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The European Network on Neutron Techniques Standardization for Structural Integrity

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@ Technical University of Munich

Workshop on the Assessment of Residual Stresses in Welds

Helmholtz Zentrum Berlin

UNCERTAINTIES DUE TO GRAIN SIZE ISSUES IN RESIDUAL STRESS DETERMINATIONS USING NEUTRON DIFFRACTION

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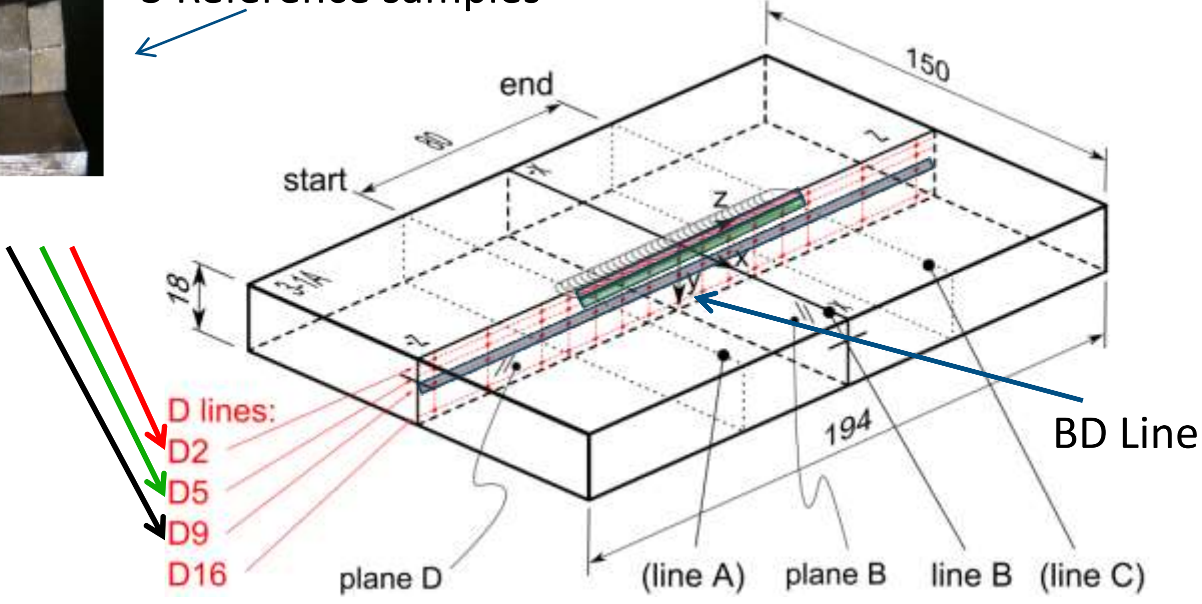
Workshop on the Assessment of Residual Stresses in Welds

With thanks to: René V. Martins, Michael Hofmann, Joana Rebelo Kornmeier, Shanmukha Moturu, Mirko Boin, Anastasius Youtsos and Carsten Ohms

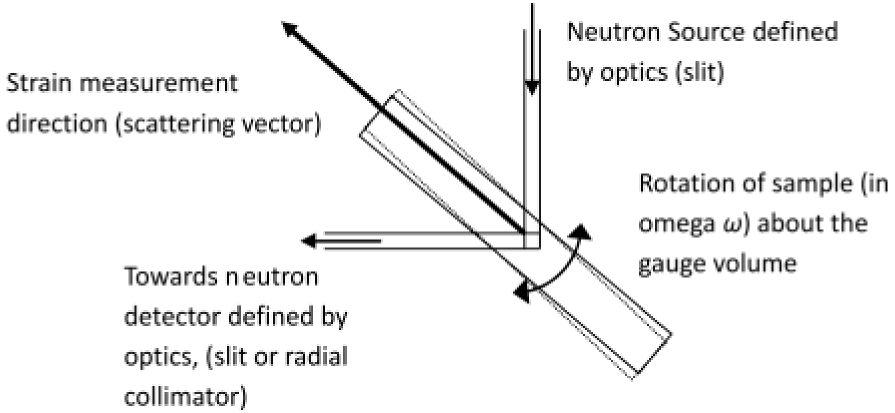
TG4: The perfect specimen to study

Main sample

3 Reference samples



Picture of the TG4 'main sample' 3-1A in-situ in the residual stress diffractometer E3 at the HZB, Berlin with an input slit optic of 3 x 3 mm^2.



Grain size varies along BD line
Oscillation (stepwise or continuous) can reduce the 'grain-size effect'

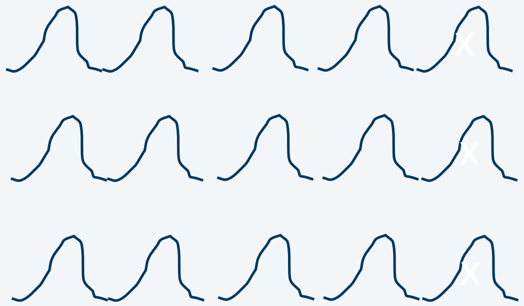
This is what we need $\rightarrow$ $u(2\theta) \approx \left(u(2\theta_{fitting})^2 + u(2\theta_{grain})^2\right)^{1/2}$ $\rightarrow$ $u(\varepsilon) \rightarrow u(\sigma)$

Multiple measurement

For one stress determination (e.g 5 measurements/more)

Peaks of 1 unit time (for example)
Sample different sets of grains
(oscillations would loose information)

X
Y
Z



Time consuming with the main sample but quick with a reference sample

Information available:

$u(2\theta)$ $\approx$ standard deviation of the 2θ fits values.

$u(2\theta_{fitting})$ and $u(2\theta_{grain})$

OSC = 0

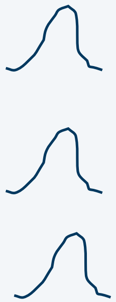
Estimation possible from quick reference measurements

Single shot

For one stress determination

Peaks of 1 unit time (for example)
oscillations or not

X
Y
Z



Measurement
5 times quicker

Information available:

$u(2\theta_{fitting})$

OSC ≥ 0

Information **not** available:

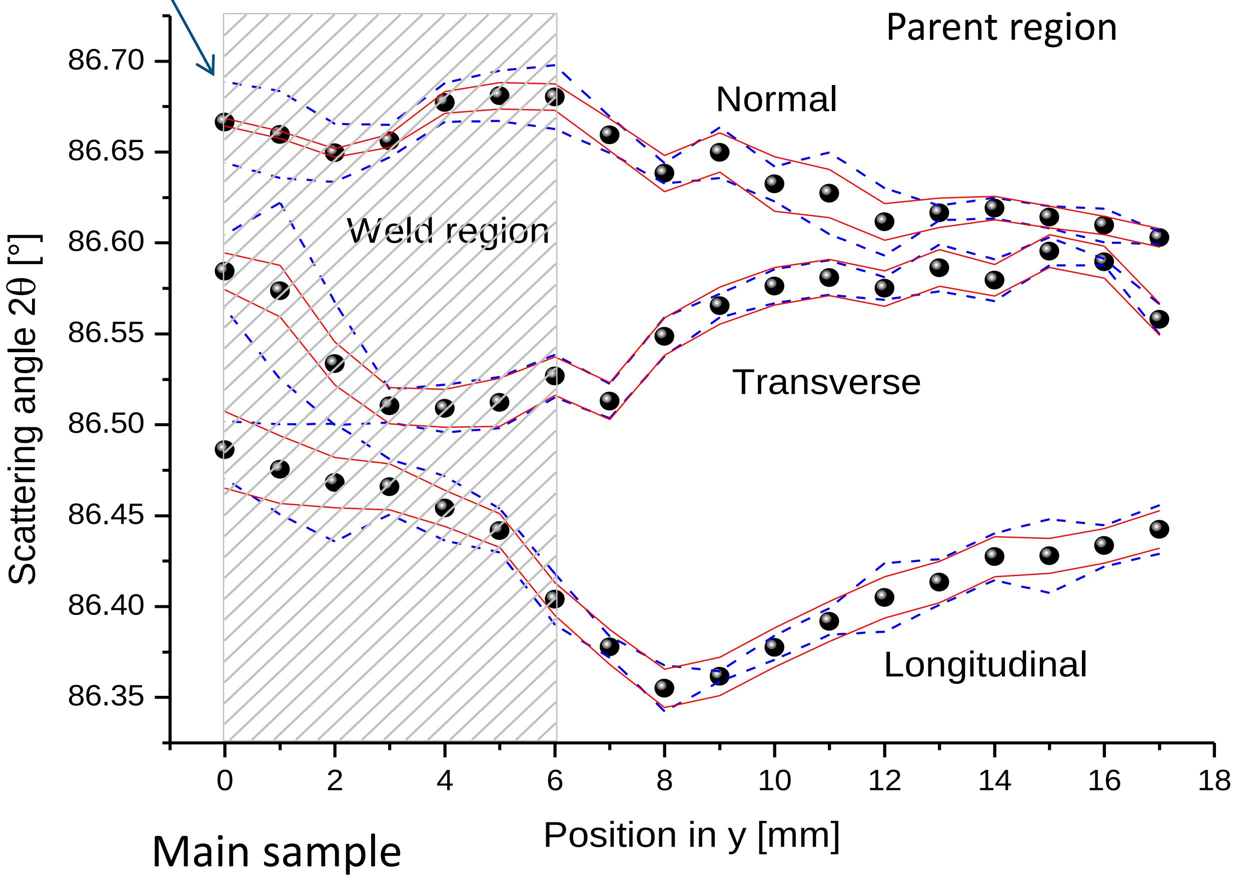
$u(2\theta_{grain})$ and $u(2\theta)$

'Modelling'

