A Study on the Utilization of an Electro-Conductive Surface Layer on a Si_3N_4 Ceramic Formed by Ion Implantation as a Crack Gage and a Strain Gage

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イオンインプランテーションにより Si_3N_4 セラミックス上に形成された導電層のクラックゲージ及び歪ゲージとしての利用に関する研究

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An experimental study on the utilization of an electro-conductive surface layer on a dielectric Si_3N_4 component formed by carbon ion (C^+) implantation as a crack gage and a strain gage was conducted. Surface cracks were generated by the Vickers indentation. The electrical resistance of the electro-conductive layer increased with the generation of the surface cracks. The resistance was also influenced by stresses which appeared in the layer. Variation of electrical resistance caused by cracking and stresses was so small that the resistance must have been measured with high accuracy.

Key-words : Si_3N_4 , Ion implantation, Carbon ion, Stress, Surface cracks, Electrical resistance, Vickers indentation

1. Introduction

Structural ceramics have been widely developed for a variety of applications. Nevertheless, the catastrophic failures of the ceramic components without any omens, owing to their brittleness, have interfered with many industrial applications. In order to detect the generation of cracks and the slow crack propagation in metals, crack gages and strain gages stuck on the component surfaces have been used. However, in high performance structural ceramics, the size and the opening displacement of the cracks, which may cause the catastrophic failures of the components, are so small that they can not be detected by commercially available crack gages.¹⁾ Furthermore, crack and strain gages can not endure severe environments, such as high temperatures and corrosive environments, in which ceramic components are often used.

Recently, some of the present authors had reported that a thin electro-conductive layer can be formed on the surface of dielectric Si_3N_4 ceramics by an ion implantation process.^{2),3)}

In this report, a study on the utilization of electro-conductive surface layer on Si_3N_4 formed by ion implantation as crack and strain gages was conducted. The effect of stresses in the ion implanted surface

layer on its electrical resistance was examined with bending tests of ion implanted specimens. Surface cracks were generated by the Vickers indentation method. The resistance of the electro-conductive layer was continuously measured during the indentation processes.

2. Experimental procedures

A hot-pressed Si_3N_4 ceramic (Toshiba Corporation) was used for experiments. Their selected properties and additives are shown in Table 1. The as-obtained ceramic body was cut into pieces of the size about 3 mm $\times$ 4 mm $\times$ 40 mm. All surfaces of the specimens were ground with a 400 grit diamond grinding wheel. The surface to be ion implanted was mirror finished with a 3000 grid diamond disk, the surface roughness R_a being about 0.1 μm . Two separate parts of the specimen surface were coated with gold by an ion sputtering method, as shown in Fig. 1. The thickness of the gold surface layers was about 1 μm . The region A (2.5 mm $\times$ 7 mm), as shown in Fig. 1, was made electro-conductive by ion implantation. The electrical resistance of the region A between the gold coated electrodes was measured.

A 1.7 MV tandem type ion accelerator was used for the ion implantation. Carbon ions (C^+) of beam energy 2 MeV were used for the implantation.²⁾ The

Table 1. Some properties and additives of the Si_3N_4 ceramic.

Bulk density (g/cm ³)	3.196	
Electrical resistivity (ohm cm)	10 ¹⁴ <	
Young's modulus* (GPa)	274	
Poisson's ratio*	0.26	
Average flexural strength** (MPa)	964	
Amount of additives (wt%)	Al ₂ O ₃	2.9
	Y ₂ O ₃	5.0

* :Measured by pulse echo method

** :Measured by four point bending test (JISR1601) at room temperature

Fig. 1. Dimensions of the Si_3N_4 specimen.

density of implanted ion was 1×10^{17} ion/cm². During the implantation process, the specimens were heated up to about 573 K. The electrical resistance was about 15.2×10^3 ohm, after the implantation.

In order to evaluate the effect of stresses appeared in the ion implanted surface layer on the electrical resistance, the resistance was measured during four-point bending tests of the specimens at room temperature. The bending tests were carried out following JIS R1601, except for the low cross head speed of 50 $\mu\text{m}/\text{min}$.

The effect of surface cracks on the resistance was evaluated. The surface cracks were generated by the Vickers indentation. The maximum indentation loads (9.8, 49, 98N) were applied for 15 s.

3. Results and discussion

Scanning electron micrographs (SEM) of the fracture surfaces of an ion implanted specimen are shown in Fig. 2. The brightness in the lower half of

Fig. 2. SEM photographs of the fracture surface of ion implanted specimen.

(a) Low magnification, (b) High magnification.

Fig. 2(a) arises from the charge-up of electrons during SEM observation. The thickness of the electro-conductive surface layer formed by the ion implantation was smaller than about 8 μm , while the flat area near the surface in Fig. 2(b), about 2 μm in thickness, was considered to be a surface layer seriously ion implanted. The microstructure and mechanical properties of the layer may be varied extensively.

