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MEASUREMENT OF RESIDUAL STRESSES ON CERAMIC MATERIALS WITH HIGH SPATIAL RESOLUTION

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INTRODUCTION

Ceramic materials are often used in high performance semiconductor devices. Brazing is used as a common technique of joining the metal pins with the ceramic chip carriers. Brazing of thermally dissimilar materials results in high stress/strain gradients in the ceramic material which ultimately leads to the inferior performance of the device. Steep stress gradients and constantly decreasing size of the chips dictate the necessity of measuring the stresses with the spatial resolution of 100 μm or higher. The standard large area diffraction is not suitable for this purpose. The present research is concerned with the residual stress measurement in brazed kovar-alumina joints using x-ray micro-diffraction technique.

X-RAY DIFFRACTION STRESS MEASUREMENT

The x-ray diffraction technique actually measures the average elastic strains (interatomic spacing) by measuring the shift in the angle at which x-rays are diffracted (Bragg angle). Stress values are obtained from these elastic strains assuming that the material is elastically linear, homogeneous, and the effective diffraction elastic constants are known. Two techniques were used to measure stresses in the present research: " $\sin^2\psi$ " and single exposure (SET) techniques.^{1,2} Both techniques are valid for the plane stress state which is a reasonable assumption since the x-rays penetrate approximately 8 μm into the alumina. The $\sin^2\psi$ technique is recommended when high confidence is required on specimens likely to display pronounced crystallographic texture; SET assumes no texture present in the material, which reduces the number of necessary measurements.

TESTING PROCEDURE AND INSTRUMENTATION

X-Ray Micro-diffraction Technique

A back reflection diffractometer incorporating a position sensitive scintillation detector (PSSD)³ was used for both SET and $\sin^2\psi$ techniques. A microfocus (focal spot 0.5 x 8

mm²) x-ray tube with a copper target was used for measurements. CuK α radiation (wavelength $\lambda = 1.541838 \text{ \AA}$) provided for the analysis of the diffraction peaks in the range of Bragg's angle $2\theta = 140^\circ$ to 155° as shown in Table 1.

Table 1. Diffraction planes and Bragg angles for α -alumina (Cu radiation).

Diffraction plane (hkl)	Bragg angle 2θ
(11-15)	142.7
(40-10)	145.5
(05-4)	149.6
(10-16)	150.6
(33-0)	152.8

The (10-16) CuK α reflection was used for stress measurements, since it assured the highest intensity of x-rays diffracted from alumina powder. A double-pinhole collimator and two position sensitive detectors were mounted on a x-y precision stage so that their position with respect to the x-ray target could be adjusted in order to provide for the maximum intensity of the incident x-ray beam. The sample was positioned using a rotary table and a translation stage; this arrangement allowed rotation of the sample with respect to the incident x-ray beam with the measurement point serving as a center of rotation and was suitable for $\sin^2\psi$ measurements.

Experimental Determination of Diffraction Elastic Constants

In order to convert the measured strains to stresses one needs to know the effective x-ray elastic constants. The bulk elastic constants mechanically measured or theoretically calculated may differ from the x-ray effective elastic constants.¹ Table 2 shows the average macroscopic elastic constants calculated from the alumina single crystal elastic constants published by Wachtman⁴ using Voigt⁵, Reuss⁶, and Hashin-Shtrikman⁷ averaging methods. For a fully dense material, the experimental data should fall within Hashin-Shtrikman upper (HS⁺) and lower (HS⁻) limits. However, for ceramic materials the elastic moduli depend strongly on the porosity and the shapes of pores⁸⁻¹⁰, decreasing rapidly as percent porosity increases. The material used in this research had 8% - 10% porosity. Therefore, a four-point bending calibration method¹ was used to determine the effective diffraction elastic constants for alumina (10-16) planes. The value of $[E/(1+\nu)]_{(1016)} = 200 \text{ GPa}$ was experimentally determined; mechanically measured bulk Young's modulus was $E = 229 \text{ GPa}$.

Table 2. Macroscopic elastic moduli of fully dense α -alumina: E-Young Modulus, ν -Poisson ratio.

	Voigt	HS ⁺	HS ⁻	Reuss
E [GPa]	408.2	403.5	402.4	397.2
ν	0.229	0.232	0.233	0.236

Effect of X-Ray Beam Size on Accuracy of Measurements

It is generally accepted that at least 10,000 grains are required in the irradiated volume so that a sufficient number of grains contribute to the diffraction pattern so as to achieve a

continuous cone of diffracted x-rays. If this correlation is not achieved, the x-ray beam is composed of discrete, discontinuous spots, and this will hinder accurate measurements.¹¹ As the size of the incident x-ray beam decreases, the number of irradiated grains also decreases. In order to determine the effect of the incident beam size on the accuracy of measurements (and, therefore, to establish the minimum size of x-ray beam which can be used), the following procedure was applied. A double-pinhole collimator with replaceable apertures was built, which allowed control of the size of the x-ray beam in the range of 10 μm to 500 μm . The four-point bending stress on the surface of the alumina bar was measured simultaneously by the x-ray diffraction technique (SET) and calculated from the strain gauge response. The accuracy of x-ray diffraction measurements was assessed from the correlation between both techniques. It has to be recognized, however, that a strain gauge measures the strain averaged over its size (i.e. 10 mm^2 in this case), whereas the x-ray technique measures stress in the irradiated volume, whose cross section with the sample surface is much smaller than the size of the strain gauge.