Multiple measurement

Very short neutron path length

$$u(2\theta) \approx (u(2\theta_{fitting})^2 + u(2\theta_{grain})^2)^{1/2}$$



Main sample

e.g. by **measuring the specimen several times** at slightly different rotation angles, -2°, -1°, 0°, +1°, +2° relative to the correct bisecting angle ω and comparing the average fit uncertainty values with the standard deviation of the 2θ values.

The 2θ scattering angle along the line of measurements in the 3-pass slot weld in the three orthogonal directions, weld longitudinal, weld transverse and plate normal. The average 2θ values are shown here in black dots.

The fitting uncertainty decreases with time, whereas the uncertainty due to grain size 'is fixed'.

The contrast is seen most clearly in the normal direction near both surfaces (y = 0 mm and 18 mm), where the short path length of the neutrons gives rise to a very strong diffraction signal in a short period of time. In the parent material (approximately starting from y = 6 mm to 18 mm) the red and blue lines are approximately the same, suggesting that using only the fitting uncertainty is adequate in this region.

However this cannot be said of the weld region where the red and blue lines diverge, indicating a $u(2\theta_{grain})$ contribution.

Multiple and 'single shot' measurement

Relative rotation (ω) [°]	Top of weld region: Transverse y=2mm 2θ [°]	Uncertainty of fit $u(2\theta_{fitting})$ [°]	Parent region: Longitudinal y=13mm 2θ [°]	Uncertainty of fit $u(2\theta_{fitting})$ [°]
+2	86.516	0.009	86.413	0.011
+1	86.520	0.010	86.406	0.011
0	86.495	0.015	86.419	0.012
-1	86.577	0.015	86.398	0.011
-2	86.560	0.010	86.431	0.012
	Standard deviation $u(2\theta) = 0.034$ $u(2\theta_{grain}) = 0.032$	Average fitting uncertainty $u(2\theta_{fitting}) = 0.012$	Standard deviation $u(2\theta) = 0.013$ $u(2\theta_{grain}) = 0.005$	Average fitting uncertainty $u(2\theta_{fitting}) = 0.011$

$$u(2\theta) \approx \left(u(2\theta_{fitting})^2 + u(2\theta_{grain})^2\right)^{1/2}$$

$$u(2\theta_{fitting}) \propto \frac{1}{\sqrt{(time)}} \quad u(2\theta_{grain}) \propto \frac{1}{\sqrt{(N_{DG})}} \Rightarrow u(2\theta) \propto \frac{1}{\sqrt{(Number\ of\ measurements*)}}$$

*Assuming the time of each measurement is the same and different grains are sampled and detected in each measurement ⇒ All uncertainties can be divided by $\sqrt{5}$ in this case

Multiple and 'single shot' measurement

$u(2\theta_{fitting}) \propto \frac{1}{\sqrt{(time)}}$ $u(2\theta_{grain}) \propto \frac{1}{\sqrt{(N_{DG})}}$ ➡ $u(2\theta) \propto \frac{1}{\sqrt{(Number\ of\ measurements*)}}$

$\sqrt{5}$

Top of weld region

Transverse
y= 2mm 2θ [°]

$u(2\theta) \approx (u(2\theta_{fitting})^2 + u(2\theta_{grain})^2)^{1/2}$

$0.034 \approx (0.012^2 + 0.032^2)^{1/2}$

Single shot Addition of 5 measurements of equal time and **sampling the same grains** (no oscillation or oscillation over the same grains)

$0.032 \approx (0.005^2 + 0.032^2)^{1/2}$

Multiple Addition of 5 Multiple measurements of equal time: **sampling different grains** (e.g. Stepwise oscillation)

$0.015 \approx (0.005^2 + 0.014^2)^{1/2}$

Parent region

Longitudinal
y=13mm 2θ [°]

$u(2\theta) \approx (u(2\theta_{fitting})^2 + u(2\theta_{grain})^2)^{1/2}$

$0.013 \approx (0.011^2 + 0.005^2)^{1/2}$

Single shot Addition of 5 measurements of equal time and **sampling the same grains** (no oscillation or oscillation over the same grains)

$0.007 \approx (0.005^2 + 0.005^2)^{1/2}$

Multiple Addition of 5 Multiple measurements of equal time: **sampling different grains** (e.g. Stepwise oscillation)

$0.005 \approx (0.005^2 + 0.002^2)^{1/2}$

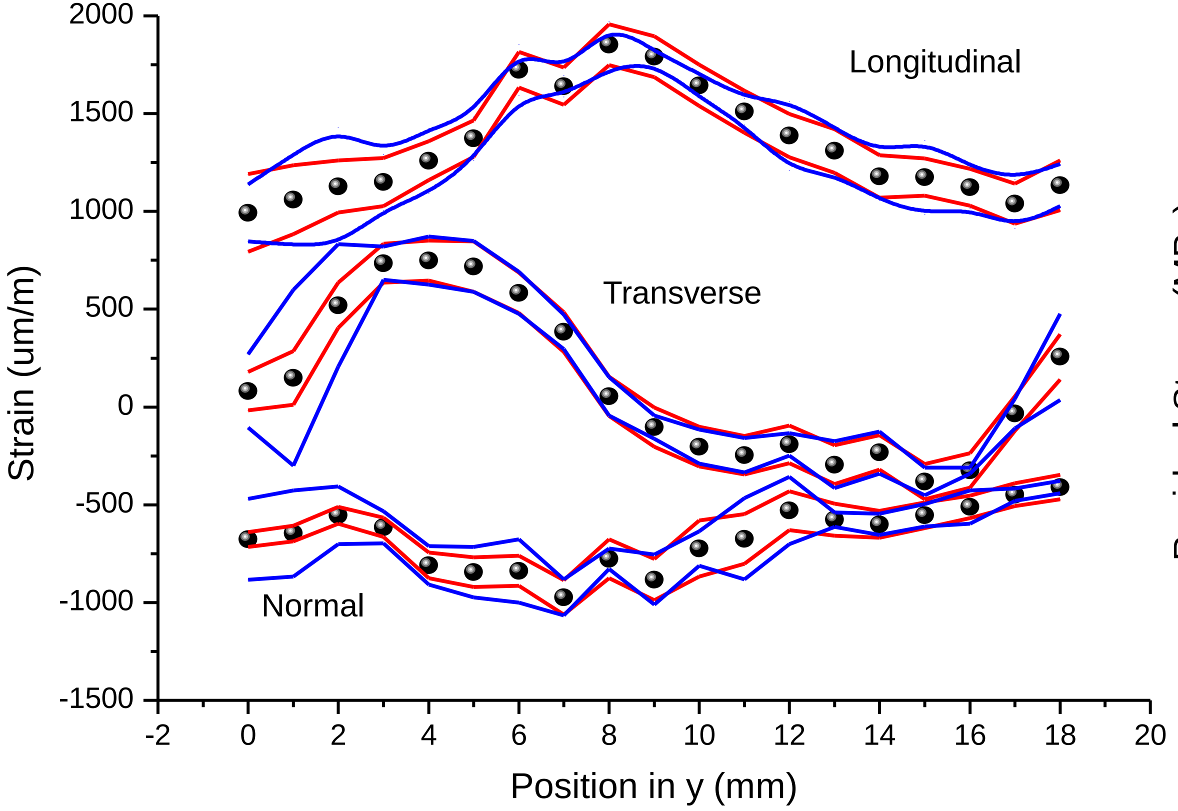
Propagation of the uncertainty in 2 theta

