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**DRAFT**

**NEUTRON DIFFRACTION QUALIFICATION OF W-LAMINATES FOR  
STRUCTURAL DIVERTOR APPLICATIONS**

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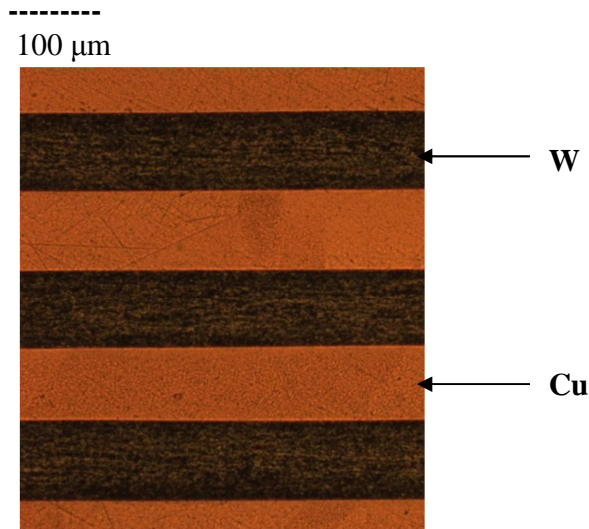
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**Abstract** - Neutron diffraction measurements have been carried out to develop a non-destructive experimental tool for characterizing the crystallographic structure and the internal stress field in W foil laminates for structural divertor applications. The model sample selected for this study had been prepared by brazing, at 1085°C, 13 W foils with 12 Cu foils. A complete strain distribution measurement through the brazed multilayered specimen and a determination of the corresponding stresses has been obtained, assuming zero stress in the through-thickness direction. The average stress determined from the technique across the specimen (over both ‘phases’ of W and Cu) is close to zero at  $-17 \pm 32$  MPa, in accordance with the expectations.

## **1. Introduction**

This experimental activity has been carried out in order to develop the neutron diffraction technique as a non-destructive experimental tool to characterize the crystallographic structure and the internal stress field in W foil laminates for structural divertor applications (1-3). As shown more specifically in ref. (3), different joining techniques are being considered to assemble such foils into larger structures and to build-up mono-blocks. The model sample selected for this study (fig. 1 from ref. 3) had been prepared by brazing at 1085°C 13 W foils (each one 0.1 mm thick) with 12 Cu foils (each one also 0.1 mm thick), in such a way to obtain a 2.6 mm thick foils, whereby stacks of 15-25 layers of each material, W and Cu, individually were prepared. For the reference specimens the foils were not brazed, but rather simply clamped together to form the stacks.



**Fig. 1-** Model brazed W-Cu multi-layer structure, after ref. (3).

The experimental investigation of this sample is complicated by the large elastic modulus ( $E_{hkl}$ ) of W, which makes it difficult to determine residual stresses accurately, and by the large grain size in the Cu, causing potential so-called grain size effects. The relatively large gauge volume in a neutron diffraction measurement does not allow for measurements in individual foils; therefore these measurements provide the averages of the strain distribution separately for the W and the Cu foils. However, if the strain across the specimen thickness varies, that variation would likely be averaged out in the measurements. Preliminary neutron diffraction measurements were carried out at the E3 diffractometer at the research reactor BER-II of the Helmholtz-Zentrum Berlin (HZB) (4-5). The experimental work was then completed at the HB5 diffractometer at the High Flux Reactor of the Joint Research Centre in Petten (6), under similar experimental conditions.