The relative variation of electrical resistance, $(R - R_0)/R_0 \times 100\%$, of the ion implanted surface layer under compressive and tensile stresses is shown in Fig. 3. R_0 and R are the electrical resistance before and during the bending test, respectively. When the tensile and the compressive stresses appeared in the ion implanted surface layer, the resistance increased and decreased, respectively.

As the results of the mechanical analysis on the elastic deformation of the specimen, about 70% of the variation of the resistance can be ascribed to the deformation of the electro-conductive surface layer due to the stresses. For example, the tensile stress of 400 MPa would cause the elastic deformation which increases the resistance by about 0.22% of the original value without any change in the intrinsic electrical resistivity of the ion implanted surface layer. Therefore, the deformation of the electro-conductive layer would have main effect on the variation of the resistance. While, about 30% of the variation of the resistance must be attributed to the change in the intrinsic electrical resistivity of the ion implanted surface layer due to the stresses.

Those stress versus electrical resistance relation-

Fig. 3. Relative variation of the electrical resistance caused by stresses appeared in the ion implanted layer during bending the specimen.

(a) Compressive stress, (b) Tensile stress.

ships suggest that the utilization of the electro-conductive surface layer formed by ion implantation would be possible as a strain gage.

SEM photographs of the indentation impression and the radial cracks generated by the Vickers indentation on the ion implanted electro-conductive surface are shown in Fig. 4. The radial cracks can be clearly identified by extremely bright areas. The dielectric nature of the Si_3N_4 induces the charge-up of electrons during SEM observation, resulting in the brightness on the photograph, while any open surface cracks can not be identified inside the impression.

The average lengths of the radial cracks (2c) and the diagonal lengths of the impressions (2a) were 52 and 36 μm , 138 and 81 μm , and 250 and 115 μm for the applied loads of 9.8N, 48N and 98N, respectively. The length of cracks propagated during an indentation process increased with the increase in the applied load.

The fracture surface of specimen with median cracks generated by the indentation is shown in Fig. 5. The sub-surface median crack can be seen on the surface. Considering the small thickness (about 8 μm) of the electro-conductive surface layer, those

Fig. 4. SEM photographs of the impression and the surface radial cracks generated by the Vickers indentation on the electro-conductive ion implanted surface (Load: 49N).

(a) Impression and surface radial cracks,
(b) Tip of surface radial crack.

Fig. 5. SEM photograph of the fracture surface of specimen with sub-surface median cracks generated by the Vickers indentation (Load: 49N).

Arrow A points a median crack,
Arrow B points a presumable front of median crack.

median cracks completely penetrate through the surface layer. The length of the surface radial cracks outside the impression, i.e., $2c-2a$, may be related to the increase in electrical resistance.

Figure 6 shows the relative variation of the electrical resistance during the indentation process. Considering the above-mentioned relatively small effect of the stresses on the resistance, and that the diagonal lengths of the impressions (2a) are much smaller than the width (2.5 mm) of the ion implanted area, most of the increase in the resistance during the indentation process can be ascribed to cracking in the electro-conductive surface layer. At the onset of each loading and unloading step, the resistance increases discretely. The increase of the resistance upon loading, was smaller than the increase upon unloading. It implies that the crack propagation during loading may be less intensive than that during unloading. Furthermore, the amount of the change in electrical resistance increases with the increase in the indentation load. The increase in the resistance may be related to the length of cracks generated by the indentations with various loads.

As shown in Fig. 6(a), the increase in the electrical resistance is as small as 1% of the original electrical resistance, even if the crack length outside the impression, $2c-2a$, is 135 μm . In order to detect such a small change in the resistance caused by cracking and stress, a very sensitive measuring system including a noise filter must be needed.

Fig. 6. Relative variations of the electrical resistance during indentation processes.

A: Loading process, B: Unloading process.
Indentation loads of (a) 98N, (b) 49N and (c) 9.8N.

4. Conclusion

A study on the utilization of an electro-conductive surface layer on a dielectric Si_3N_4 component formed by carbon ion (C^+) implantation as a crack gage and a strain gage was experimentally conducted. The electrical resistance of the ion implanted layer increases with the generation of the surface cracks. It is also influenced by stresses appearing in the layer. The increase in the resistance is so small that the resistance must be measured with high accuracy, in order to detect the generation of surface cracks as well as to evaluate the stress levels.

References

- 1) N. Muto, H. Yanagida, M. Miyayama, T. Nakatsuji, M. Sugita and Y. Ohtsuka, *J. Ceram. Soc. Japan*, 100, 585–88 (1992).
- 2) K. Saitoh, M. Ikeyama, H. Niwa, Y. Miyagawa, S. Miyagawa and K. R. Padmanabhan, Proceedings of 48th Government Industrial Research Institute, Nagoya Seminar (1990) p. 5.
- 3) K. Saitoh, M. Ikeyama, H. Niwa, Y. Miyagawa, S. Miyagawa and K. R. Padmanabhan, Extended Abstract of 51st Autumn Meeting of The Japan Society of Applied Physics, 1990, No. 2 (1990) p. 477.