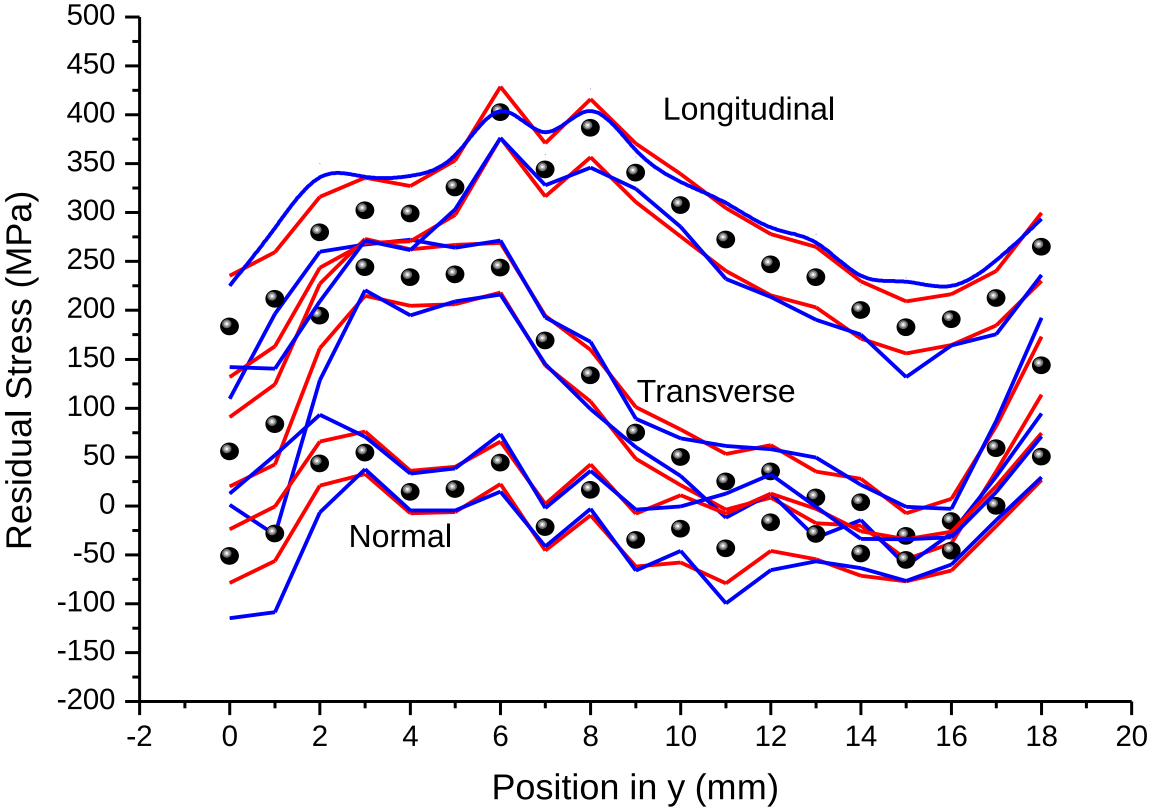
$u(2\theta) \longrightarrow u(\varepsilon) \longrightarrow u(\sigma)$

Measured BD line **5 times.** Instead of oscillating specimen, made 5 scans in -2, -1, 0 , 1, 2 degrees offset in omega. **For each data set we are effectively looking at different sets of grains**. Looked at results individually. Looked at summed results.

$$u(2\theta) \approx (u(2\theta_{fitting})^2 + u(2\theta_{grain})^2)^{1/2}$$

The uncertainty values shown represent the values we should get if we had only measured once. Because we have **measured 5 times**, the uncertainties will actually be **SQRT(5) times less**.





Multiple measurement and 'single shot' measurement

Need a more realistic value →

$$u(\sigma_{xx}) \cong \left[\left(\frac{E_{hkl}(1-\nu_{hkl})}{(1+\nu_{hkl})(1-2\nu_{hkl})}u(\varepsilon_{xx})\right)^2 + \left(\frac{\nu_{hkl}E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\right)^2\left(u(\varepsilon_{yy})^2 + u(\varepsilon_{zz})^2\right)\right]^{\frac{1}{2}}$$

$$u(\varepsilon) = \frac{1}{tan\theta_0}\left[u(\theta_{fitting})^2 + u(\theta_{grain})^2 + u(\theta_{0-fitting})^2 + u(\theta_{0-grain})^2\right]^{\frac{1}{2}}$$

$$u(2\theta) \approx (u(2\theta_{fitting})^2 + u(2\theta_{grain})^2)^{1/2}$$

$$u(2\theta_0) \approx (u(2\theta_{0-fitting})^2 + u(2\theta_{0-grain})^2)^{1/2}$$

Multiple measurement: time consuming

Multiple measurement: quick

'Main Sample'

'Reference sample'

$u(2\theta_{fitting})$ → Check if this is accurate in the first place.

$u(2\theta_{grain})$ → See if we can 'model' this from material properties and instrumental parameters for single shot measurements.

Counting statistics versus grain size statistics

Equation from:
Withers, P. J.,
Daymond, M. R. &
Johnson, M. W. (2001).
J. Appl. Crystallogr. **34**,
737-743.

$$u(2\theta) \approx \left(u(2\theta_{fitting})^2 + u(2\theta_{grain})^2\right)^{1/2}$$

'Wimpory model'

$$u(2\theta_{fitting})^2 \approx \left(\frac{SD_{Gauss}{}^2}{I}\right)\left[1 + 2\left(2^{\frac{1}{2}}\right)\frac{B}{H}\right]$$

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

Time Dependent $u(2\theta_{fitting}) \propto \frac{1}{\sqrt{(time)}}$

Not Time Dependent $u(2\theta_{grain}) \propto \frac{1}{\sqrt{(N_{DG})}}$

Parameter	Description
SD_{Gauss}	$FWHM = 2(2ln2)^{1/2} * SD_{Gauss}$
I	Integrated intensity (area under Gaussian or Voigt divided by Bin Size)
B	Background value at centre of peak
H	Height of Gaussian or Voigt Peak
N_{DG}	Number of detected diffracting grains

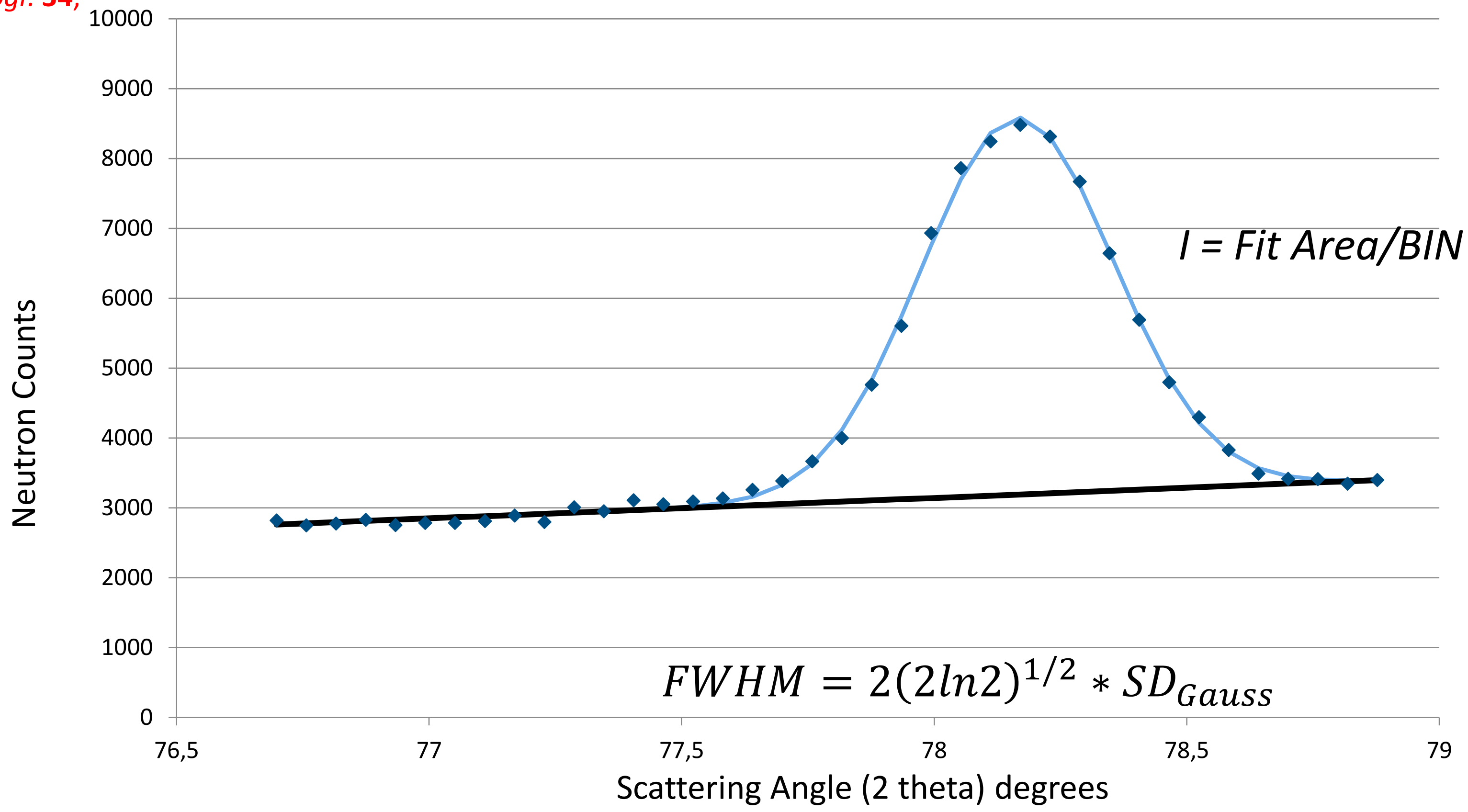
Is the returned fitting uncertainty correct?