## 2. Experimental technique and data analysis

Reference is made to the literature (7-8) for a general presentation on the use of neutron diffraction for strain and stress determination and more specifically to (9-14) for some applications to fusion technology. The measurement of strains and stresses by neutron or X-ray diffraction is based on the well known Bragg law

$$2d_{hkl} \sin \theta = n\lambda \quad (1)$$

relating the spacing,  $d_{hkl}$ , between crystallographic lattice planes characterized by Miller indices  $hkl$  with the wavelength,  $\lambda$  and the angle  $2\theta$  where the reflection is observed. The main advantage of utilizing neutron beams with respect to X-rays is their deeper penetration into the materials, attaining even up to a few cm in certain cases, e.g. in steels. Defining the strain  $\varepsilon$  as

$$\varepsilon = \frac{(d - d_0)}{d_0} \quad (2)$$

where  $d$  and  $d_0$  are strained and un-strained lattice spacings, respectively,  $\varepsilon$  can be determined by the shift in the position of the Bragg peaks. The broadening of these peaks is determined through the changes their full width at half maximum (FWHM), which is generally related to crystallographic grain size or local variations of strain. Following Eq. (2) there is a need for a 'strain-free' lattice spacing  $d_0$  in order to calculate absolute residual strains. The measurements of this are obtained from the reference specimens described before.. In general, if the assumption is made that  $X, Y, Z$  are the principal directions of deformation (in this case  $Z$  is perpendicular to the layers), then the residual stresses components are given by:

$$\begin{aligned} \sigma_x &= \frac{E}{(1+\nu)(1-2\nu)} [(1-\nu)\varepsilon_x + \nu(\varepsilon_y + \varepsilon_z)] \\ \sigma_y &= \frac{E}{(1+\nu)(1-2\nu)} [(1-\nu)\varepsilon_y + \nu(\varepsilon_x + \varepsilon_z)] \\ \sigma_z &= \frac{E}{(1+\nu)(1-2\nu)} [(1-\nu)\varepsilon_z + \nu(\varepsilon_x + \varepsilon_y)] \end{aligned} \quad (3)$$

where  $E$  is the Young modulus of the investigated material and  $\nu$  the Poisson's ratio. To be precise for the case of diffraction based stress measurements, instead of Young's modulus and Poisson's ratio, lattice plane specific elasticity constants are used. In the case of the investigated sample the normal stress is assumed to be equal to zero and the in-plane strains are assumed to be equal:

$$\varepsilon_{xx} = \varepsilon_{yy} \quad \sigma_{zz} = 0 \quad (4).$$

Tab. 1 reports the parameters utilized to determine the strains and stresses in the multi-layered sample (15). It has to be emphasized again that the strain and stress values, reported in section 3 below, refer to an average taken over 13 single W layers and 12 single Cu layers, respectively; given the high standardization level reached by this joining technique (3), it can safely be assumed that such strain and stress values are well representative of those associated to a single W-Cu-W module.

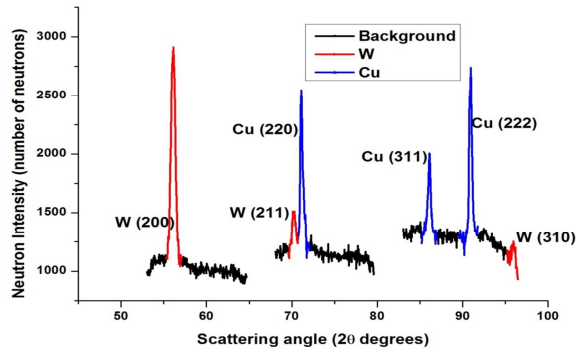
**Table 1** - Elasticity constants used in the stress determinations

| Material     | $hkl$ | Scattering angle<br>2 $\theta^\circ$ | $E_{hkl}$ (GPa)<br>From [15] | $\nu_{hkl}$<br>From [15] |
|--------------|-------|--------------------------------------|------------------------------|--------------------------|
| Tungsten (W) | 110   | 69.68                                | 401                          | 0.28                     |
| Copper (Cu)  | 111   | 75.56                                | 165                          | 0.3                      |

