Effects of X-ray Beam Collimation on the Measurement of Residual Stress Distribution in a Si_3N_4 /Steel Joint

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X-ray collimation and diffraction conditions were optimized to evaluate the detailed distribution of residual stress quantitatively around the ceramic/metal interface, and the results were applied to the Si_3N_4 /Cu/steel system. The residual stress value increased and finally saturated as the collimation and irradiation area decreased in the Si_3N_4 region close to the interface of the joint. The normal stress value across the interface saturated below 0.2 mm² to 240 MPa for Si_3N_4 /t0.2 mm Cu/steel, and to 400 MPa for Si_3N_4 /steel. It is recommended that $\phi 0.1$ mm collimation is suitable to evaluate its detailed distribution along the interface in which the irradiation area is 0.016–0.031 mm². Several characteristic features of distribution were successfully observed along the joined interface of Si_3N_4 /t0.2 mm Cu/steel having a lapped surface; first large tensile normal stresses at both edges, secondly the minimum stress points 1–2 mm inside the edge which was originated by stress relief through a localized plastic deformation of the Cu buffer, and thirdly the maximum normal stress across the interface in the center region of the specimen which had never been predicted and could be caused by the three dimensional effect of the joined plate.

KEY WORDS: residual stress; normal stress; stress distribution; ceramic/metal joint; Si_3N_4 /steel; brazing; X-ray $\sin^2\psi$ method; beam collimation; FEM analysis.

1. Introduction

Among the items of ceramic/metal joining, the formation of a strong interface and residual stress reduction are the key factors for its actual application.¹⁾ Since residual stress, caused by mismatches in thermal expansion and the elastic moduli between the ceramic and metal, affects the joint strength, it has been relaxed by plastic deformation of a ductile metal, such as Cu,¹⁾ or by thermal expansion coefficient control in the interlayer. The evaluation of the residual stress distribution around the interfaces has also been requested from the viewpoints of quality assurance and the verification of FEM/BEM or analytical prediction. Though several residual stress measurement methods have already been developed for ceramics, the X-ray $\sin^2\psi$ method is the most hopeful candidate because it is a non-destructive one and is applicable to diamond ground surfaces.

Since the X-ray method has been conventionally applied to metal components as one of the inspection tools, the irradiation area was too large to know the detailed residual stress distribution. For example, the heat affected zone of a steel welded portion has large grains over 0.1 mm in diameter, and more than 10 mm² was necessary to maintain the reliability measurement. On the other hand, ceramic has a fine grain, such as a $\phi 3 \times 10 \mu\text{m}$ column-like structure for $\beta\text{-Si}_3\text{N}_4$, and so it is possible to decrease the irradiated area to below 0.05 mm². The residual stress distribution with a large gradient around the anomaly

point of the ceramic/metal joint should rather be measured with a collimated X-ray beam. Nevertheless, it has never been reported for joints other than large area irradiation.²⁾

In this study, first, the effects of an X-ray irradiated area on the residual stress value were investigated, and secondly presented is a detailed distribution successfully measured around the interface of a Si_3N_4 /steel joint using $\phi 0.1$ mm collimated Cr-K α .

2. Formulations in X-ray Method

2.1. Residual Stress Measurement

The residual stress was measured from the diffracted X-ray peak shift with an incident angle ψ , which is well known as the $\sin^2\psi$ method. The residual stress values can be quantitatively calculated by Eq. (1)²⁻⁴⁾:

$$\sigma_R = -\frac{E}{2(1+\nu)} \cdot \cot \theta_0 \cdot \frac{\pi}{180} \cdot \frac{\partial(2\theta)}{\partial(\sin^2 \psi)}$$

$$= K \cdot \frac{\partial(2\theta)}{\partial(\sin^2 \psi)} \dots \dots \dots (1)$$

where, σ_R : residual stress
 E : Young's modulus
 ν : Poisson's ratio
 θ_0 : standard Bragg's angle
 ψ : X-ray incident angle
 K : stress constant.

