Experimental report

18/05/2020

Proposal: **INTER-468** **Council:** 4/2019

Title: Calibration protocol measurements ffor brightnESS2 EU project

Research area:

This proposal is a new proposal

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Experimental team: Ranggi sahmura RAMADHAN

Local contacts: Sandra CABEZA

Samples: Aluminium
Steel

Instrument	Requested days	Allocated days	From	To
SALSA	6	9	31/08/2019	06/09/2019
			17/01/2020	20/01/2020

Abstract:

Calibration protocol measurements for BrightnESS^2 EU project

Experiment: INTER-468
Experimental Team: Sandra CABEZA, Thilo PIRLING, Ranggi Sahmura RAMADHAN

Background. Strain scanning by neutron diffraction is one of the most versatile and powerful analysis tools at the disposal of industries, and it has proven to be beneficial in the research and development of products in vital sectors (aerospace, energy, manufacturing, etc). Technical development and standardisation of neutron diffraction for residual stress measurement have been performed in the past [1] and the ISO 21432:2019(en) [2] serve as the "best practice" guideline for residual stress measurement with neutron diffraction. Despite these efforts, however, neutron diffraction has not yet become a common characterization tool within industry. One of the main factors are the perceived disparity of 'measurement quality' between experiments and/or neutron strain scanning instruments, originates from the lack of reporting of positional error (i.e., uncertainties between the intended and effective measurement position) from instrumental setup and the lack of benchmarking of this parameter between the instruments. Therefore, to advance from the previous standardisation initiatives and to contribute towards the integration of neutron diffraction strain scanning as routine characterisation tool within industry, this work approached new quantitative analyses of specific instrument and sample geometries. To achieve this, calibration measurements using specially-design sample were carried out on SALSA in this experiment.

Methods. The instrument setup used primary and secondary collimator, with GV of 0.6×2×0.6 mm^3. A set of calibration sample was tested, comprising: 1) Calibration Foil & pin sample; 2) 5-wall sample, and; 3) Tube sample, Figure 1. **Pin scan** using the Foil & pin sample, was performed to investigate the relationship between detector angular position ($2\theta_{det}$) with the GV position. The pin was scanned towards the incident beam at different detector angular position: $2\theta_{det}$ = 93.5º and 49.8º, measuring reflection Fe(211) and Fe (110), Figure 2. The intensity curves were fitted by Gaussian to determine the position of the GV for each detector position. **Foil scans** were performed to determine the position of reference point with respect to the centre of ω-rotation. The scans were made to the top and bottom foil, with the detector angle at $2\theta_{det}$ = 93.5º and ω = 46.75 º, -43.25º, and -133.25º. Neutron counts at a narrow wavelength range around the Bragg peak position were integrated and used to plot the intensity curve of each scan. By comparing the alignment position and the measured position of the foil, the offset can be determined. Using graphical analysis, the centre of ω-rotation position relative to the reference point can be determined. **Wall scan,** Figure 3, on the 5-wall and the Tube sample were used to 1) determine the precision of the sample alignment system: positions of the wall surface measured by neutron were compared against the alignment positions and the standard deviation of the offsets was then used to determine the precision; 2) determine the accuracy of the entry curve analysis software: the resulting entry curve from the scans were fitted using the software, and the results were compared against coordinate measurement machine (CMM).

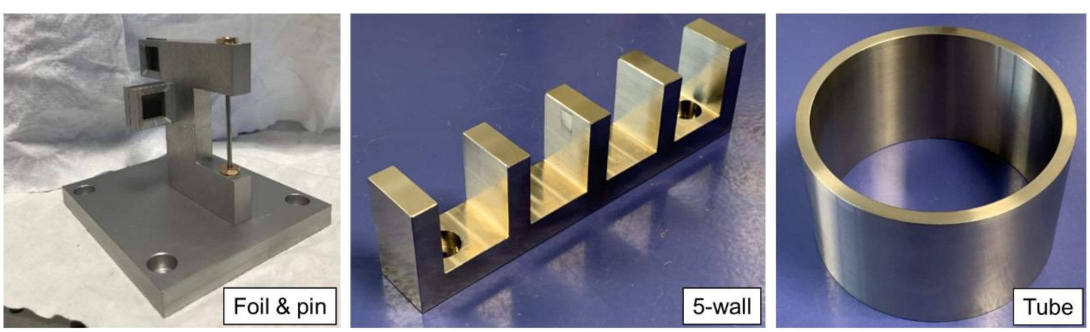

Figure 1 Calibration samples with specific geometry: Foil & pin, 5-wall, and Tube sample.