2th(°)	2th err (°)	Fit Area	FWHM (°)	SDGauss (°)	H	B	BIN (°)	I
78.1650	0.0018	2583	0.450	0.191	5394	3190	0.0589	43854

Equation from: Withers, P. J., Daymond, M. R. & Johnson, M. W. (2001). *J. Appl. Crystallogr.* **34**, 737-743.

$$u(2\theta_{fitting})^2 \approx \left(\frac{SD_{Gauss}{}^2}{I}\right)\left[1 + 2\left(2^{\frac{1}{2}}\right)\frac{B}{H}\right]$$

$u(2\theta_{fitting})$ = 0.0015°



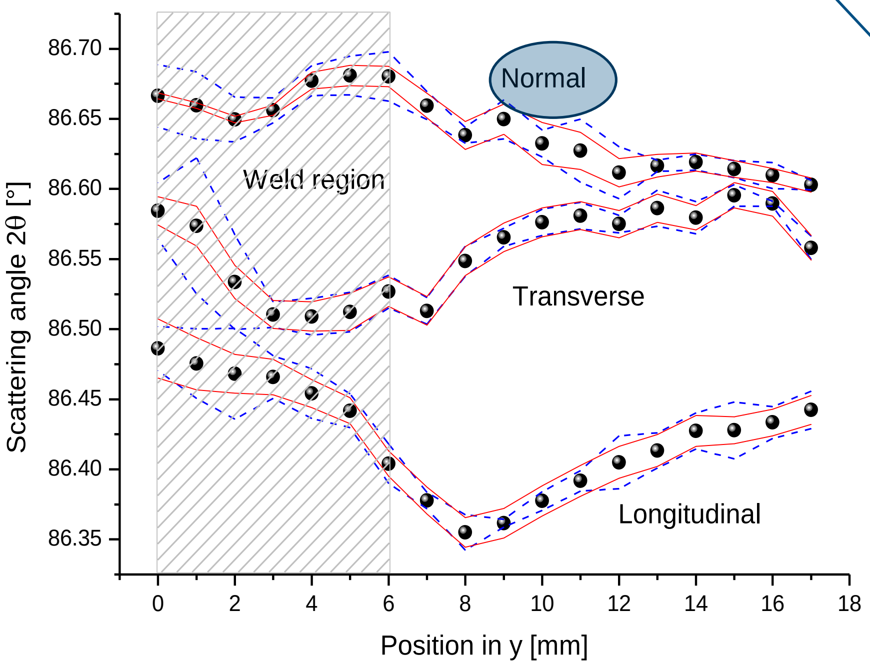
Geometrical area
Assuming a triangle
*=H*0.5*
*=5394*0.5*
=2697

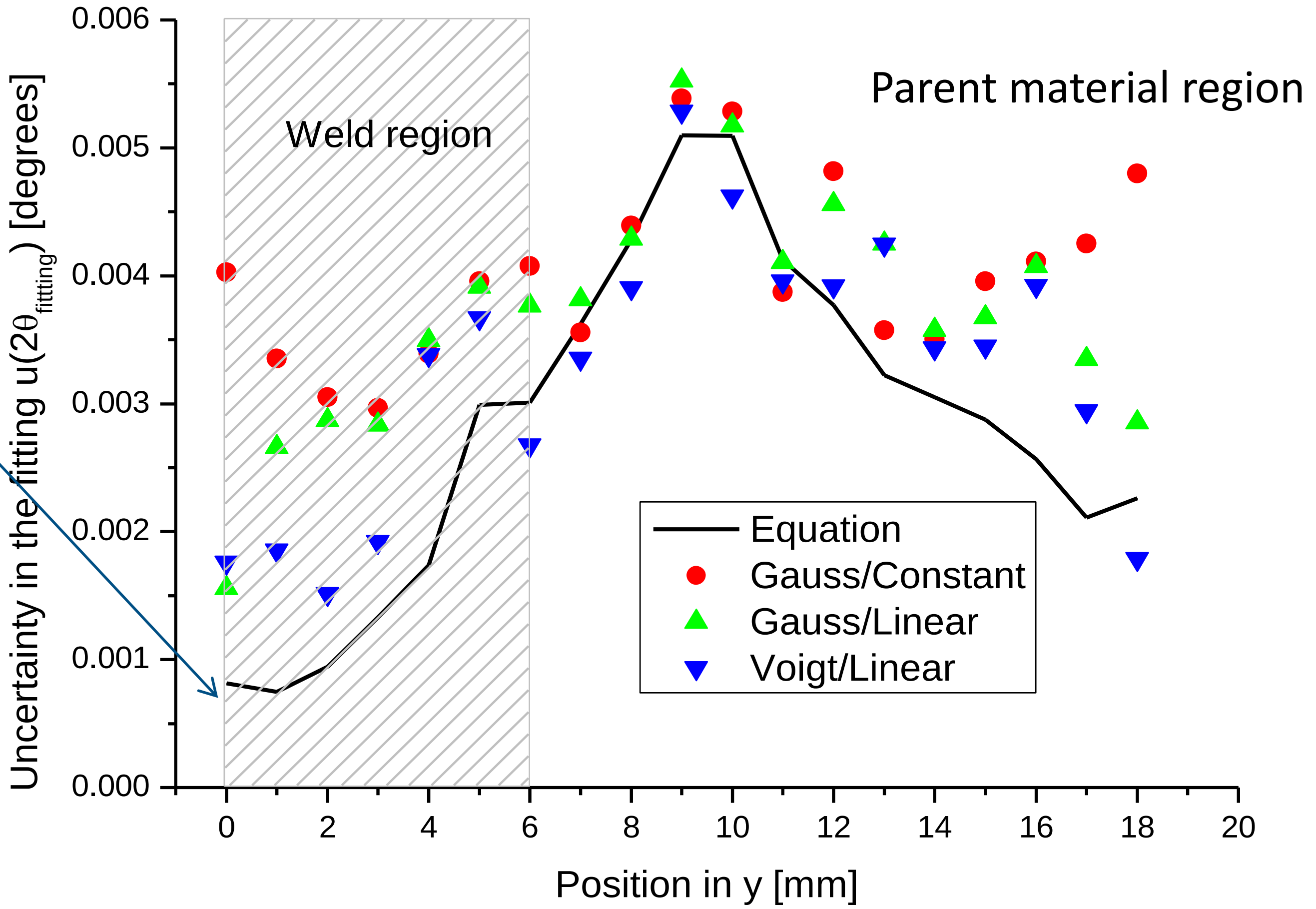
Gaussian fit area = 2583

Number of neutrons
(Integrated Intensity)
I = Fit Area/BIN
= 2583/0.0589 = 43854

Checking the fit uncertainty with the equation

$$(2\theta_{fitting})^2 \approx \left(\frac{SD_{Gauss}{}^{2}}{I}\right)\left[1+2\left(2^{\frac{1}{2}}\right)\frac{B}{H}\right]$$





The returned fitting uncertainty values from the fit program are closer to the equation values in this order:

Gaussian/Constant background =worst, Gaussian/Linear background = better, Voigt/Linear background =best

'Wimpory Model'

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

T. Gnaeupel-Herold, H. J. Prask, R. J. Fields, T. J. Foecke, Z. C. Xia, U. Lienert, A synchrotron study of residual stresses in a Al6022 deep drawn cup, Mater. Sci. Eng., A 366 (2004) 104–113.

