RESIDUAL STRESS DISTRIBUTION IN THE SURFACE NEAR THE JOINING BOUNDARY OF $\beta$-Si$_3$N$_4$ AND CARBON STEEL

S. TANAKA* and K. OGUISO**

* New Material Engineering Lab., TOSHIBA Corporation
** Research & Development Dep., Rigaku Corporation

Studies were made about the effect of the size of the irradiation area on the stress value at the time of X-ray stress measurement of very small regions to the extent of 0.1 to 0.5 mm dia. Selected as the object for measurement was a joined body of ceramics and metal in view of the fact that their thermal expansion coefficient greatly differs from each other. We have learned as a result that the use of an X-ray stress analyzer where a microfocus X-ray tube and a PSPC are combined enables measurement of residual stresses in a very small region of about 0.3 mm dia.

1. Introduction

The recent development of new materials is noteworthy. From the viewpoint of practical use, however, it is often the case that an important problem arises as to how to fabricate them together with existing materials. The same is the case with the joining of ceramics and metals, where residual stresses caused by mismatch in the thermal expansion coefficient and elastic constant as well as the stress concentration are said to greatly affect the joining strength. In particular, the region near the joining boundary is the so-called specific point, and while residual stress distribution analysis by the finite element method is being used for such regions, there is an increasing demand for a method that enables actual measurement [1]. As an expedient method to realize it, the indentation fracture technique [2] is available. But this method may not be said to be a satisfactory one because local residual stress relief due to cracks should also be taken into account.

For the X-ray stress measurement method an aggregate of fine crystal grains like ceramics is an ideal object to handle in terms of the isotropy. However, conventional X-ray stress measuring units generally tend to use a broad focus X-ray tube in order to get continuous Debye rings even from crystal grains of tens of microns or so by broadening the area for irradiation. When, therefore, dealing with the stress measurement of a very small section of 2 to 3 mm, there occurs a need to narrow down the incident beam resulting in shortage of the incident X-ray intensity. To overcome the problem, we have already been conducting X-ray stress measurement by using a high-brilliance fine focus X-ray tube and collimating the beam with a 0.3 mm dia. collimator to irradiate the region near the joining boundary. A position-sensitive proportional counter (PSPC) is also used together [3]. This paper reports our examinations of the relations between the stress distribution and the irradiated area, and of the joined structure and the residual stress distribution.

2. Test Pieces

Fig. 1 shows the joined structure and the dimensions of test pieces used for the measurement. Vacuum brazing by the activation metal method was carried out for the joining of these test pieces. In Fig.
1(A), $\beta$-silicon nitride ($\text{Si}_3\text{N}_4$) and carbon steel S45C were directly joined to each other. In Fig. 1 (B), a copper sheet of 0.2 mm thickness was inserted for the purpose of thermal stress relaxation. In Fig. 1(C), S45C of 3 mm thickness, sandwiched on both sides with copper sheets of 0.2mm thickness, was put between two $\text{Si}_3\text{N}_4$ pieces and was joined. The surfaces for measurement remained in the same state as when they had undergone the surface grinding finish as individual parts prior to joining. The flow of the brazing material to the vicinity of the joining boundary was within 0.1 mm, and the boundary line was clearly distinguishable.

3. Outline of the X-ray Stress Analyzer

Besides the capability as a linear detector, the PSPC is characterized by not being affected by the X-ray source output variations unlike other counter scanning methods. It may be said to be an optimum counter for the measurement herein discussed which deals with very small regions and which requires sufficiently collimated incident beams. Needless to say, however, it is impossible to infinitely make thinner the beam, and there are also restrictions by the X-ray tube brilliancy of itself in addition to those by the crystalline state and the crystal grain size of the object for measurement.