### 3. Results and discussion

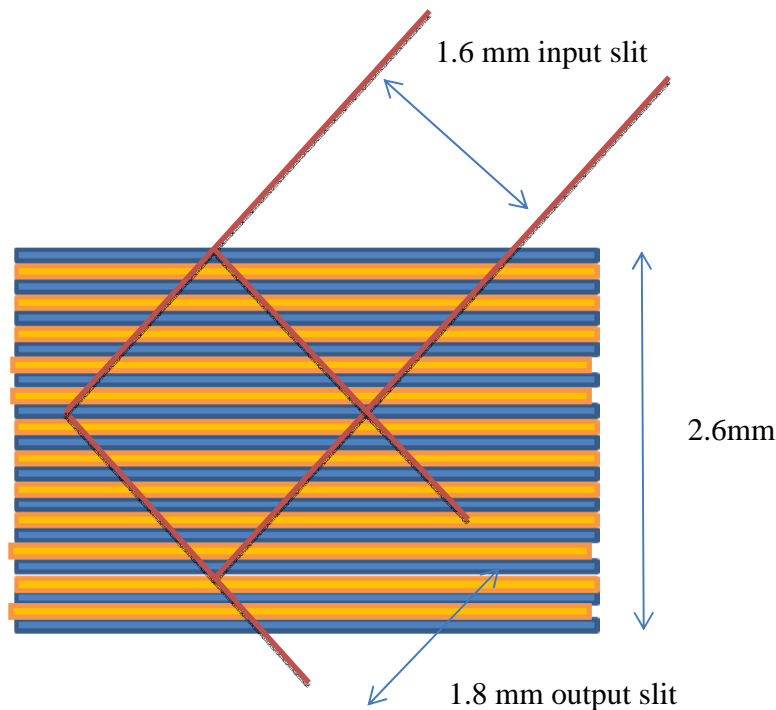
A preliminary neutron diffraction test of the multi-layered sample was carried out at BER-II at Helmholtz-Zentrum Berlin, resulting in the spectra shown in fig. 2; it is clear that the two

different materials W (bcc structure) and Cu (fcc structure) give rise to two different Bragg patterns, with their peak separation sufficient for strain and stress determination in both constituents.



**Fig. 2** - Neutron diffraction pattern measured at HZB, with a wavelength  $\lambda = 1.489 \text{ \AA}$ , for W-Cu prototype multi-layer.

Concerning the main series of neutron diffraction measurements, the neutron diffractometer HB5 at the high Flux Reactor in Petten, NL, has a thermal neutron wavelength of  $2.56 \text{ \AA}$ , derived from a Cu monochromator with  $hkl$  (111) at a take-off angle of about  $76^\circ$ . An input slit of 1.6 mm and an output slit of 1.8 mm were used to define the gauge volume. Figure 3 implies that the such defined nominal gauge volume is totally within the sample, thus avoiding potential surface effects.

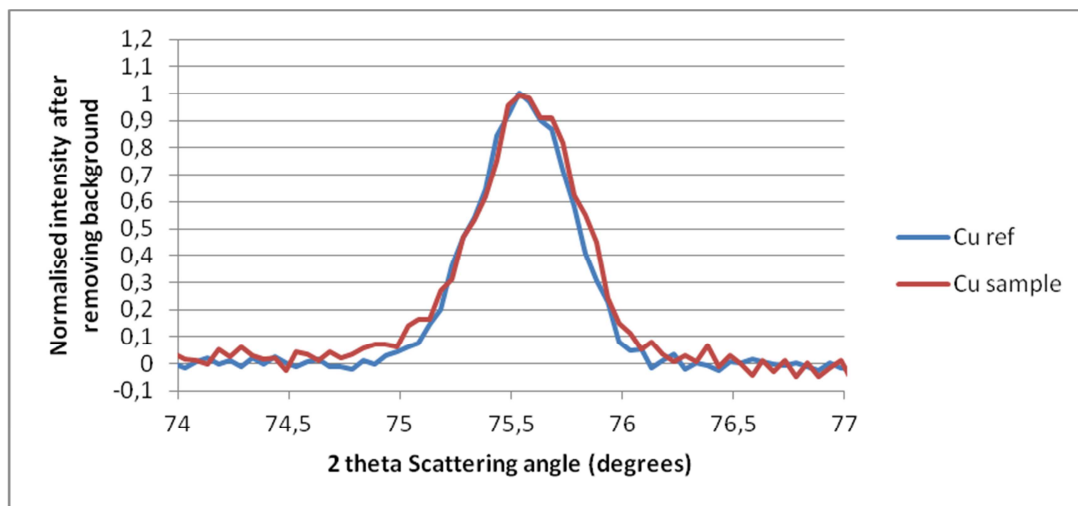


**Fig. 3** – Sketch of the brazed specimen showing the size of the neutron beam gauge volume with respect to the sample dimensions.