$$P \approx \frac{(D_H + \eta_M) * (OSC + \omega_M)}{4\pi} * m^{hkl}$$

From multiple measurement

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right)$$

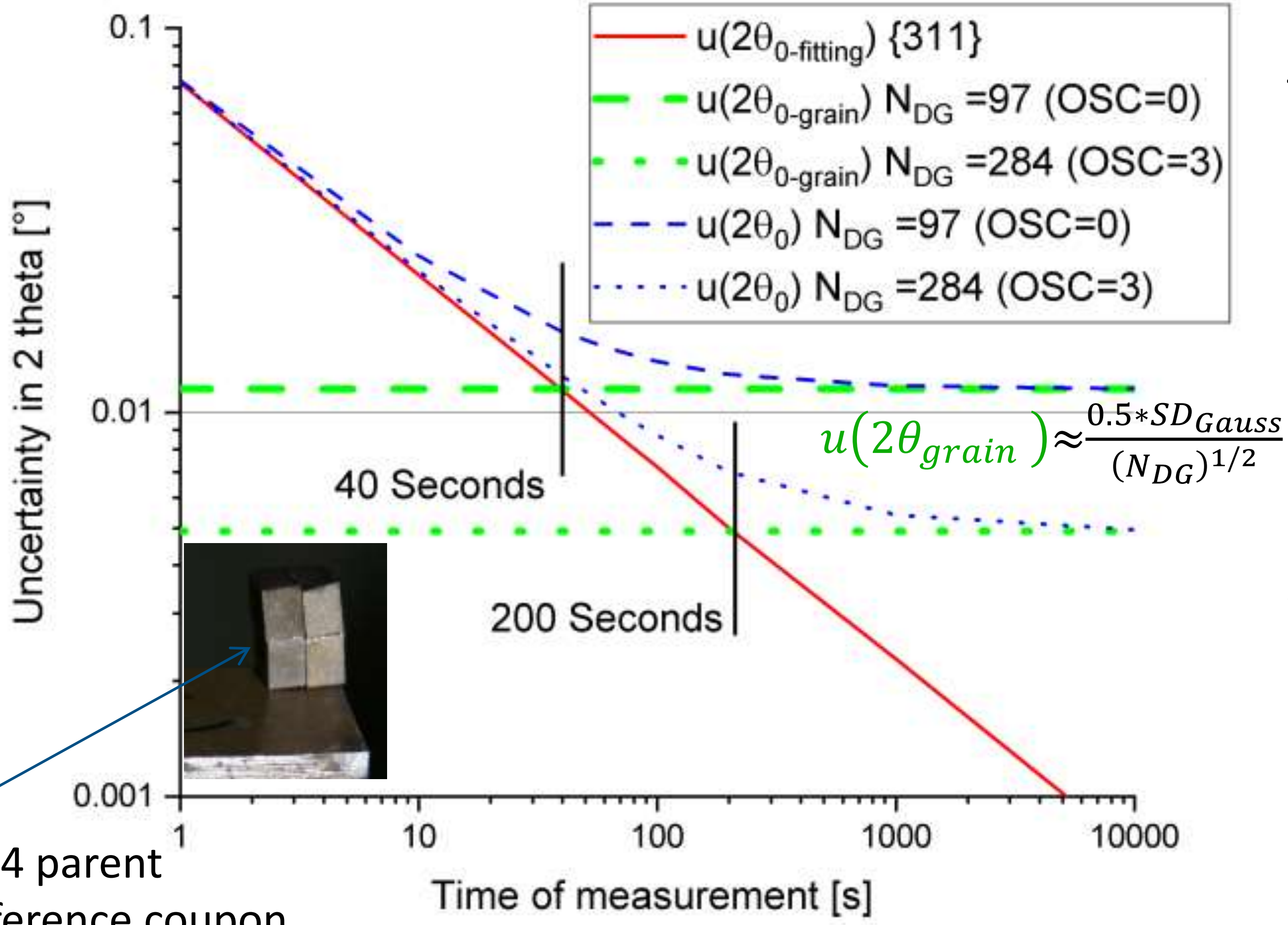
From instrument and material parameters (single shot)

Symbol	Parameter	How to increase N_{DG}
gv	Gauge Volume	Increase
S_G	Grain size	Decrease
D_H	Angular detector height	Increase
η_M	Grain mosaicity in azimuthal direction, i.e. along the diffraction ring	Increase
OSC	The total angular oscillation of the sample around the ω-axis	Increase
ω_M	Grain mosaicity around the ω-axis	Increase
m^{hkl}	multiplicity of the particular Bragg reflection	Increase

Radians { (D_H, η_M, OSC, ω_M)

Uncertainty versus Time and N_{DG}

$$u(2\theta_0) \approx \left(\left(u(2\theta_{0-fitting})\right)^2 + u(2\theta_{0-grain})^2 \right)^{1/2}$$



TG4 parent reference coupon on E3

This shows the fitting uncertainty as a function of time for the {3 1 1} reflection for the 'black' TG4 parent reference coupon on E3.

The grain size uncertainty for OSC=0 and OSC=3 are also shown.

For this particular experimental set-up and specimen, it takes 100 seconds to get to a fitting uncertainty of about ±0.01°.

If one does not oscillate (OSC=0) the total uncertainty cannot get any lower as one has a value of N_{DG} ≈97 which places an upper bound of the value of $u(2\theta_{0\text{-grain}}) \approx \pm 0.0115°$.

It takes 1000 seconds to get to a fitting uncertainty of about ±0.005°. If one oscillates (OSC=3) the total uncertainty cannot get any lower as one has a value of $N_{DG} \approx 284$ which places an upper bound of the value of $u(2\theta_{0\text{-grain}}) \approx \pm 0.0049°$.

Testing the model with the reference samples

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

From multiple measurement

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right)$$

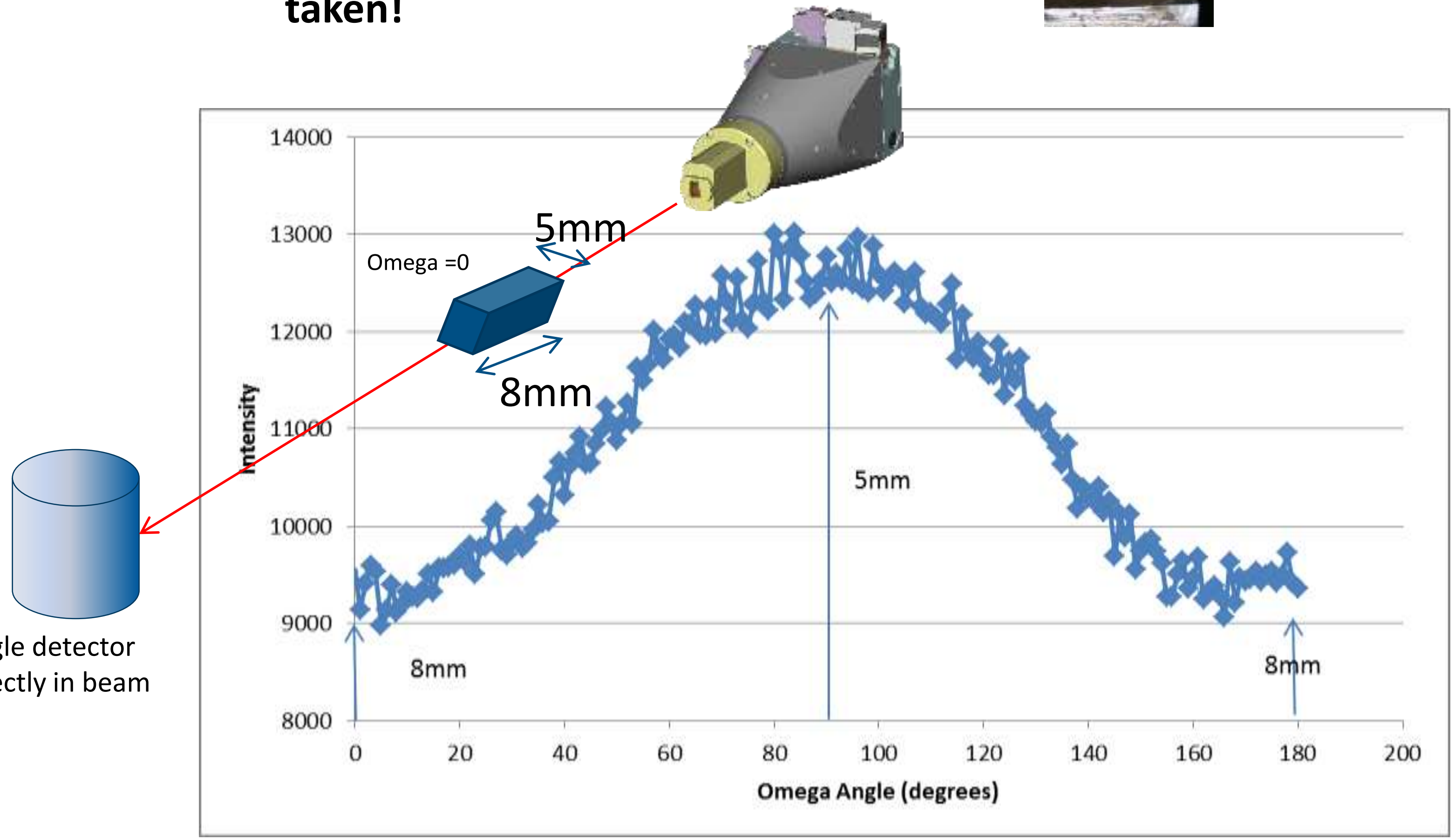
$$P \approx \frac{(D_H + \eta_M) * (OSC + \omega_M)}{4\pi} * m^{hkl}$$

Experiment	Expectation
Increase gv (constant P)	S_G Value remains constant, N_{DG} value increases
Increase P (m^{hkl}) (constant gv)	S_G Value remains constant, N_{DG} value increases
Increase P (OSC) (constant gv)	S_G Value remains constant, N_{DG} value increases
$u(2\theta_{grain})$ versus SD_{Gauss}	Should be a straight line going through zero

Estimating the number of diffracting grains from measurement

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

From multiple measurement
Care has to be taken!