The measured stress value was obtained from the surface layer of the ceramic about several ten microns

in depth, which is dependent on the X-ray penetration depth.

The diffraction peak used for the stress calculation should be chosen as one among those of isolated and high angle positions. The recommended diffraction plane, $2\theta_0$ and d -value for four representative ceramics are listed in Table 1 for three different characteristic X-rays. Table 1 was rearranged from the work by Tanaka.³⁾ Cr-K α and Cu-K α are popular X-rays for ceramics, but different in penetration depth (Cr < Cu). For β -Si₃N₄, the authors used mainly Cr-K α with collimation, and in some cases V-K α as large area (2×4 mm) irradiation. Full diffraction profile of Si₃N₄ by Cr-K α between 10° – 150° is shown in Fig. 1. (212) diffraction peaks at 131.47° satisfied the above-mentioned conditions. In this study, the stress constant K in Eq. (1) was -90.08 kg/mm^2 (882.8 MPa) in Si₃N₄ (212) diffraction with $E=29\,082 \text{ kg/mm}^2$ and $\nu=0.27$.

2.2. X-ray Collimation and Irradiated Area

An X-ray collimator with one pin hole was used to measure the residual stress in the localized area. The X-ray irradiated area diffused into ellipse having

Table 1. Recommended X-ray diffraction peaks for residual stress measurement for several ceramics and steel.

X-ray	V-K α	Cr-K α	Cu-K α
β -Si ₃ N ₄	(411) $2\theta_0=152.67^\circ$ $d=1.238 \text{ \AA}$	(212) $2\theta_0=131.47^\circ$ $d=1.256 \text{ \AA}$	(323) $2\theta_0=141.45^\circ$ $d=0.816 \text{ \AA}$
α -Al ₂ O ₃		(1, 0, 10) $2\theta_0=135.03^\circ$ $d=1.239 \text{ \AA}$	(416) $2\theta_0=136.11^\circ$ $d=0.830 \text{ \AA}$
ZrO ₂ (Y-TZP)		(133) $2\theta_0=152.09^\circ$ $d=1.180 \text{ \AA}$	(026) $2\theta_0=140.22^\circ$ $d=0.819 \text{ \AA}$
SiC (6H)		(116) $2\theta_0=121.69^\circ$ $d=1.311 \text{ \AA}$	(306) $2\theta_0=134.09^\circ$ $d=0.837 \text{ \AA}$
α -Fe		(211) $2\theta_0=156.08^\circ$ $d=1.170 \text{ \AA}$	

a larger diameter than that of a pin hole, ϕC , with incident beam inclination. Typical configurations of the X-ray optical system used in this experiment are shown in Fig. 2 (Rigaku MDG). The diameters of the irradiation, dx , dy , and its area, dS , can be calculated using Eqs. (2) to (4);

$$dx = 2(L_1 + L_2) \cdot \tan \theta_x / 2 - a \quad \dots\dots\dots (2)$$

$$dy = \left[\frac{1}{\sin(\omega + \theta_y/2)} + \frac{1}{\sin(\omega - \theta_y/2)} \right] \times L \cdot \sin \theta_y / 2 \quad \dots\dots\dots (3)$$

$$\text{where, } \theta_x = 2 \tan^{-1} \{ (\phi C / 2 + a / 2) / L_1 \}$$

$$= 2 \tan^{-1} \{ (\phi C / 2 + b \sin \alpha / 2) / L_1 \}$$

$$L = (L_1 + L_2) - \frac{b \sin \alpha}{2 \tan \theta_y / 2}$$

$$dS = \pi/4 \cdot dx \cdot dy \quad \dots\dots\dots (4)$$

where, a, b : size of focus

α : tilt angle of y focus

θ_x, θ_y : fan angle

ω : incident angle in y direction related with ϕ as $\phi + \omega = 60^\circ$.

