| ESRF | **Experiment title:** Residual stresses around hydride blisters in Zr-2.5Nb pressure tubes | **Experiment number**: MA287 |
|---|---|---|
| **Beamline**: ID15A | **Date of experiment**: from: 14/07/2007 to: 20/07/2007 | **Date of report**: 15/01/2009 |
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## Report:

We have used energy dispersive synchrotron X-ray diffraction experiments to measure the strain field introduced by a hydride blister grown on a section of a pressure tube from a CANDU nuclear reactor. After charging the tube section with a homogeneous hydrogen concentration of 300 wt ppm, the blister was produced by creating a small cold spot on its surface (~200$^o$C), while the bulk was kept at a temperature of 338$^o$C over a period of 1008 hours. The blister studied here is ellipsoidal in shape, with its long axis along the tube axial direction (Fig. a). The experiments were performed on the wiggler beam line ID15A at the ESRF using a polychromatic beam of high-energy X-rays (60-300keV). A typical spectrum is shown in Fig.1, and the experimental arrangement shown in Fig. 2b

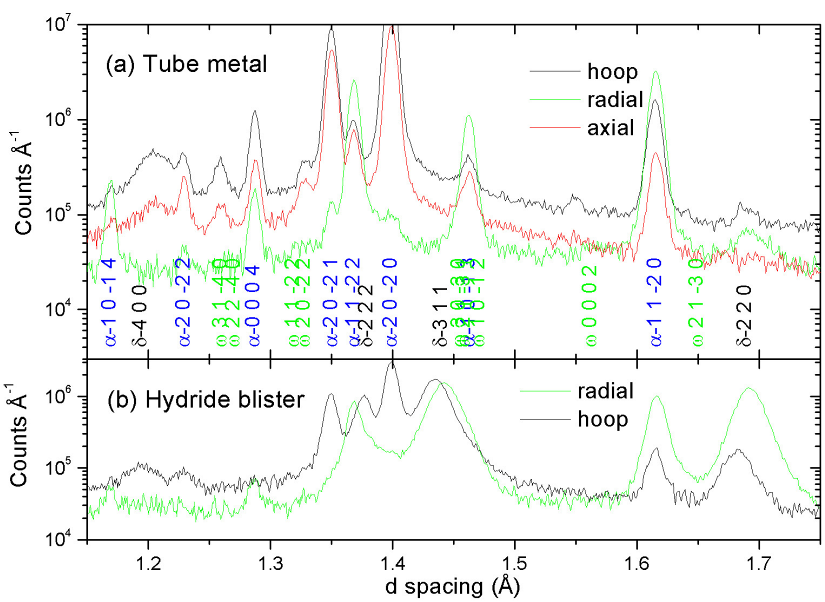


**Figure 1: Diffraction spectra in coupon samples: (a) tube metal and (b) hydride blister**

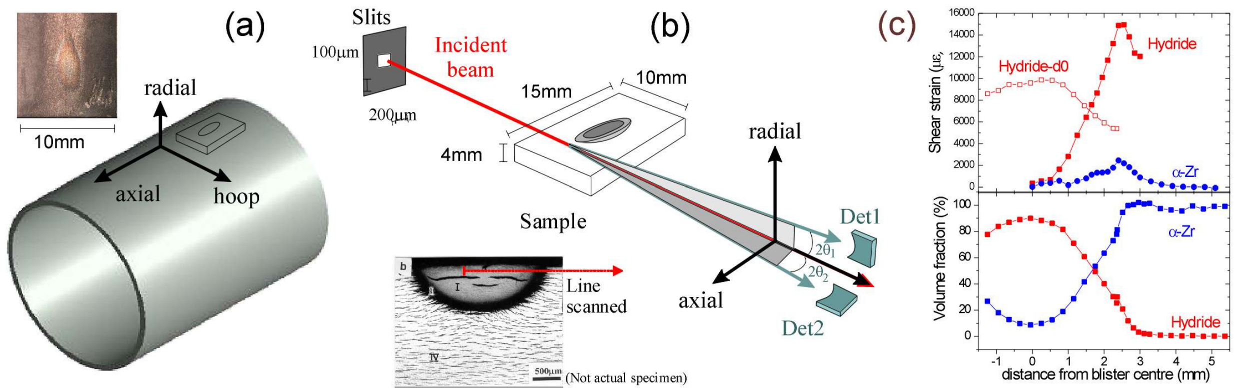


**Figure 2: The set-up for investigating the residual strains and hydride concentration in blisters in Zr-Nb alloys, (a) sample set-up, definition of axis and photo of blister surface, (b) diffraction set-up and macrograph line sampled on preliminary experiments (c) Shear strain in the radial-axial plane and phase volume for the two main phases.**

In this set-up the scattering angle is fixed and the diffracted beam is discriminated on the basis of the photon energy. The high energy was essential to provide bulk penetration through several mm of Zr, and still a lot of the low-energy part (larger d-spacing) is cut-off due to attenuation. However, no alternative techniques exists which provides the penetration and spatial resolution.During the experiments we recorded the diffraction spectra on a 2D mesh surrounding the several blisters, but for this report we present only the analysis performed for the line scan indicated at the bottom of Fig. 2b.

The blister is composed of mainly two crystallographic phases (δ-ZrH and α-Zr), with volume fractions varying with position, as given by the bottom graph in Fig. 2c. very large shear strains were found in the hydride phase both within and around the blister, whilst relatively small strains were seen for α-Zr. Large shear strains were also seen for the hydrides in a small $d_0$-specimen cut from the sample, which appear to be proportional to the hydride phase volume.A sharp drop in Young modulus has been reported for the hydride composition of the blister [1], on the other hand we recently observed a surprisingly large strain in hydrides during tensile loading [2] which may be explained by a phase transformation, and which brought doubts about the predicted stress values. The main uncertainty in these stresses results from the uncertainty in the elastic constants of the hydride phase.

However, in the simple analysis, assuming a fixed Young's modulus, the maximum stresses appear at the blister-matrix interfaces. Near the tube outer surface, we found large compressive stresses of (-450±90) MPa along the blister long axis, and tensile stresses (+320±90) MPa along the tube hoop direction. The stresses outside the blister compared well with finite elements simulations found in the literature. These preliminary results were presented in Ref [3], and we are reworking the analysis in light of the possible transformation.

A proposal will follow to investigate the origin of the large strains observed in "unstressed" solid zirconium hydride and the elastic constants of solid zirconium hydride,

The results show the feasibility of technique, and at the same provided valuable information on the crystallophic nature of the blister and the strain state. This was the first time that such a study had been attempted.

**References**


1 M. P. Puls, S.Q. Shi, and J. Rabier, "Experimental studies of mechanical properties of solid zirconium hydrides," Journal of Nuclear Materials 336, 73-80 (2005).

2 A Steuwer, J. R. Santisteban, M Preuss et al., "Evidence of stress-induced hydrogen ordering in zirconium hydrides," Acta Mat 57 (1), 145-152 (2009).

3 J. R. Santisteban, A. Steuwer, G. Domizzi et al., presented at the International Conference on Residual Stress (2008), 2008 (unpublished).