ESRF	**Experiment title:** RESIDUAL STRESSES IN METAL/CERAMIC (Ni/Si_3N_4) DIFFUSION BONDS	**Experiment number**: ME-374
Beamline: ID-11	**Date of experiment**: from: 04-April-2002 to: 10-April-2002	**Date of report**: 26-02-2003 *Received at ESRF:*
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Report:

In the ceramic / metal joining process, due to the difference in the elastic modulus and thermal expansion coefficient of ceramic and metals, high stress concentration and residual stress are induced near to the joining interface. Such stresses are responsible of the possible plastic deformation and cracking and thereby affect the mechanical properties of the joints. Therefore, it is necessary to evaluate the residual stress magnitude and distribution along the interface because these data influences the strength of the joints due to the fact that a reduction of the stress increases the strength of the joints. Under these circumstances, it becomes of great interest to determine the residual stress distribution near the joint interface in regions inside the bulk of the samples.

High resolution X-ray scanning diffractometry has been performed at 80 keV to reduce the absorption due to the ceramic body, and, consequently, scan lines can be selected well inside the ceramic in order to study the strain and stress fields along the whole sample.

Here is the report of the experimental results of the directly obtained strain data along several lines passing perpendicularly through the interface inside silicon nitride blocks joined to a nickel block. The studies have been focussed to ceramic/metal joints and X-ray scattering volume have been selected to determine the strain in regions along some characteristic lines near the surface, as well as, in the symmetrical centre of the ceramic blocks. Diffraction experiments was performed in the bragg-brentano geometry (θ–2θ

arrangement) at the kappa diffractometer. The axial strains (perpendicular to the bonding interface) were measured using slits of 0.05 mm x 2.00 mm (height x width) and the radial strain (paralell to the bonding interface) were measured using slits of 0.05 mm x 0.50 mm (height x width).

Using the Bragg's equation, the strain (ε) is defined as the relative variation of the actual lattice spacing (d) respect to the strain-free spacing (d_0) and is related with the angle shift ($\Delta\theta$) by

$$\varepsilon = (d-d_0)/d_0 = -\cot(\theta)\,(\Delta\theta)$$

The position of the peaks shows a negligible variation when a ceramic block (before joining) is compared with the powder ($\beta-Si_3N_4$ powder was obtained by milling of a previous conformed ceramic block). On that way, it is possible to establish that strains before the joining process is always less than $\pm 5 \times 10^{-5}$ at any part of the block. The higher angle value gives a higher precision in the determination of the relative variation of strains. In the following, measurements of the (301)-diffraction peak will be considered to determine the behaviour of the strain in the joined samples.

Several samples are studied: SiN-Ni(1) was prepared under a uniaxial pressure of 50 MPa at a temperature of 1060ºC and the cooling rate up to room temperature was 2ºC/min; and SiN-Ni(2) sample was prepared similarly but at different temperature of 1150ºC.

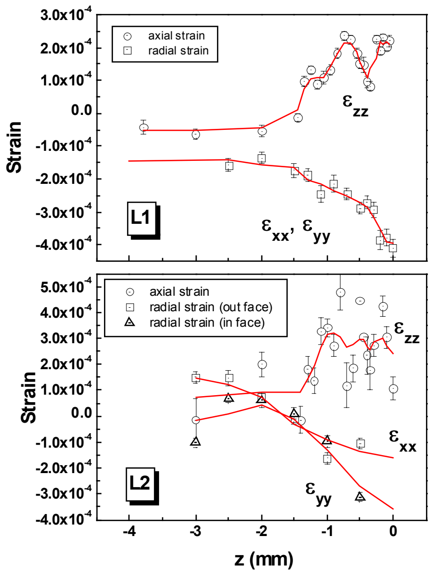

Fig. 1

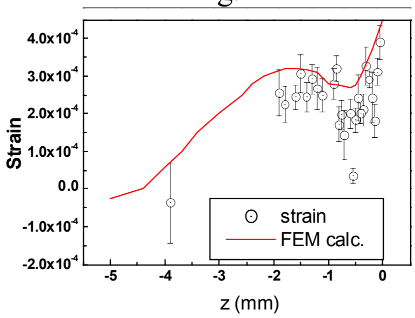

Fig. 2

Fig. 1 presents the axial strain data obtained from sample SiN-Ni(2) along two different lines perpendicular to the joining interface, L1 is at the centre of the sample and L2 at the centre of the face (here, axial denotes the strain component parallel to the applied axial pressure and therefore perpendicular to the joining interface). The origin of the z coordinate has been fixed at the metal-ceramic interface and position steps between scattering volumes have been taken shorter near the interface. It can be observed that, for the axial component, there is a non-monotonous behaviour of the strain.

Additionally, Finite Element Method calculations have been performed in order to fit the experimental data. Fig. 2 shows the agreement between experimental results and calculations for sample SiN-Ni(1).

Those results permit the future study of some other ceramic/ceramic joining based on the coating of each ceramic body with a metallic thin film as the Ni or even with a low temperature brazing material as the TiCuSil alloy.