ELSEVIER

Journal of Nuclear Materials 258–263 (1998) 1005–1009

journal of nuclear materials

High-temperature residual strain measurements, using neutron diffraction, in brazed Cu/CFC graphite divertor structures [1]

M. Ceretti [a], R. Coppola [b,*], E. Di Pietro [c,2], C. Nardi [c]

[a] *LLB, CEA-Saclay, 91191 Gif-sur-Yvettte Cedex-F, France*
[b] *ENEA-Casaccia, CP 2400, 00100 Rome-I, Italy*
[c] *ENEA-Frascati, ERG-FUS, CP 2400, 00100 Rome-I, Italy*

Abstract

This work presents a study of residual strain evolution in a three layer castelled mock-up for ITER obtained brazing carbon fibre composite (CFC) graphite with dispersion strengthened Cu (and with a GLIDCOP interlayer). Neutron diffraction measurements were carried out, on graphite at 30°C, 300°C and 600°C and on dispersion strengthened (DS) Cu at 600°C. For each material, the reference, unstrained value of the lattice parameter was obtained by measuring a powder of the same material at the same temperatures. While in DS Cu negligible strain values are found at high temperature, a more complicated evolution of the strains is observed in CFC graphite relating to sample geometry.

1. Introduction

It is well known that divertor components, such as brazed Cu/CFC graphite structures, must withstand thermal loads which modify the strain field and consequently the component reliability and lifetime. It is therefore of utmost importance to characterise the strain evolution with temperature, especially near the brazing. Neutron diffraction provides an appropriate experimental tool for these measurements, as it combines penetration depths of few mms inside the materials with linear spatial resolutions as low as 0.5 mm. Furthermore, ancillary equipments such as furnaces or mechanical testing machines can be installed on neutron diffractometers thus allowing in-situ measurements.

The general interest of neutron scattering in material science is well known [1,2] and studies concerning more specifically fusion reactor materials are reviewed in Refs [3,4]. Reports on neutron diffraction stress measurements on fusion relevant components can be found in Refs. [5–14].

This paper concerns castelled joints obtained by brazing carbon fibre composite (CFC) graphite with dispersion strengthened (DS) copper and is the continuation of preliminary neutron diffraction stress studies [8,13] carried out on similar components at room temperature. After describing the experimental details the results obtained at different temperatures in the two materials are presented and discussed.

2. Experimental conditions

The investigated sample was a castelled mock-up obtained by brazing CFC graphite (Dunlop) with an interlayer of OFHC Cu, brazed in turn to DS Cu. A sketch of the sample, designed for optimizing heat dissipation, is shown in Fig. 1, together with its sizes; the main axes defined for the neutron diffraction measurements are also shown in Fig. 1

The neutron diffraction measurements have been carried out using the G5.2 diffractometer installed at the "Orphée" reactor of Laboratoire Léon-Brillouin (LLB, CEA-CNRS Saclay). It is recalled that the

* Corresponding author. Fax: +39-6 3048 4747; e-mail: coppolar@infos1.casaccia.enea.it.

[1] Work carried out in the frame of ITER Task T 222 sub. 2.

[2] Present address: ITER-Naka JWS, 801-1 Mukouyama, naka, machi, naka-gun, 311-01 ibaraki-ken, Japan.

0022-3115/98/$19.00
PII: S0022-3115(98)00074-9

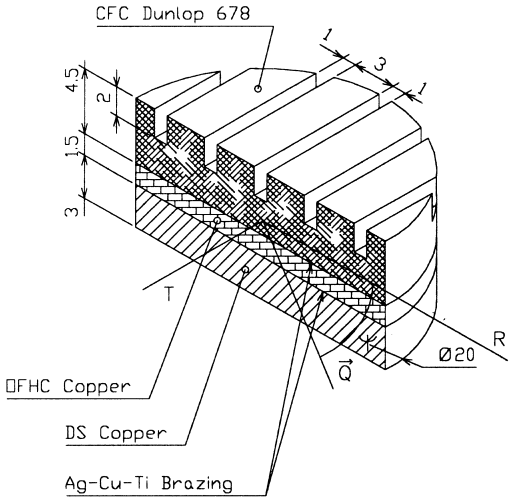

Fig. 1. CFC/ DS Cu/Glidcop cylindrical brazed structure. The diffraction vector Q and the chosen tangential (T) and radial (R) axes are reported together with the angle ψ. Figures are in mm.