Results The pin scan results on SALSA shows that the change of detector angular position of 44° (from $2\theta_{det}$ = 93.5º to 49.5º) ***moved the GV position ~100 μm***. This GV displacement needs to be considered for sample characterisation for that requires measurements at multiple detector positions. The possible causes of this effect might be the misalignment between the centre of $2\theta_{det}$-rotation and ω-rotation and/or other issues related to mechanical components which drive and guide the movement of the diffracted optics/ detector support.

Figure 4 shows the reference point position with respect to the centre of ω-rotation on SALSA. It can be observed that the reference point was misaligned from the centre of ω-rotation for ~310 μm. This large misalignment can be attributed to the defects on the calibration sample, i.e., misalignment between the fiducial sphere and the main pin in the SALSA calibration sample. The determination of SALSA reference point was carried out by tracking the fiducial sphere using the camera and the image recognition software. The beam apertures were aligned to the vertical pin, which is supposed to be precisely on the same position below the sphere. However, it was later observed that there was a ~200 μm misalignment between the sphere and the pin due to manufacturing defects of the inserts of the pin. This misalignment is within the order of the reference point to the centre of ω-rotation displacement as measured on SALSA.

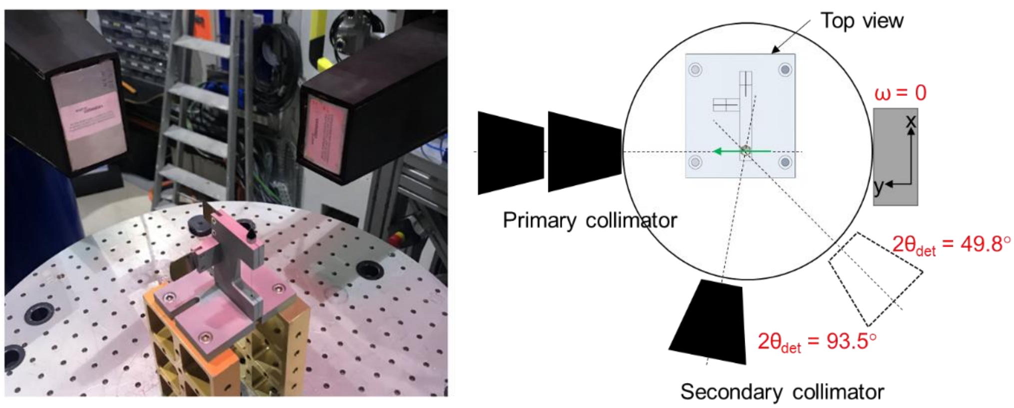

Figure 2 Pin scan exercise on SALSA

From wall scan exercise, the offset between alignment position and measured position of the wall is used to determine the precision of the sample alignment system. The standard deviation of the offsets represents the ability of the sample alignment system in repeatedly arriving to the same position, in this case simulated by the surface of the walls, thus the precision of the alignment system. It was observed that that the measurements with higher counting times (longer measurements) has the lowest standard deviation, thus ***highest precision at 50 μm***. The measurements with shorter counting times yields lower precision at 110 μm for transmission geometry and 140 μm for reflection geometry. The lower precision, however, are due to the lower statistical quality of the data and thus higher fitting uncertainties. Therefore, the results from higher counting times measurement reflects the precision of the sample alignment system better.

The accuracy of the entry curve analysis software was determined from the average difference of the features dimensions measured by wall scan and by CMM. For the MathCad-based code used on SALSA, it can be observed that the differences of the wall scan and the CMM result for ***flat surfaces perpendicular to the beam propagation plane*** have an average of ***~40 μm***. For the ***curved surface at the radial line***, the differences was **~60 μm.** These accuracies were around or better than 10% of the GV width, which is the generally accepted accuracy.