If the sample is **not round**, the single detector in the primary neutron beam can be used to indicate thickness for the normalization of the intensity data.

This was one of the many advantages of measuring on E3

Increase gv	S_G Value remains constant, N_{DG} value increases
Increase P (m^{hkl})	S_G Value remains constant, N_{DG} value increases

hkl {3 1 1} m^{hkl} =24

Results of the ***hkl*** **{3 1 1}** Bragg reflection (***P*=0.01131**) for TG4 reference coupons SET Y.

		Ave $2\theta_0$	SD_{Gauss} [°]	B/H	Fitted steps	Ave I	$u(I)$	N_{DG}	$u(2\theta_0)$ [°]	$u(2\theta_{0-fitting})$ [°]	$u(2\theta_{0-grain})$ [°]	gv [mm³]	S_G [µm]
Parent	1	86.616	0.178	1.02	11	14346	2120	**46**	0.0068	0.0029	0.0062	**2**	**42**
Parent	2	86.613	0.183	0.33	28	13343	1253	**113**	0.0094	0.0022	0.0091	**8**	**84**
Parent	3	86.616	0.187	0.18	28	13900	1182	**138**	0.0072	0.0019	0.0069	**18**	**91**
Parent	4	86.613	0.192	0.12	28	13795	1007	**188**	0.0073	0.0019	0.0070	**32**	**109**
Weld Top	1	86.635	0.213	0.22	55	12907	3746	**11.9**	0.0148	0.0024	0.0146	**18**	**137**
Weld Top	2	86.629	0.215	0.15	55	13201	3933	**11.3**	0.0141	0.0022	0.0139	**32**	**159**
Weld Bottom	1	86.647	0.197	0.40	74	11451	8664	**1.7**	0.0432	0.0027	0.0431	**8**	**227**
Weld Bottom	2	86.637	0.199	0.18	109	14719	10245	**2.1**	0.0280	0.0020	0.0280	**18**	**221**
Weld Bottom	3	86.637	0.204	0.13	55	13821	9931	**1.9**	0.0301	0.0020	0.0301	**32**	**276**

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

hkl {2 2 2} m^{hkl} =8

Results of the ***hkl*** **{2 2 2}** Bragg reflection (***P*=0.00377**) for TG4 reference coupons SET Y.

		Ave $2\theta_0$	SD_{Gauss} [°]	B/H	Fitted steps	Ave I	$u(I)$	N_{DG}	$u(2\theta_0)$ [°]	$u(2\theta_{0-fitting})$ [°]	$u(2\theta_{0-grain})$ [°]	gv [mm³]	S_G [µm]
Parent	1	91.645	0.209	2.98	11	5462	1447	**14.3**	0.0281	0.0087	0.0267	**2**	**69**
Parent	2	91.639	0.213	1.00	28	4706	965	**23.8**	0.0133	0.0061	0.0119	**8**	**63**
Parent	3	91.637	0.215	0.54	28	4782	882	**29.4**	0.0116	0.0049	0.0105	**18**	**76**
Parent	4	91.635	0.220	0.38	28	4681	691	**45.9**	0.0121	0.0046	0.0112	**32**	**94**
Weld Top	1	91.658	0.227	0.47	51	6281	3861	**2.6**	0.0360	0.0044	0.0357	**18**	**165**
Weld Top	2	91.659	0.230	0.33	55	5584	3839	**2.1**	0.0296	0.0043	0.0293	**32**	**174**
Weld Bottom	1	91.664	0.232	1.22	42	7399	7276	**1.0**	0.0603	0.0057	0.0600	**8**	**175**
Weld Bottom	2	91.660	0.234	0.87	62	5494	5625	**1.0**	0.0531	0.0059	0.0528	**18**	**210**
Weld Bottom	3	91.657	0.234	0.62	40	3961	4644	**0.7**	0.0571	0.0062	0.0567	**32**	**267**

$$S_G \approx \left(\frac{P*gv}{\left(\frac{0.5*SD_{Gauss}}{u(2\theta_{grain})}\right)^2}\right)^{1/3}$$