determination of internal stresses by means of neutron (or X-ray) diffraction is based on the well-known Bragg's law

$$n\lambda = 2d_{hkl} \sin \theta_{hkl}, \tag{1}$$

relating the angle 2θ where the diffraction peak is observed for a given wavelength, λ, to the corresponding lattice spacing d_{hkl} for the Miller indices (hkl). If d_0 is the value of the unstrained lattice spacing the strain in a direction ε perpendicular to the diffracting planes is obtained as

$$\varepsilon = \frac{d_{hkl} - d_0}{d_0}, \tag{2}$$

and then, providing at least six independent values of ε and the stiffness coefficients matrix, C, the stress tensor is obtained as

$$\sigma = C\varepsilon. \tag{3}$$

ε is generally expressed as a function of the Euler angles ψ and ϕ which correspond to the physical rotation of the sample, obtained by an Euler cradle. Namely, if the shear components of the strain tensor are zero, one can write

$$\varepsilon_{\psi\phi} = \left(\varepsilon_{xx} \cos^2 \phi + \varepsilon_{yy} \sin^2 \phi - \varepsilon_{zz}\right) \sin^2 \psi + \varepsilon_{zz}, \tag{4}$$

so that from a linear regression in $\sin^2 \psi$, the principal strains can be determined. The stress value is then obtained by solving Eq. (3). A review of the different mathematical methods to solve this equation system is given in Ref. [15] for instance. In the present measurements the linear size of the selected neutron probe, was 2 mm corresponding to a diffracting volume of some 10 mm^3. The experimental conditions for graphite were the following: the neutron wavelength was 0.47 nm, with a 2θ angle of approximately 90° for the (0 0 2) reflection. For DS Cu the neutron wavelength was 0.286 nm the angle θ_0 at 87° and the reflection the (1 1 1). Figs. 2 and 3 show the diffraction lines of the reference CFC graphite and DS Cu powders respectively. The instrumental resolution is about 0.5° for the case of CFC graphite and 0.4° for the case of DS Cu. It has to be noted that, as already discussed in an earlier paper [13] the diffraction peak from CFC contains a contribution from fibres and a contribution from the matrix, which cannot be distinguished with the available instrumental resolution. Therefore the measured lattice spacing refers to an average over the selected diffracting volume, which is large enough to integrate over these two phases, fibre and matrix, both in the reference powder and the sample.

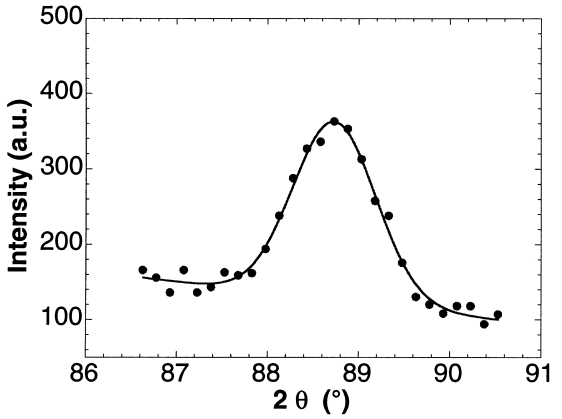

Fig. 2. (0 0 2) diffraction line for CFC graphite reference powder at RT.

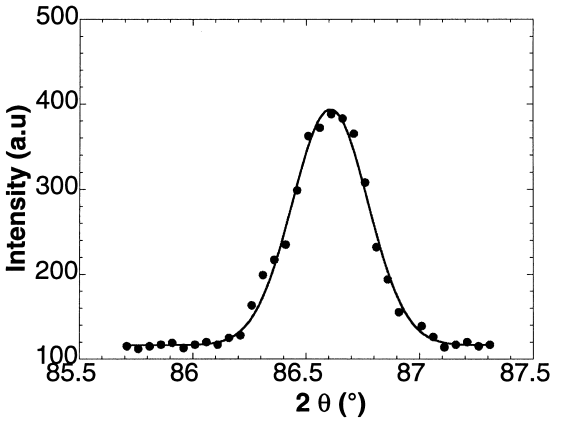

Fig. 3. (1 1 1) diffraction line for DS Cu reference powder at RT.