A comparison of normalized diffraction lines in the stack of Cu foils for reference measurements and in the brazed multi-layered sample, corresponding to the Cu (111) reflection, is shown in fig. 4: the peak positions are very close to each other, since in this case  $d \sim d_0$  and the stresses are very low.

The measurements in Cu were done with a larger gauge volume height and additionally with oscillation of the specimen, called rocking, in order to obtain better grain size statistics. For W this was not necessary because of the much smaller grains present in the material.

In both constituents, the out of plane strain was not measured. This was because of the strong texture in the W foil emanating from the foil production process, resulting in the situation that the W (110)-crystallographic plane rendered no signal in the through-thickness direction. As already stated, in the subsequent data analysis it was assumed that the through-thickness stress was zero in both W and Cu.

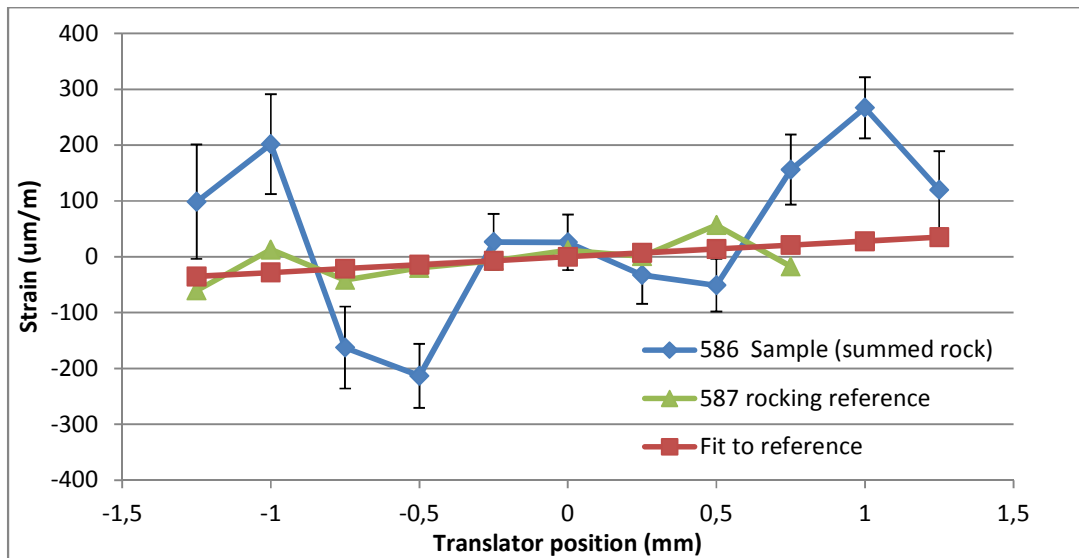


**Fig. 4** - Normalized diffraction peaks for Cu (111) in the brazed sample and in the reference stack of Cu foils.

Concerning the evaluation of stress based on the elastic constants presented in Table 1, the large difference in  $E$  between W and Cu makes a considerable difference on the final stress uncertainty. Typically, for Cu the scattering angle  $2\theta$  is determined to about  $\pm 0.008$  degrees corresponding to about  $\pm 90 \mu\text{m/m}$  in strain and  $\pm 23 \text{ MPa}$  in stress. For W an uncertainty of  $\pm 0.008$  degrees at scattering angle  $69.68^\circ$  corresponds to about  $\pm 100 \mu\text{m/m}$  in strain and  $\pm 59 \text{ MPa}$  in stress.