Since the irradiation area dS changes with the ϕ (or ω) angle through dy , the measured residual stress values can be recognized as an average in such a range of dS . For example, the dependence of dx , dy , and dS on the X-ray incident angle ω or ϕ is demonstrated in Fig. 3, which was calculated for the case of

Fig. 1. Diffraction profile of β -Si₃N₄ by Cr-K α . ((212) plane ($2\theta_0=131.47^\circ$) was mainly used for the measurement of residual stress distribution.)

Fig. 2. X-ray optical systems and irradiated area in case of $\phi 0.1 \text{ mm}$ collimation.

$\phi 0.1$ mm collimation by setting $a=0.5$ mm, $b=10$ mm, $\alpha=6^\circ$, $L_1=242$ mm, and $L=250$ mm. In the measured $\psi(\omega)$ range, dy changed from 0.17 to 0.33 mm and dS from 0.016 to 0.031 mm². For the case of a $\phi 0.3$ mm collimation system, dS has already reported as 0.13–0.17 mm².⁵⁾ These dS values will be

Fig. 3. X-ray incident angle (ω or ψ) dependence of irradiated diameter and area in $\phi 0.1$ mm collimation system used in this study ($5 < \psi < 35^\circ$).

Fig. 4. Two types of $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joined specimens used for measurement of residual stress distribution.

discussed together with their effect on the residual stress value (Chap. 4).

3. Experimental

The specimens used were two $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joints brazed by $\text{Ti}-\text{Cu}-\text{Ag}^{6)}$ in which 13.0 mm low carbon steel (S45C) was sandwiched by pressureless sintered Si_3N_4 plates via a 0.2 mm Cu buffer layer, as shown in Fig. 4. The dimensions and surface finishings of the specimens were (a) $4 \times 40 \times 3$ mm with a ground surface and (b) $30 \times 40 \times 3$ mm with a lapped surface. The bonding strength was maximized to 350 MPa in 4 point bending by optimizing the buffer thickness.⁶⁾ An as-bonded $\text{Si}_3\text{N}_4/\text{steel}$ rectangular bar without a Cu buffer layer having a dimension of $12 \times 12 \times 40$ mm was used to compare the collimation effect.

The residual stress values were measured under three kinds of X-ray conditions, Cases 1 to 3, listed in Table 2. In these cases, the X-ray collimator and the irradiated area were different to check their effects on the residual stress value, 8 mm² in Case 1, max. 0.17 mm² in Case 2 and max. 0.031 mm² in Case 3 for Si_3N_4 . The optical system, shown in Fig. 2, corresponded to Case 3 having a $\phi 0.1$ mm collimator, in which case the X-ray power was increased up to 5 kW using a rotating target. The detected lattice plane was selected from Table 2 as (212) for Si_3N_4 and (211) for steel with $\text{Cr}-\text{K}\alpha$ as a reliable one with small penetrating X-ray. The directions of the residual stress locally obtained were vertical (y) and parallel (x) to the joined interface, as a normal stress, for every point measured.

4. Effects of Beam Collimation on Residual Stress Value

Because of the averaging effect, the residual stress values are influenced by X-ray collimation and the irradiation area especially in the region having a large stress gradient, such as that close to the interface or the anomaly point. To check these effects, collimation was changed as shown in Cases 1 to 3 of Table 2 in the neighborhood of the interface of the slim specimen (a).

Fig. 5 shows the axial distribution of the residual

Table 2. Three experimental conditions used in this study for measurement of X-ray residual stress distribution.