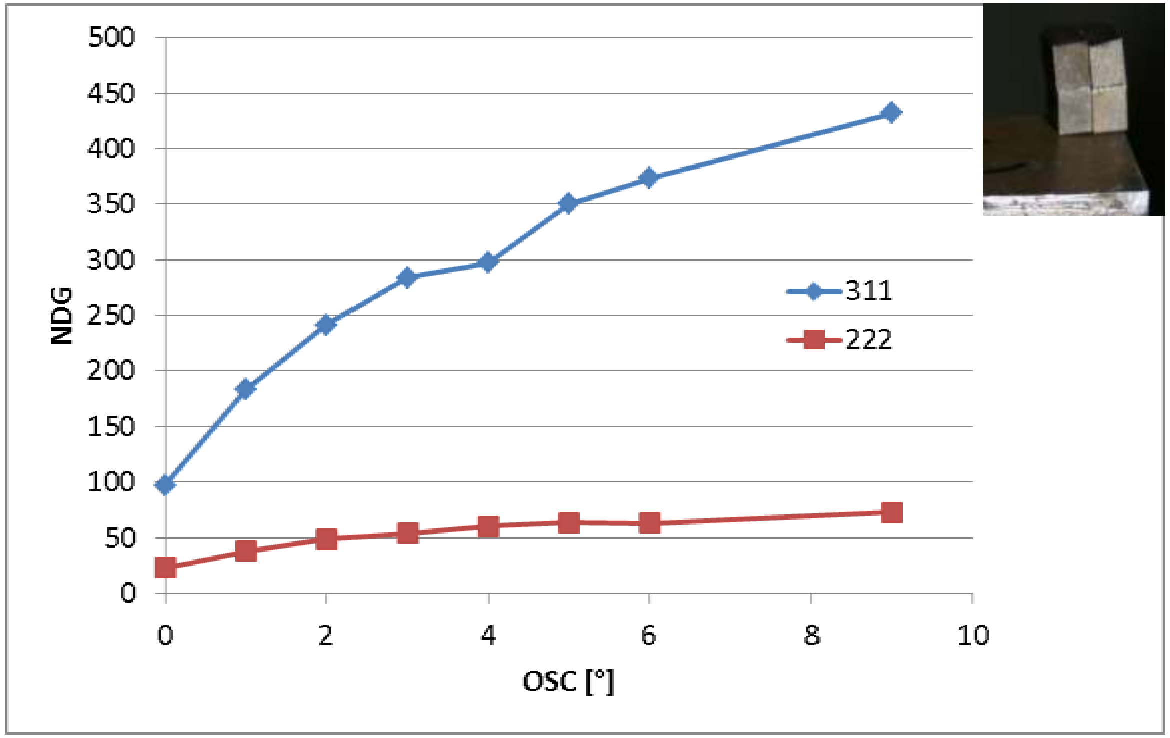
Mosaicity

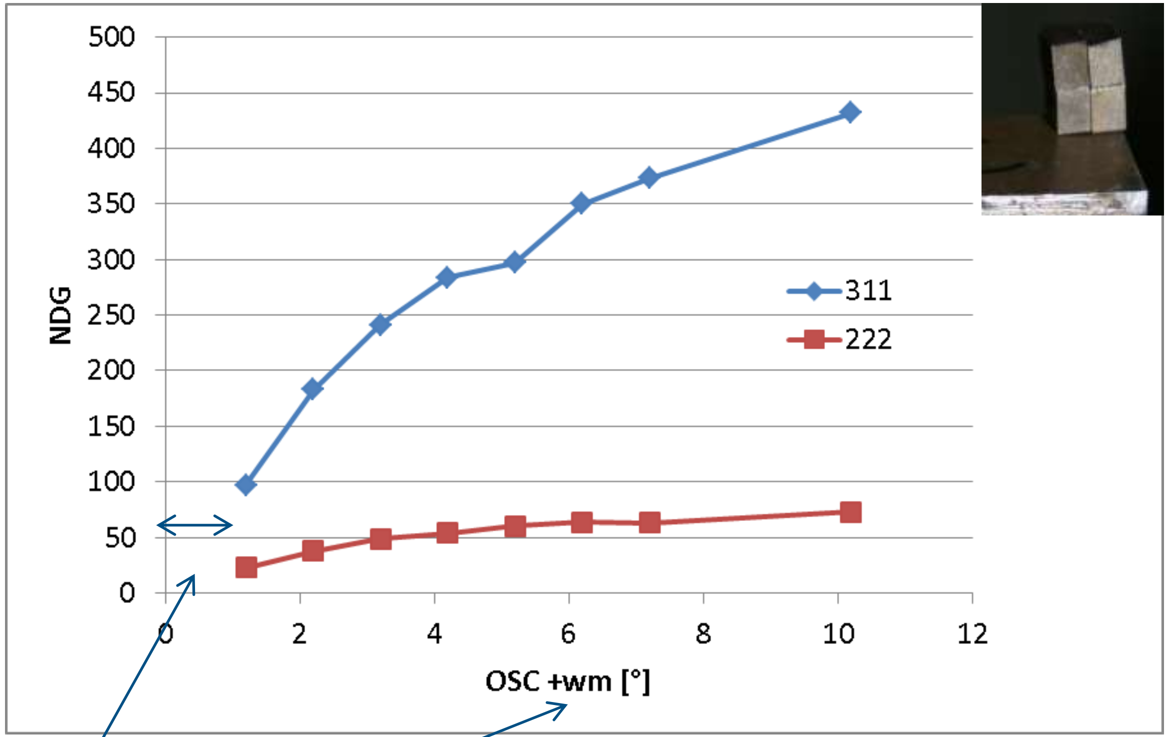
Increase P (OSC)	S_G Value remains constant, N_{DG} value increases

Assumed to be the same

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

$$P \approx \frac{(D_H+\eta_M)*(OSC+\omega_M)}{4\pi} * m^{hkl}$$





For the analysis we used an estimated value of ω_m□1.2°

Measurements made at the ESRF by the JRC saw a range of mosaicity values of up to 1.55° for the {1 1 1} reflection.

Stepwise Oscillation of the TG4 'black' parent reference coupon 311

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

$$u(2\theta_{0-fitting})^2 \approx \left(\frac{SD_{Gauss}{}^2}{I}\right)\left[1 + 2\left(2^{\frac{1}{2}}\right)\frac{B}{H}\right]$$

$$P \approx \frac{(D_H + \eta_M) * (OSC + \omega_M)}{4\pi} * m^{hkl}$$

$$S_G \approx \left(\frac{P * gv}{\left(\frac{0.5 * SD_{Gauss}}{u(2\theta_{grain})}\right)^2}\right)^{1/3}$$

$$u(2\theta_0) \approx \left(u(2\theta_{0-fitting})^2 + u(2\theta_{0-grain})^2\right)^{1/2}$$

$$u(2\theta_{grain-0}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

row	N_{DG}	No. of peaks	OSC [°]	Average $2\theta_0$ [°]	$u(2\theta_0)$ [°]	$u(2\theta_{0\text{-fitting}})$ [°]	$u(2\theta_{0\text{-grain}})$ (a) [°]	$u(2\theta_{0\text{-grain}})$ (b) [°]	P	S_G [µm]
1	97	219	0	85.3688	0.0120	0.0037	0.0115	0.0111	0.0115	100
2	183	109	1	85.3688	0.0077	0.0026	0.0073	0.0081	0.0210	91
3	241	73	2	85.3688	0.0065	0.0021	0.0061	0.0070	0.0306	92
4	284	54	3	85.3687	0.0053	0.0018	0.0049	0.0065	0.0401	87
5	297	43	4	85.3687	0.0048	0.0016	0.0045	0.0063	0.0497	87
6	350	36	5	85.3687	0.0050	0.0015	0.0048	0.0058	0.0592	97
7	373	31	6	85.3687	0.0045	0.0014	0.0043	0.0057	0.0687	95
10	432	21	9	85.3688	0.0035	0.0011	0.0033	0.0053	0.0974	89

Increase P (OSC)	S_G Value remains constant, N_{DG} value increases

Average FWHM of peaks = 0.52°

Stepwise Oscillation of the TG4 ‘black’ parent reference coupon 222

$$N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

$$u(2\theta_{0-fitting})^2 \approx \left(\frac{SD_{Gauss}{}^2}{I}\right)\left[1 + 2\left(2^{\frac{1}{2}}\right)\frac{B}{H}\right]$$

$$P \approx \frac{(D_H + \eta_M) * (OSC + \omega_M)}{4\pi} * m^{hkl}$$

$$S_G \approx \left(\frac{P*gv}{\left(\frac{0.5*SD_{Gauss}}{u(2\theta_{grain})}\right)^2}\right)^{1/3}$$

$$u(2\theta_0) \approx \left(u(2\theta_{0-fitting})^2 + u(2\theta_{0-grain})^2\right)^{1/2}$$

$$u(2\theta_{grain-0}) \approx \frac{0.5*SD_{Gauss}}{(N_{DG})^{1/2}}$$

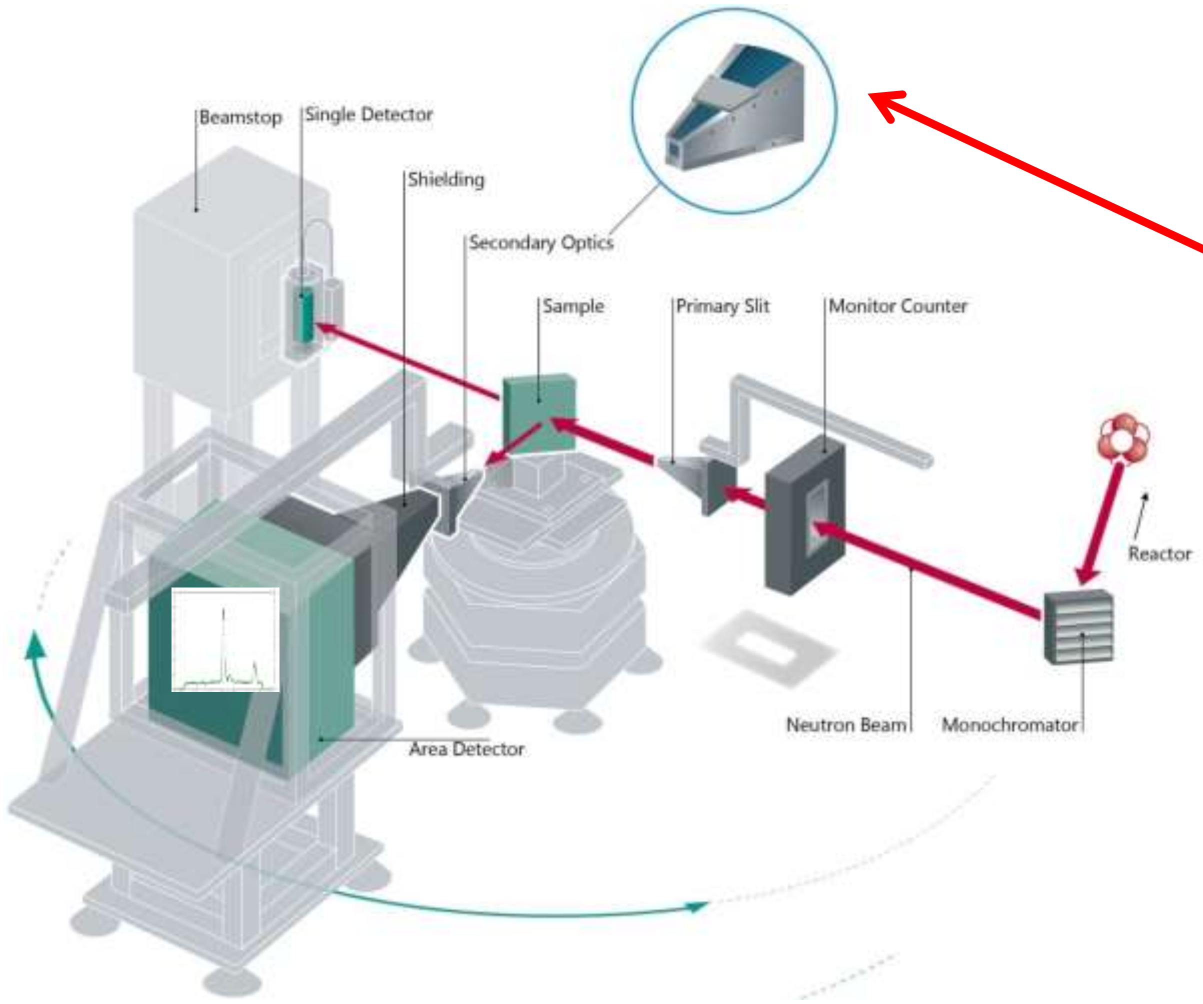
row	N_{DG}	No. of peaks	OSC [°]	Average $2\theta_0$ [°]	$u(2\theta_0)$ [°]	$u(2\theta_{0\text{-fitting}})$ [°]	$u(2\theta_{0\text{-grain}})$ (a) [°]	$u(2\theta_{0\text{-grain}})$ (b) [°]	P	S_G [µm]
1	23	219	0	90.1512	0.0260	0.0113	0.0235	0.0249	0.0038	106
2	38	109	1	90.1512	0.0176	0.0080	0.0157	0.0194	0.0070	99
3	49	73	2	90.1512	0.0153	0.0065	0.0138	0.0170	0.0102	103
4	54	54	3	90.1513	0.0136	0.0056	0.0124	0.0161	0.0134	105
5	60	43	4	90.1514	0.0135	0.0050	0.0125	0.0154	0.0166	114
6	64	36	5	90.1513	0.0124	0.0046	0.0115	0.0148	0.0197	114
7	63	31	6	90.1512	0.0122	0.0043	0.0114	0.0149	0.0229	119
10	73	21	9	90.1514	0.0112	0.0035	0.0106	0.0139	0.0325	128

