer than that of S-SN because of crack deflection and crack bridging caused by large elongated grains of L-SN.\textsuperscript{15} The dependence of crack trajectory on grain shape and size was similar to that often observed in mechanical indentation test of silicon nitride ceramics.

Figure 6 shows optical micrographs of both channel openings of (a) S-SN and (b) L-SN with 0.64 mm thickness after dielectric breakdown tests. Each specimen has a tortuous breakdown channel in the direction of thickness after breakdown tests for S-SN and L-SN. One or both edges of the tortuous breakdown channel has a crater-like structure with a larger diameter than that of the channel. Similar observations has already been reported in literature of alumina.\textsuperscript{24,25} The diameter of the channel of S-SN was larger than that of L-SN. The channels of L-SN was more tortuously and longer than that of S-SN because of crack deflection and crack bridging caused by large elongated grains of L-SN.\textsuperscript{15} The dependence of crack trajectory on grain shape and size was similar to that often observed in mechanical indentation test of silicon nitride ceramics.

Figure 7 shows SEM images of both channel openings of (a) S-SN and (b) L-SN after dielectric breakdown tests. The channel openings displayed two different types of morphology in both S-SN and L-SN. One channel opening had concentric deposits around the channel. Another channel opening showed bare grains and no deposits. Some researchers reported that alumina grains of channel opening melted after breakdown test, and they...
considered the temperature of the alumina grains at channel opening should have exceeded the melting point of alumina (2054°C).\textsuperscript{17,28} Si\textsubscript{3}N\textsubscript{4} could decompose without melting when temperature was higher than 1830°C at 0.1 MPa atmosphere. That was why the morphologies of the channel openings of Si\textsubscript{3}N\textsubscript{4} and alumina were different. It is considered that the deposits around the channel opening of the Si\textsubscript{3}N\textsubscript{4} specimens was silicon due to decomposition of Si\textsubscript{3}N\textsubscript{4} by dielectric breakdown.

4. Conclusions

Dielectric breakdown strengths of 0.25, 0.32 and 0.64-mm-thick Si\textsubscript{3}N\textsubscript{4} specimens with small and large grains were evaluated. 0.25, 0.32 and 0.64-mm-thick Si\textsubscript{3}N\textsubscript{4} specimens with small grains had 36.8, 35.1 and 24.9 kV/mm of average breakdown strength, and those with large grains had 29.5, 27.1, and 21.4 kV/mm of average breakdown strength, respectively. At all thicknesses, average breakdown strengths of Si\textsubscript{3}N\textsubscript{4} specimens with small grains were higher than those of Si\textsubscript{3}N\textsubscript{4} specimens with large grains. Average breakdown strengths of both Si\textsubscript{3}N\textsubscript{4} specimens increased with decreasing thickness, and the results was accordant with the experimental formula about the relationship between breakdown strength and thickness. Electric breakdown occurs due to microstructural defect, therefore it is assumed that a larger thickness specimen has a statistically higher probability of such defects, and thereby leading to a lower breakdown strength.

Both the top and bottom surfaces of the Si\textsubscript{3}N\textsubscript{4} specimens had holes of 50 to 250\(\mu\)m in diameter after

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**Fig. 6.** Optical micrograph of both channel openings of [(a) and (b)] S-SN and [(c) and (d)] L-SN with 0.64 mm thickness after dielectric breakdown test.

**Fig. 7.** SEM images of both channel openings of [(a) and (b)] S-SN and [(c) and (d)] L-SN after dielectric breakdown test.
breakdown test. Concentric cracks were observed around the holes. Both Si₃N₄ specimens with small and large grains had tortuous breakdown channels in the direction of thickness after breakdown test. One or both edges of the tortuous breakdown channel showed a crater-like structure where the opening was larger than the channel. The channel of Si₃N₄ with large grains was more tortuously and longer than that of Si₃N₄ with small grains because of crack deflection and crack bridging caused by large elongated grains. The channel openings had two types of morphologies. One channel opening had concentric deposits around the channel, and the other opening showed bare grains and no deposits. It is considered that the deposits were silicon resulting from the decomposition of silicon nitride during breakdown test.

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