The initial size of the x-ray beam was 470 μm (irradiated area 0.02 mm^2) and the detectors were placed 35 mm from the sample surface. Data collection time was 50 seconds. The good correlation between the XRD and strain gauge data is shown in Figure 1. The standard deviation of XRD measured stresses was ± 15 MPa. Subsequently, the beam size was reduced to approximately 170 μm . The data collection time was increased to 300 seconds. The results of the simultaneous four-point bending strain gauge and XRD measurements are shown in Figure 2. Two strain gauges and two independent strain indicators were used for measurements. The x-ray diffraction measurements were taken at various points in the region between the strain gauges at the same strain level. From Figure 3 it is seen that the XRD stresses differ depending on the location. This stems from the fact that different groups of grains can be subjected to different strains due to their orientation and grain interactions. The smaller the beam size, the smaller is the number of grains over which the strain is averaged. For the beam size of 170 μm and mean grain size of 6 μm , the estimated number of diffracting grains is approximately 20. In a polycrystalline aggregate the stresses are not uniformly distributed among the grains. The variance can be as high as $\pm 50\%$, depending on the elastic anisotropy of single crystals.^{12,13} However, the average strain measured by XRD correlates well with the strain gauge measurements (correlation factor $R^2 = 0.998$). The precision of measurements was approximately ± 15 MPa. The beam size of 170 μm was taken as the limit since it allowed one to average the measured strains over approximately 20 grains. If one assumed that the minimum number of grains could be as low as 10, then the initial beam size would be 120 μm . However, measuring stresses with a beam size less than 170 μm (for alumina mean grain size 6 μm) would not provide for measurement from a statistically significant number of grains. In general, the macroscopic state of stress (or strains) cannot be uniquely determined from measurements on a small number of grains.

Measurement of Residual Stresses in Alumina in the Vicinity of a Singular Pin

Initially, the residual stresses around the singular pin were mapped using a beam size of 470 μm . The stresses were measured in two perpendicular directions (Figure 3). The linear dependence of d-spacing on $\sin^2\psi$ indicated that there was no preferred crystallographic orientation (texture) and, therefore, the SET could be applied without sacrificing accuracy. Subsequently, the SET with the beam size of 170 μm was used to map the residual stresses around the singular Kovar pin brazed to an alumina substrate. The radial and tangential stresses were measured at locations showed in Figure 4: points #1,2 correspond to the location adjacent to the pin; points #3,4 are the midpoints between two brazing pads. The results of measurements are presented in Table 3.

Table 3. Residual stresses in diamond in the vicinity of the cracks $K_{IC} = 1.1 \text{ MPa}\sqrt{\text{m}}$.

Point #	Radial stress (MPa)	Tangential stress (MPa)
1	-7.7 ± 2.4	296.1 ± 12.6
2	-34.0 ± 5.0	0.7 ± 7.7
3	-43.4 ± 20.3	154.0 ± 14.7
4	-4.2 ± 10.5	35.7 ± 25.2

Figure 1. Correlation between the x-ray diffraction (SET) residual stress measurements and stress calculated from the strain gauge measurements; beam size $470 \mu\text{m}$.

Figure 2. Correlation between the x-ray diffraction (SET) residual stress measurements and stresses calculated from strain gauge measurements; beam size $170 \mu\text{m}$.

The radial stresses are slightly compressive in all cases. Tangential stresses are tensile and much higher in one direction than in the other. The residual stress measured at the edge of the sample (far away from the pin) was 0.1 ± 1.1 ksi. The stress field around a singular pin is not symmetric due to the fact that the pins usually are not brazed at the center of the brazing pad, and this eccentricity varies from pin to pin. The measured stress field can also be affected by the orientation distribution and size of grains in the alumina in the vicinity of the pin. The diffraction patterns showed that for a given volume penetrated by x-rays, the number of grains (i.e. grain size) and their orientation vary hindering, therefore, the determination of a unique stress, causing the particular strain state in a small group of grains.

Figure 3. Locations of measurement points around a singular pin; dimensions in mm.

CONCLUSIONS

A fast x-ray diffraction technique has been developed for measuring the residual stresses with high spatial resolution in ceramic materials. This resolution is limited by the mean size of grains and the radiation type. The effective diffraction elastic constants were experimentally determined for alumina as $(E/1+\nu)_{(1016)} = 200$ GPa. The accuracy of XRD measurement of residual stresses with the spatial resolution of $170 \mu\text{m}$ and precision ± 15 MPa was verified experimentally by strain gauge measurements. The stress field around a singular Kovar pin brazed to alumina was asymmetric with high tangential stresses in the vicinity of the pin decreasing with the distance from the pin.

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