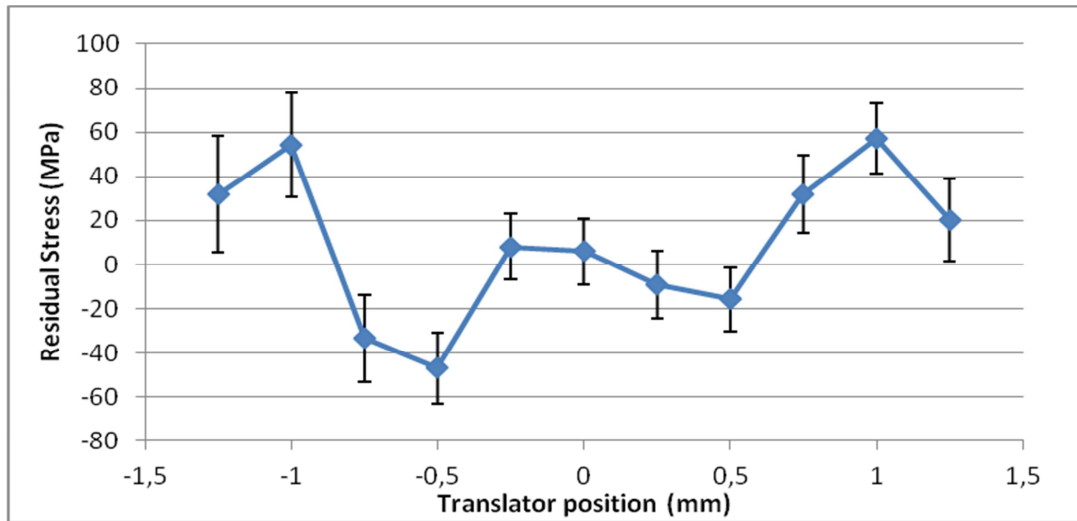
Measurements were not just taken in one point in the centre of the specimen (position 0 mm in figure the following figures) but also at several additional positions through the thickness of the specimen by translating it horizontally up to  $\pm 1.5 \text{ mm}$  in order to establish possible variations in

the strain distribution. Figures 5 and 6 show strains and stresses in Cu. The strain distribution is relatively symmetrical and the reference has a very small gradient which could possibly be attributed to a surface effect, which appears not to be very strong on HB5 at the  $2\theta$  angle used. The strains were calculated from the average value of  $2\theta_0$ , meaning that the strain distribution in the brazed specimen is realistic. The translator positions are different from the actual measurement positions at the edges because the gauge volume is only partially immersed at the edges and therefore the effective centroid of the gauge volume moves away from its geometric centre. Assuming the Cu extends from -1.2 to 1.2 mm, the gauge volume is only 50% immersed at a position of -1.2 and 1.2 mm. The effective measurement location at this translator position would be about 0.4 mm closer to the centre of the specimen. It has to be pointed out that the strains measured in the Cu are relatively small. Typically one measures strain to an accuracy of about  $\pm 100 \mu\text{m/m}$ ; in these measurements the accuracy is generally better. Concerning the stresses (fig. 6) overall the Cu is in slight tension, with low associated uncertainties thanks to the low value of the applicable elasticity modulus. Also in the case of W a strain distribution through the thickness of the specimen is detected (fig. 7). The reference has also a gradient, again very small on HB5 at the  $2\theta$  angle used. The gradient observed is negative in this case.