	Case 1		Case 2		Case 3
	Si_3N_4	Steel (S45C)	Si_3N_4	Steel (S45C)	Si_3N_4
Apparatus	Rigaku MSF	Rigaku SPPC	Rigaku PSPC		Rigaku MXD
X-ray tube	V-K α , Ti-filter	Cr-K α , V-filter	Cr-K α , V-filter		Cr-K α , V-filter
	30 kV, 6 mA	30 kV, 10 mA	40 kV, 35 mA		50 kV, 100 mA
Collimator	$2 \times 4 (\times 5)$ mm	1×2 mm	0.3 mm ϕ		0.1 mm ϕ
Irradiated area	$> 8 (\times 5)$ mm ²	> 2 mm ²	$0.13-0.17$ mm ²		$0.016-0.031$ mm ²
ψ angle (deg)	0, 5, 10, 15, 20, 25, 30, 35, 40		0, 15, 25, 30, 40		5, 15, 20, 25, 30, 35
	Iso-inclination		Side inclination		Iso-inclination
Lattice plane	$\beta\text{-Si}_3\text{N}_4$ (411)	Fe (211)	$\beta\text{-Si}_3\text{N}_4$ (212)	Fe (211)	$\beta\text{-Si}_3\text{N}_4$ (212)
	$2\theta_0=152.67^\circ$	$2\theta_0=156.1^\circ$	$2\theta_0=131.47^\circ$	$2\theta_0=156.1^\circ$	$2\theta_0=131.47^\circ$
Stress constant	$K=-48.59$ kg/mm ²	$K=-32.44$ kg/mm ²	$K=-90.08$ kg/mm ²	$K=-32.44$ kg/mm ²	$K=-90.08$ kg/mm ²

stress in the y direction, σ_{Ry} , measured conventionally with 2×4 mm V-K α irradiation (Case 1). The σ_{Ry} value at the "interface" was about 70 MPa in tensile and those for the two sides coincided well with each other. The axial distribution on the center line of the same specimen was also obtained with $\phi 0.3$ mm collimation (Case 2: irradiated area was $0.13\text{--}0.17$ mm 2) in the region 2.0 mm away from the interface, as shown in Fig. 6. It is noteworthy that fine distributions of the normal stress could be obtained within 2.0 mm away from the interface, and that the average value of σ_{Ry} corresponded to about 70 MPa (Actually, that in the whole region along the interface should be additionally averaged.). The maximum value of σ_{Ry} reached 240 MPa in tensile very close to the interface and σ_{Rx} was 100 MPa in compression. In the case of $\phi 0.1$ mm collimation (Case 3), the irradiated area ranged between $0.016\text{--}0.031$

Fig. 5. Residual stress axial distribution in $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joint (a) measured with 2×4 mm X-ray irradiation (hatched) in Si_3N_4 . (Distributions of both sides—solid and open circles—coincide well with each other.)

Fig. 6. Residual stress axial distributions in $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joint (a) measured with $\phi 0.3$ mm collimated Cr-K α . (More detailed distributions could be obtained in the same range as Fig. 5.)

mm 2 , as shown in Fig. 3, and σ_{Ry} was as high as 250 MPa.

It is clear that the smaller the irradiation area becomes the measured residual stress value saturates at the same position of the specimen. The feature of such saturation with dS are lucidly shown in Fig. 7, in which that of a directly brazed $\text{Si}_3\text{N}_4/\text{steel}$ is also compared. In Fig. 7, all the results measured for Cases 1 to 3 are summarized including Figs. 5 and 6. The residual stress values, σ_{Ry} , increased as the X-ray irradiated area decreased and saturated below 0.2 mm 2 in both systems. This fact means that the X-ray beam should be collimated smaller than $\phi 0.3$ mm, and $\phi 0.1$ mm is recommended for residual stress measurement in a ceramic/metal joint.

The effect of residual stress reduction by a Cu

Fig. 7. Dependence of residual stress value, σ_{Ry} , on X-ray irradiated area, dS , in Si_3N_4 very close to interface joined with steel. (Solid square denotes that for another cut-off $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joint (a). See text and Fig. 3 on the range of irradiation.)

buffer layer is also seen in Fig. 7. The residual stress in directly brazed $\text{Si}_3\text{N}_4/\text{steel}$, which reached up to 400 MPa, was reduced to below 250 MPa mainly by the plastic deformation of ductile Cu. An optimized Cu layer thickness will minimize the residual stress around a ceramic/metal joined interface as reported before.¹⁾