As the main objective of these measurements was to investigate the behaviour of the component at high temperatures, the sample was placed in a thermocouple furnace, in He, then installed on the rotating table of the G5.2 diffractometer. Using this equipment only the angle ψ could be varied, for a fixed ϕ value and a three-dimensional analysis, including the determination of the stresses, was not possible. More specifically, it was not possible to obtain information on strains in the direction perpendicular to the brazing interface (laying in the diffraction plane, see Fig. 1), while radial and tangential strains correspond to those measured for $\sin^2\psi$ values of 0 and 1 respectively. As the lattice parameters of each of the two investigated materials, CFC graphite and DS Cu, vary with temperature, in order to determine the strain values, at high temperature, it was necessary to prepare milled powders (approximately 3 cm^3) for both CFC graphite and DS Cu and to measure the respective diffraction peaks at different temperatures. In this way, a calibrated, temperature-dependent value of the unstrained lattice parameter was available to determine the strain at high temperature in each of the two materials. In Eq. (2) ε is therefore replaced by $\varepsilon(T)$. Strains in graphite were subsequently measured at 30°C, 300°C and 600°C (maximum available). Then strains in DS Cu were only measured at 600°C. As the time required for each measurement was 15–20 h the whole component underwent a complex thermal treatment.

3. Results and discussion

In order to have a preliminary check on the homogeneity of the mock-up, first the as-received component was characterised, at room temperature, by measuring the strain in the four points indicated in Fig. 4(a), in the graphite, as close as possible to the brazing surface. The strain values, shown in Fig. 4(b), are quite scattered and suggest the presence of texture effects. The graphite powder was studied at the following temperatures: 30°C, 100°C, 300°C, 450°C, and 600°C. The results, given in Fig. 5 and Table 1, show a good agreement with data available in the bibliography [16]. The sample was then installed in the furnace and the room and high temperature strain measurements were carried out, in position 4. For each temperature, the strain value was determined by using the reference d_0 values of Fig. 5. The results are given in Fig. 6. First it has to be noted that the deformation trends exhibit deviations from linearity, to be very likely ascribed to texture effects as seen in previous studies. At room temperature a negligible radial strain is found, while in the tangential direction a tensile deformation of approximately 0.0006 is found. With increasing the temperature, radial strain becomes negative (−0.0006 approximately) and tangential one negligible. Taking into account the experimental error bars and the dispersion of the points, no significant difference can be detected between 300°C and 600°C data.

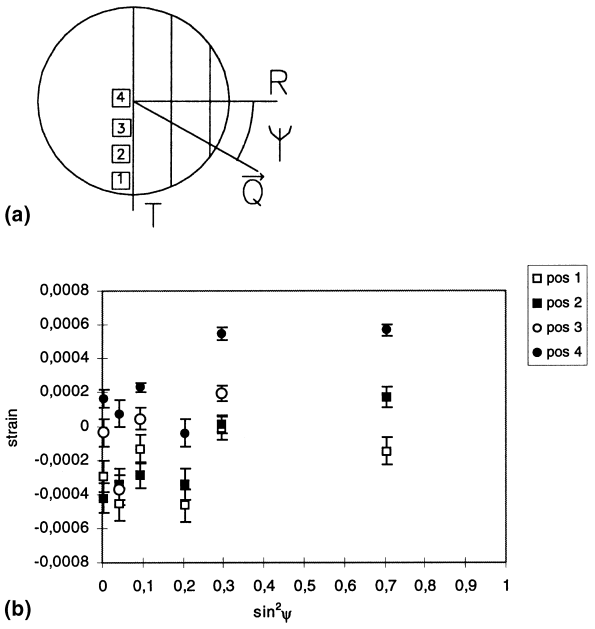

Fig. 4. (a) Points investigated at room temperature on the CFC graphite side in the sample of Fig. 1; (b) corresponding deformation values.