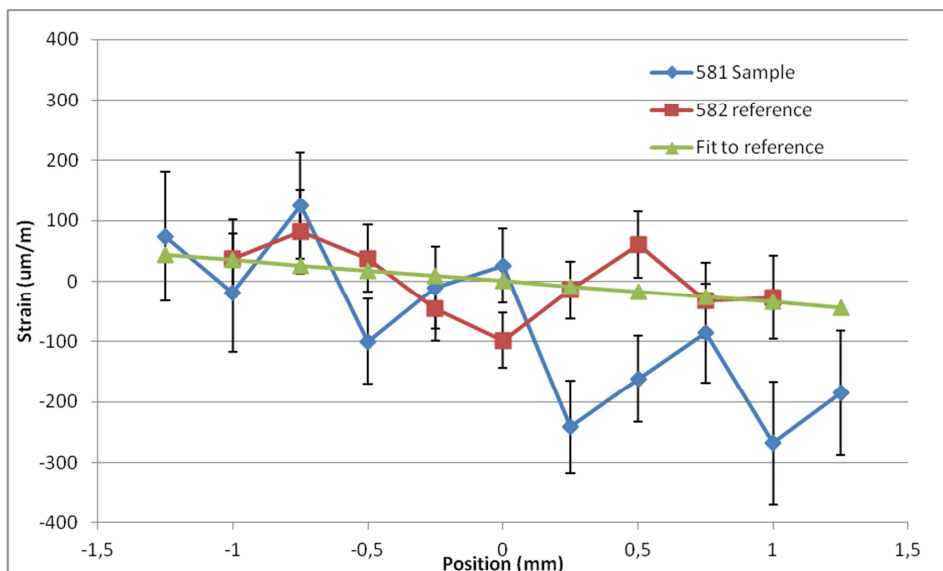
**Fig. 5** - In-plane strains across specimen in Cu (blue line), un-strained reference (green line) and linear fit to reference measurements (red line).



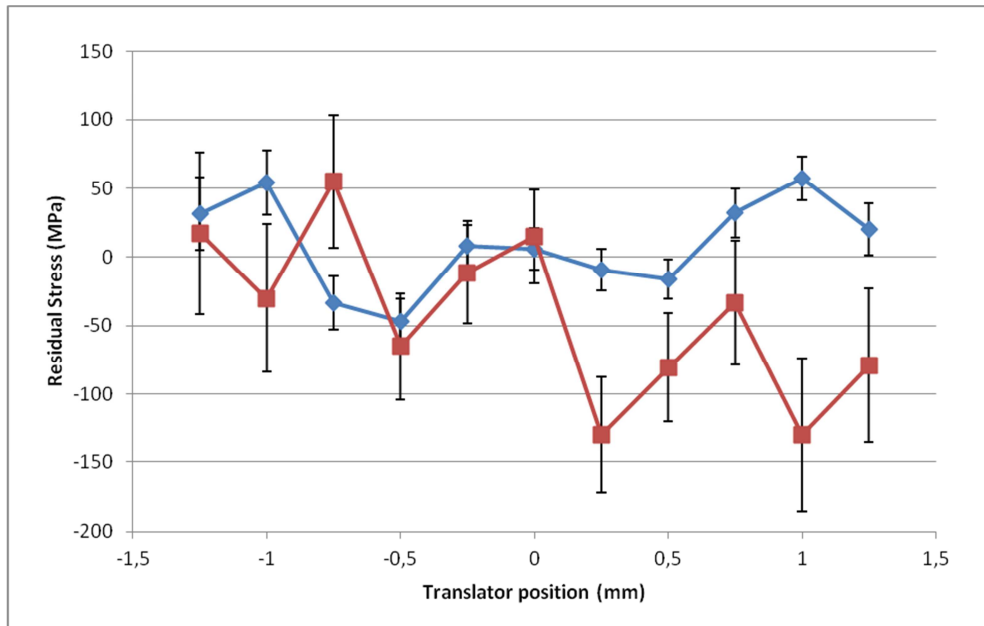


**Fig. 6** - In-plane stress across specimen in Cu, assuming normal stress = 0 MPa.

The strains in W, again calculated from an average value of  $2\theta_b$ , appear not to be as symmetrically distributed as in the Cu layers. Also in this case the accuracy is generally better than  $\pm 100 \mu\text{m/m}$ . Overall the strain in the W is in compression. Having the Cu in tension and the W in compression on the average, albeit with low levels of strain/stress, would be in agreement with expectations as the coefficient of thermal expansion is significantly higher for the Cu than for the W. Figure 8 shows the stresses for both materials (assuming the normal stress is zero MPa). The data quality for the W is slightly inferior to the Cu data.