5. Residual Stress Distributions around a $\text{Si}_3\text{N}_4/\text{Steel}$ Joined Interface

The residual stress distribution was measured along the interface of lapped $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}(\text{S45C})$ 30 mm in width (shape (b) in Fig. 4) with $\phi 0.1$ mm collimated Cr-K α . The points measured were on a line 0.1 mm away from the interface, and their intervals were tight around the anomaly points. Two normal stresses, σ_{Rx} and σ_{Ry} , were obtained on each point by specifying the X-ray incident direction. The characteristics of the distribution shown in Fig. 8 were as follows:

- 1) large tensile residual stress in σ_{Ry} about 200 MPa at both edges, and also tensile in σ_{Rx} ;
- 2) steep stress drops (or changes into compression) in two stresses 1–2 mm inside the edge;
- 3) σ_{Ry} maximum at the center region of the specimen;
- 4) small compressive stresses in σ_{Rx} ; and
- 5) asymmetry profile of distribution.

Now, the observed stress distribution will be discussed with a comparison of stress analysis. Large tensile stresses at the edges of the joint has been generally predicted even by a two dimensional FEM analysis, but a large maximum at the center region was not predicted. The σ_{Ry} maximum at the center region was supposed to stem from three dimensional

Fig. 8. Detailed distributions of two normal stresses, σ_{Rx} and σ_{Ry} , in lapped Si_3N_4 along interface of $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$ joint (b). (Measurement was on line 0.1 mm away from the interface.)

effect from inside the rectangular bar which is planned to be proved in the near future by three dimensional FEM analysis. The steep stress drop 1–2 mm inside originated by a stress relief through the localized plastic deformation of the Cu buffer. Asymmetry distribution came from an unbalance in specimen preparation.

The authors believe that this is the first study to evaluate such a detailed profile of residual stress in a ceramic/metal joint quantitatively. Non-destructive evaluations of actual joined components and the verification of analysis can be achieved by the collimated X-ray method developed. Further experiments in various dimensions and combination of joints will surely reveal the nature and origin of stress distribution.

6. Conclusions

The effects of X-ray collimation in the $\sin^2\psi$ method were studied to develop the most effective evaluation method of the residual stress distribution around the interface of a ceramic/metal joint. The optimized conditions were applied to measure the detailed residual stress distribution along the interface of $\text{Si}_3\text{N}_4/\text{Cu}/\text{steel}$. The results were as follows;

(1) For Si_3N_4 , (212) diffraction ($2\theta_0 = 131.47^\circ$) by Cr-K α was selected as the first candidate with small penetration depth. The second was (323) plane by Cu-K α .

(2) In the Si_3N_4 region close to the interface of the joint, the residual stress values increased and finally saturated as the X-ray collimation and irradiation area decreased from $2 \times 4 \text{ mm}^2$ to below 0.03 mm^2 . The normal stress value across the interface saturated below 0.2 mm^2 to 240 MPa for $\text{Si}_3\text{N}_4/0.2\text{Cu}/\text{steel}$, and to 400 MPa for $\text{Si}_3\text{N}_4/\text{steel}$.

(3) It should be noted that $\phi 0.1$ mm collimation having irradiation areas 0.016 – 0.031 mm^2 is recommended to evaluate quantitatively the detailed residual stress distribution along the joined interface.

(4) Large tensile normal stresses at both edges, the minimum stress points 1–2 mm inside the edges, the maximum σ_{Ry} at the center region of the specimen, and the asymmetry profile of distribution were successfully observed along the joined interface of $\text{Si}_3\text{N}_4/0.2\text{Cu}/\text{steel}$ 30 mm wide with a lapped surface.

(5) The maximum normal stress in the center region was first observed which could have been caused by the three dimensional effect of the joined plate. The minimum stress points were originated by stress relief through localized plastic deformation of the ductile Cu buffer layer.

(6) The effective measuring method of the residual stress detailed distributions in ceramics can be achieved by $\phi 0.1$ mm collimated X-ray as a tool of non-destructive evaluation in actual joined components and the verification of FEM analysis.

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