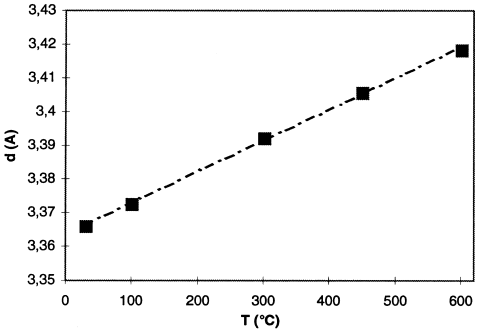

Fig. 5. Lattice parameters measured by neutron diffraction as a function of temperature for CFC graphite reference powder.

Table 1
Comparison between d vs. T for CFC as from Ref. [16] and as from neutron diffraction

Temperature (°C)	$d_{calculated}$	$d_{experimental}$
40	3.3665	
300	3.3925	3.3923 ± 0.0003
600	3.4225	3.4187 ± 0.00025

$y = A + Bx, A = 3.36248 \pm 0.00104$, $B = 0.0001 \pm 2.86 \times 10^6$, $R = 0.99866$.

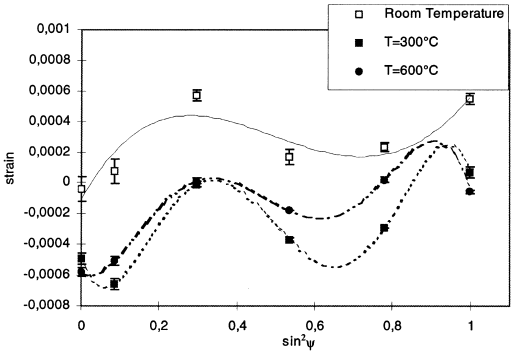

Fig. 6. Deformation in CFC graphite, in point 4 (see Fig. 4(a)), as a function of temperature.

Fig. 7 shows the calibration of DS Cu reference powder as a function of temperature. Also in this case the strain values were determined by using the reference d_0 of the DS Cu powder measured at 600°C. The strain in Cu (measured at the centre of the component and as close as possible to the brazing surface) is also shown in Fig. 7. The obtained value coincides with the one of the reference powder, so that the deformation is zero. This is entirely consistent with what is known on the temperature resistance of DS Cu.

It has to be pointed out that these results may be affected by the combination of the different thermal treatments performed on the whole mock-up, which may have nonnegligible consequences especially on the microstructural evolution (precipitates) of GLIDCOP.

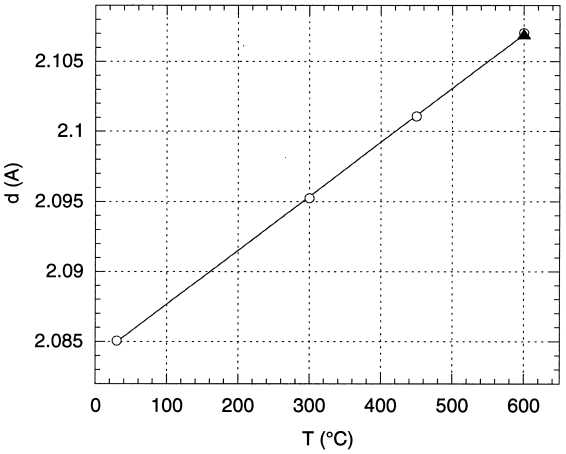

Fig. 7. Lattice parameters measured in DS Cu powder, as a function of temperature; the strain value measured in the DS Cu of the sample in Fig. 1 (in a point symmetrical with respect to point 4 of CFC), at 600°C, is also reported (full triangle).

4. Conclusions

The results presented in this report contain an original contribution to the general problem of determining the evolution of strain field, under fusion relevant conditions, in brazed CFC graphite/Cu mock-up's. In fact it has been shown that neutron diffraction is an accurate technique providing reliable values of the strain field both in CFC graphite and in the brazing metal. It will be however possible to derive more quantitative conclusions from these series of experiments only on the basis of FEM calculations reproducing the samples and the experimental conditions investigated in this study. On the other side, it must be pointed out that the geometry of the investigated sample is a very "refractory" one, as the calculations are extremely sensitive to the hypothesis put on the heat exchange from the castelled volume. Indeed, if the exchange area is evaluated only as the one of the upper surface, a very slow temperature reduction is obtained, while if the full surface of the castellation is evaluated, including the lateral walls, the time of temperature reduction drops dramatically.