Fig. 2 shows the overall view of the Rigaku PSPC system micro X-ray stress analyzer used for this measurement. The regions for measurement were determined with a microscope whose focal spot depth is 50 microns and whose magnification is 40. The measurement was conducted by the $\sin^2 \psi$ side inclination method at a $5^\circ$ incident angle. The X-ray stress analyzer has a computercontrolled X-Y stage for the sample holder and allows stress distribution measurement exactly as programmed. The X-ray conditions are given in Table 1. For calculations of the stress constants, we used the X-ray elastic constant measurement values obtained by Hanabusa et al. [4].

4. Examination of the X-ray Irradiated Area

It is expected that residual stresses in the vicinity of the joining boundary present steep variations in a very small region. In such a case there is a likelihood that the form of the stress distribution curve is smoothed depending upon the X-ray irradiated area. We accordingly made some examination about this.

Fig. 3 shows the relationship between the divergent optical system of the X-ray stress analyzer and the irradiated area in the case of the side inclination method. The pinhole diameter Od value is used for the collimator designation and the divergence angle $\omega$ is determined by the scatter pinhole S and the collimator length $L_1$. The designed values are listed in Table 2. The irradiated area $\phi A$ will be broadened as the incident angle $\psi$ is inclined. The equation (1)

$$A = \frac{1}{\cos \theta_1} \left\{ \frac{1}{\cos \theta_1} \right\}^2 + \left( \frac{1}{\cos \theta_1} \right)^2 - \frac{2 \cos (\theta_1 + \theta_2)}{\cos \theta_1 \cdot \cos \theta_2} \frac{1}{\cos \psi}$$

Table 1 Conditions of X-ray stress measurement.

| Lattice plane | $\beta$-Si$_3$N$_4$ (212), 2$\theta$=131/5 |
| X-ray tube   | CrK$\alpha$ 30 kV, 45 mA |
| Collimator  | $\Phi$ 0.1, 0.3, 0.5, 1, 2 |
| Detector    | 100 mm PSPC |
| Tilt Angle  | Side Inclination Method $\psi$=0, 15, 25, 30, 40$^\circ$ |
| Stress constant | $K = -90.8 \text{ kg/mm}^2$ |
where
\[ \tan \theta_1 = \frac{(S - d)}{2L} \]
\[ \tan \theta_2 = \frac{(S + d)}{2L} \]

the relations between the irradiated area \( \phi A \) and the incident angle \( \psi \). The calculation result is also put in Table 2. The actual irradiated area would take an elliptic shape having the longer axis in the direction of the measured stress. But since the shorter axis in the traverse direction is constant, the longer axis is indicated as the diameter in Table 2, for convenience sake. Fig. 4 shows the measurement result of the residual stress distribution in the longitudinal direction on the center line of specimen A with collimators 0.3, 0.5 and 1 mm dia. The abscissa denotes the distance from the joining boundary along the center line on the \( \text{Si}_3\text{N}_4 \) surface and the ordinate represents the residual stress value. The circles on the center line show the X-ray irradiated area at \( \psi = 40^\circ \) with respect to each of the collimators. For reference, moreover, on the ordinate are shown the specimen width values which are identical with those on the abscissa.

The same applies to Fig. 5(A) through Fig. 5(C) but with no particular mention on it. With the 0.3 mm dia. or the 0.5 mm dia. collimator, no overlap of the irradiated area is seen even at the adjacent measuring points; whereas, with the 1 mm dia. collimator, an overlap by about a half of the irradiated area is recognized at the respective two adjacent measuring points. While the use of the 0.3mm dia. collimator may not necessarily offer the accurate distribution, the fact is that broadening of the area for irradiation resulted in a somewhat lower distribution, possibly due to leveling-off by the adjacent stress values. With this in mind, we invariably used the 0.3mm dia. collimator for the succeeding measurements. We had also tried a 0.1 mm collimator but the resulting intensity was insufficient to assure us of the reliability of the measured value. Moreover, it took three hours or more for measurement per piece. We therefore gave up the use of this collimator for our present experiment.

### Table 2

The variation of X-ray irradiated area \( \phi A \) with collimated diameter \( \phi d \) and divergent angle \( \omega \).