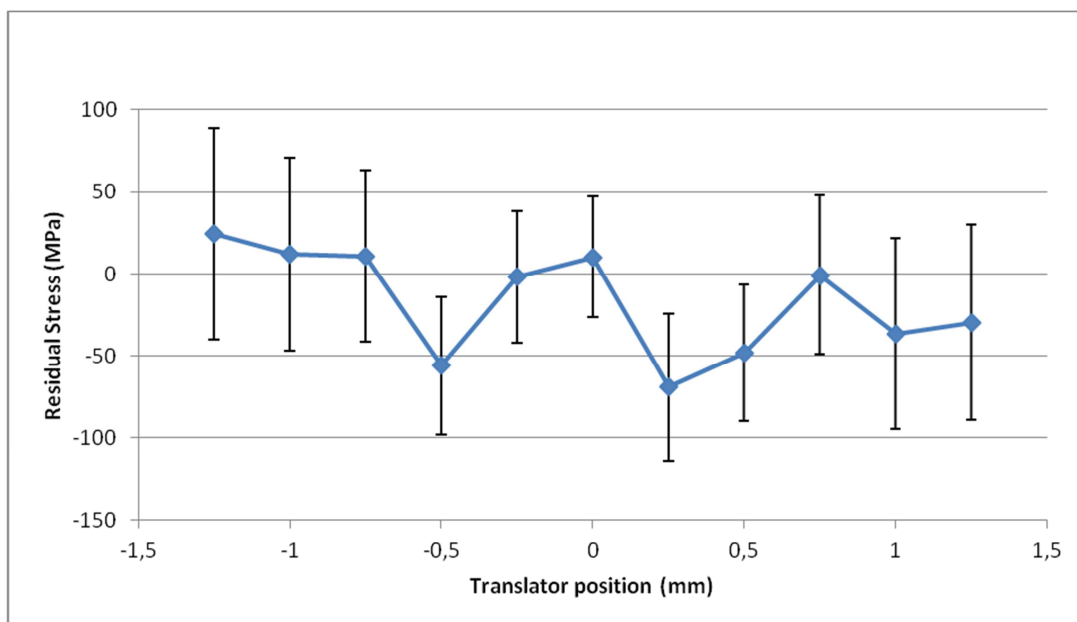


**Fig. 7** - In-plane strain across specimen in W (blue line), un-strained reference (green line) and linear fit to reference measurements (red line),



**Fig. 8** – Comparison of in-plane stresses across specimen in W (red line), Cu (blue line) assuming normal stress is 0 MPa.

Theoretically the average of the stresses should equate to zero and to within the statistical uncertainty this seems to be the case for most measurement positions (Fig. 9): the average over all positions is -17 MPa with a standard deviation of  $\pm 32$  MPa.



**Fig. 9** - Average of Cu and W stresses in the multi-layered specimen.

## 4 Conclusions

A complete strain distribution measurement through a brazed multilayered specimen from 0.1 mm thick W and Cu foils and a determination of the corresponding stresses has been obtained, whereby in-plane stresses were derived based on the assumption of zero stress in the through-thickness direction. The quality of the Cu stress measurement is augmented by the low elasticity modulus and the stress distribution observed was relatively symmetrical. The W stress determination was disadvantaged from the rather large elasticity modulus and larger scatter in the results. However, the average stress across the specimen (averaged over both constituents, W and Cu) is close to zero at  $-17 \pm 32$  MPa, which would be in agreement with expectations.

Therefore, neutron diffraction appears as a well suited experimental tool to non-destructively characterize the strain and stress distribution also in these challenging multi-layered divertor samples. The accuracy of the results could be further improved by the measurement of a single W-Cu-W module, large enough to provide a significant diffracting volume, to more precisely check the correspondence with the average stresses determined in the multi-layer. If the material layers used for such a specimen were thick enough, information about the interface stresses could possibly even be derived. Finally, neutron diffraction can be utilized to investigate much larger samples, including prototype mono-blocks, as well as to check stress evolution with temperature by *in-situ* measurements in suitable furnaces, under vacuum or controlled atmosphere.

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