There are also several points requiring further investigations and calibration of the experimental method. First, it should be clarified whether the semiempirical method reported in Ref. [13] for correcting the diffraction data from textured CFC graphite and determining the stress value in this material may be more generally applied. Representative samples of the graphitized fibres are necessary to try and distinguish the different strain

contributions in the diffraction peaks. The crystallographic texture can moreover be used to correct deviations from linearity in linear regressions. A preliminary check of the crystallographic texture in the samples to be investigated for stress or strain measurements is recommended.

Concerning the sample preparation, a careful control of the initial strain level, prior to brazing, is indispensable if sound and reliable comparisons with FEM are expected. It should also be discussed whether in-situ temperature measurements may be replaced by room temperature measurements on annealed mock-up's. It would be interesting to compare these in-situ results with those which might be obtained heating for a given time an identical mock-up and carrying out the neutron diffraction measurements at room temperature.

References

[1] M.T. Hutchings, C.G. Windsor, Industrial Applications in: K. Skoeld, D.L. Price (Eds.), Methods of Experimental Physics, vol. 23, Neutron Scattering, Academic Press, New York, 1987, Part C, p. 405.
[2] M. Fontana, F. Rustichelli, R. Coppola (Eds.), Proc. Int. School od Physics "E. Fermi", Course CXIV, Industrial and Technological Applications of Neutrons, Lerici, North-Holland, Amsterdam, 1992.
[3] G. Albertini, R. Coppola, F. Rustichelli, Phys. Rep. 233 (3) (1993) 137.
[4] G. Albertini, F. Carsughi, R. Coppola, F. Rustichelli, Plasma Dev. Op 3 (1994) 293.
[5] G. Albertini, M. Ceretti, R. Coppola, A. Lodini, M. Perrin, F. Rustchelli, Proc. NATO Workshop, in: M.T. Hutchings, A.D. Krawitz (Eds.), Measurements of Residual and Applied Stress using Neutron Diffraction, Oxford, 1991, NATO ASI Series, vol. 610, p. 525.
[6] G. Albertini, M. Ceretti, R. Coppola, R. Jakeman, A. Lodini, P. Mariani, R. Matera, M. Perrin, F. Rustichelli, G. Vieider, Proc. 18th SOFT, Rome, 1992, Fusion Technology, 1993, p. 147.
[7] G. Albertini, M. Ceretti, R. Coppola, S. Ghia, A. Lodini, P. Mariani, M. Perrin, F. Rustichelli, Met. Sci. Technol. 11 (1993) 18.
[8] M. Ceretti, R. Coppola, E. Di Pietro, A. Lodini, M. Perrin, A. Piant, F. Rustichelli, J. Nucl. Mater. 212–215 (1994) 1617.
[9] C. Braham, M. Ceretti, R. Coppola, A. Lodini, F. Rustichelli, S. Tosto, Proc 18th SOFT, Karlsruhe, 1994, Fus. Techn., 1995, 415.
[10] M. Ceretti, R. Coppola, A. Lodini, M. Perrin, F. Rustichelli, Physica B 213/214 (1995) 803.
[11] A.J. Allen, R. Coppola, M.T. Hutchings, M. Valli, C.G. Windsor, Mater. Lett. 23 (1995) 265.
[12] R. Coppola, in: A. Lodini, M. Perrin (Eds.), Analyse des contraintes résiduelles par diffraction des rayons X et des neutrons, CEA, 1995, p. 269.
[13] G. Albertini, M. Ceretti, R. Coppola, A. Lodini, E. Di Pietro, J. Nucl. Mater. 233–237 (1996) 954.
[14] C. Braham, M. Ceretti, R. Coppola, A. Lodini, presented at ICRS-5, Linkoeping, 1997.
[15] P. Pascal, Nouveau Traité de Chimie Minérale, Masson, Paris, 1968.
[16] I.C. Noyan, J. Cohen, Residual stress measurements by diffraction and interpretation, Mater. Res. Eng., Springer, Berlin, 1987.