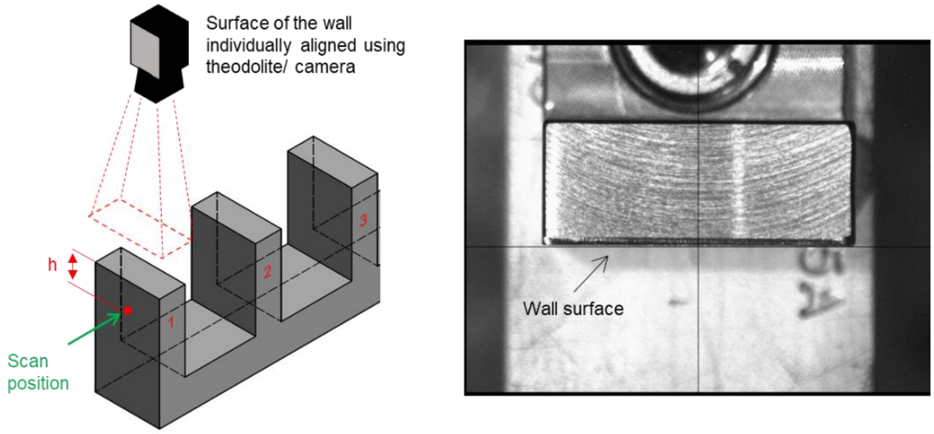

Figure 3 Wall scan exercise using 5-wall sample on SALSA. Field-of-view of the telecentric camera is shown

Conclusion. The results indicated that SALSA can achieve positioning uncertainties around or better than 10% of the GV width, which is the generally accepted criterion for residual stress measurement using neutron diffraction. The calibration methods were established, while some upgrades might be required for the calibration samples. Further studies is required to gain more information regarding the instrument setup (e.g., evaluation of GV position as a function of re-adjusted detector positions) and analysis software (implementation of more geometric models for the entry curve analysis software used for sample alignment).

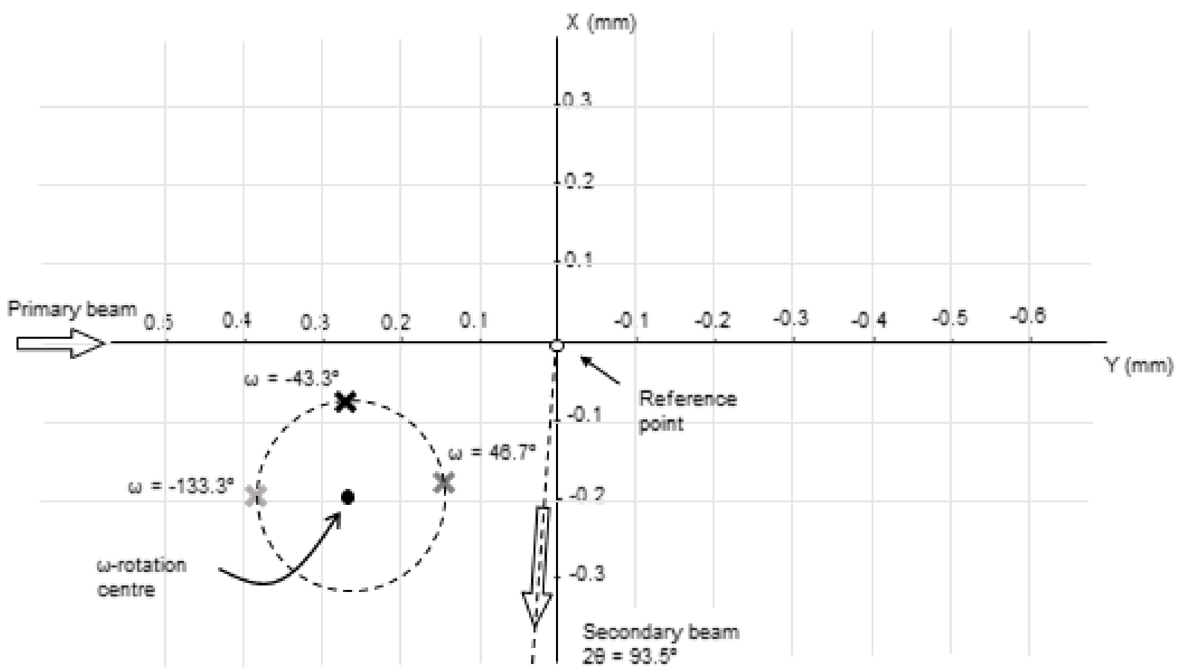

Figure 4 Reference point relative to centre of ω-rotation on SALSA

Reference

[1] G. A.Webster. Neutron diffraction measurements of residual stress in a shrink-fit ring and plug. Technical Report VAMAS Report no. 38, London, 2000.

[2] Non-destructive testing—Standard test method for determining residual stresses by neutron diffraction. Standard, International Organization for Standardization, December 2019.