<table>
<thead>
<tr>
<th>( \phi d )</th>
<th>( \omega^\circ )</th>
<th>( \phi A(\psi=0) )</th>
<th>( \phi A(\psi=40) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.13</td>
<td>0.15</td>
<td>0.38</td>
</tr>
<tr>
<td>0.3</td>
<td>0.40</td>
<td>0.41</td>
<td>0.53</td>
</tr>
<tr>
<td>0.5</td>
<td>0.68</td>
<td>0.67</td>
<td>0.88</td>
</tr>
<tr>
<td>1.0</td>
<td>1.32</td>
<td>1.36</td>
<td>1.77</td>
</tr>
</tbody>
</table>

The measured regions were either located on a straight line 0.3 mm away from the joining boundary and parallel to it or on the center line of the specimen. Both of them were on the \( \text{Si}_3\text{N}_4 \) side, anyway. Fig. 5(A) through Fig. 5(C) illustrate the measurement results, and in each case (a) shows the residual stress distribution along the joining boundary and (b) shows the same distribution along the center line, In Fig.
Fig 5. The measurement results of specimens A, B and C. In each case, (a) and (b) respectively show the residual stress distribution along the boundary line and that along the center line.
5(A), Si₃N₄ and S45C are directly joined without a copper sheet as an intermediate material. It is seen that as the measured region nears the center area along the boundary, the tensile residual stress tends to increase in the z direction and the compressive residual stress tends to increase in the z direction. The end regions show tensile residual stresses of several kg/mm² in both directions. Along the center line, on the other hand, as the measured region becomes more distant from the boundary, a gradual decrease in the residual stress is observed in each direction, virtually reaching an equilibrium at 2 mm away. For both (a) and (b), σ_z and σ_t are symmetrical. Fig. 5(C) shows the measurement result in only the a. direction but the tendency appears about the same as in Fig. 5(C). Notably, the residual stress decreased by 20 to 30 percent due to the effect of the copper sheet as a buffer against the thermal stress. Fig. 5(B) presents an inexplicable distribution. While it shows roughly the same pattern as in Figs. 5(A) and 5(C) for the σ_z direction along the boundary, it gives an entirely different pattern for the σ_t direction. Further, the distribution along the center line in (b) shows a discontinuous point existing at a 1 mm distance from the boundary. What is common to (a) and (b) are those distributions that appear to respond to each other in the σ_z and σ_t directions. The cause is not clear at this moment, but it may be surmised that there are complicated elasto-plastic constraints due to extremely small unjoined portions at the boundary. Since this sort of thing is unavoidable in brazing, it seems necessary to increase the measurement example of specimens of the same joined structure treated under the same condition and to clarify the correspondence to the observed unjoined boundary through some possible means.

For the analysis by the finite element method, Kobayashi et al. conducted the thermal elasto-plastic analysis of joined structures in the form of specimen B [5]. The result shows that the stress distribution in Si₃N₄ near the boundary is virtually identical with (b) of Fig. 5(A) and 5(C).

6. Summary
The measurement result by X-ray diffractometry of surface residual stress distributions near the joined boundary between the ceramic and metal may be summarized as follows.

1) It has been found out that a reduction of the area for irradiation with a 0.3 mm dia. collimator permits X-ray stress measurement of such an extremely small regions near the joined boundary.
2) The surface residual stress along the joined boundary is minimal at a 1 to 2 mm distance from the edge, and shows the maximum single peak distribution at the center.
3) The surface residual stress along the center line of the joined body presents a high stress concentration toward the boundary, and the distribution in the center-line direction and that in the direction normal to it are reversely symmetrical to each other.
4) Insertion of a copper sheet between the ceramic and metal will decrease the residual stress in the vicinity of the boundary.
5) An irregular stress distribution may take place depending upon the joining condition at the boundary.

What we have done is no more than the X-ray stress measurement itself. We intend to work successively on clarification of the correspondence to the analysis by the FEM, the observed joined boundary, the bending test, and so on.

References

[This paper was presented at International Conference on Residual Stress Second (NANCY, FRANCE, Nov., 1988)]