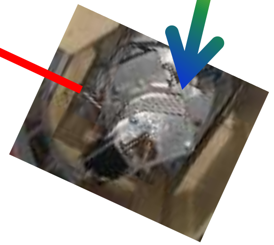
Increase P (OSC)

S_G Value remains constant, N_{DG} value increases

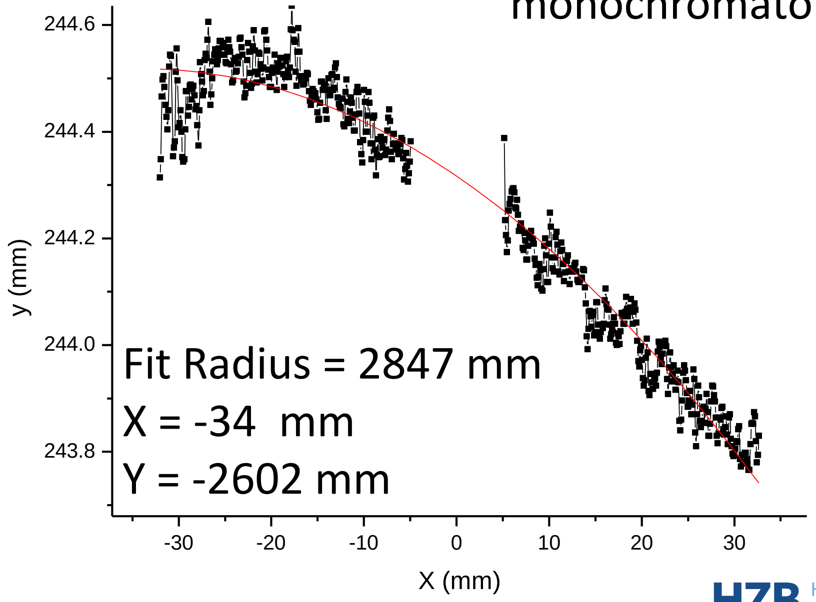
Average FWHM of peaks = 0.57°

$u(2\theta_{grain})$ versus SD_{Gauss} | Should be a straight line going through zero



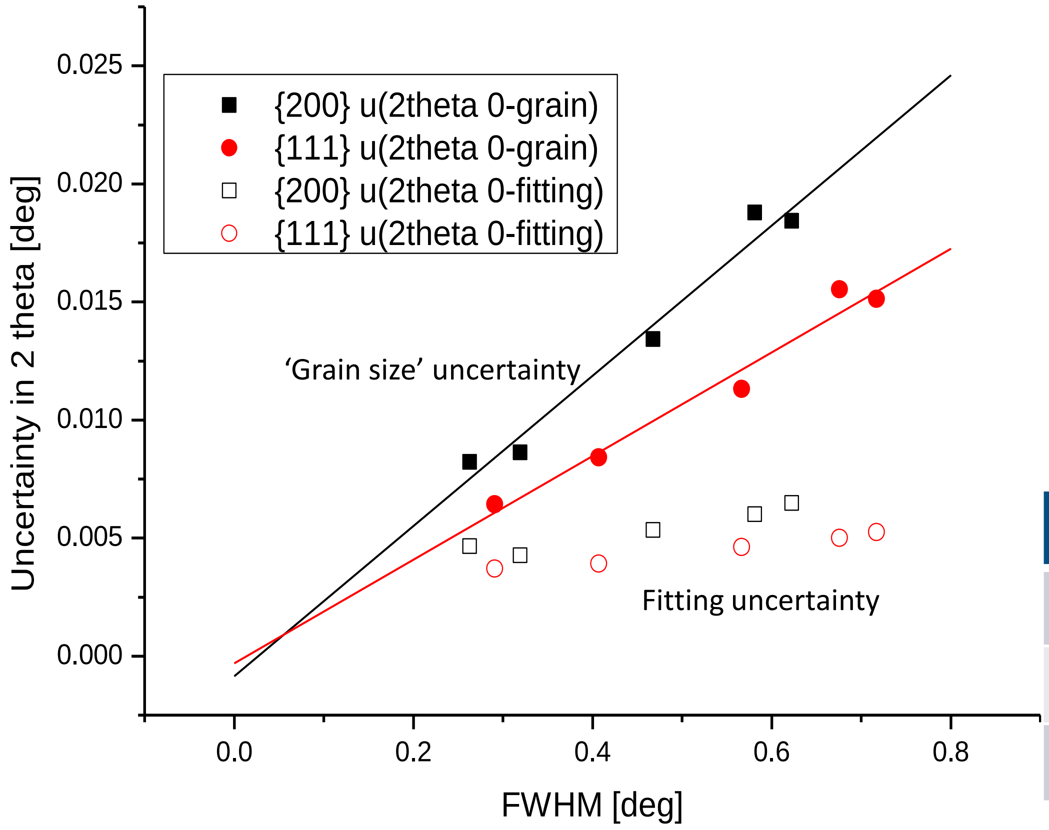


Bending radius of monochromator



$u(2\theta_{grain})$ versus SD_{Gauss} — Should be a straight line going through zero

Grain size uncertainty contribution as a function of instrument resolution compared to corresponding fitting uncertainties for this particular data set.



Adjusted for path length

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}} = \frac{0.21 * FWHM}{(N_{DG})^{1/2}} \qquad N_{DG} \approx \left(\frac{I}{u(I)}\right)^2$$

hkl	$2\theta_{hkl}$	m^{hkl}	N_{DG}	N_{DG}
200	48.6°	6	45 ± 10	69 ± 14
111	41.8°	8	94 ± 20	121 ± 6
111/200		1.33	2.10 ± 0.91	1.75 ± 0.44

Greater proportion of Debye –Scherrer ring on detector

Verification of the model

Experiment	Expectation	Result
Increase gv	S_G Value remains constant, N_{DG} value increases	Verified
Increase P (m^{hkl})	S_G Value remains constant, N_{DG} value increases	Verified
Increase P (OSC)	S_G Value remains constant, N_{DG} value increases	Verified
$u(2\theta_{grain})$ versus SD_{Gauss}	Should be a straight line going through zero	Verified

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}} \approx \frac{0.5 * SD_{Gauss}}{\left(P * \frac{gv}{(S_G)^3}\right)^{1/2}}$$

$$S_G \approx \left(\frac{P * gv}{\left(\frac{0.5 * SD_{Gauss}}{u(2\theta_{grain})}\right)^2} \right)^{1/3}$$

Applying the model to the main sample

	HZB ref	HZB ref	FRM II (a)	FRM II (b)	HZB
Date	2013	2013	2009	2010	2009
Sample	Ref SET Y	Ref SET Y	TG4 3-1A plate	TG4 3-1A plate	TG4 3-1A plate
Ref	SET Y	SET Y	SET Z	SET Z	SET W
{h k l}	{2 2 2}	{3 1 1}	{3 1 1}	{3 1 1}	{3 1 1}
m^{hkl}	8	24	24	24	24
ω_M [°]	1.2	1.2	1.2	1.2	1.2
η_M [°]	1.2	1.2	1.2	1.2	1.2
D_H [°]	15	15	10	12	12
OSC[°]	0	0	6	8	10
Optics [mm³]	3×3×2	3×3×2	2×2×2	3×3×2.1	3×3×3
Type of Optics	ROC	ROC	Slits	ROC	Slits
2θ[°]	91.6	86.6	92.5	92.9	86.5
gv [mm³]	18	18	8.01	18.92	27.05
P	0.00377	0.01131	0.04691	0.07065	0.08601
SD_{Gauss} [°]	0.215	0.187	0.246	0.265	0.177

$$P \approx \frac{(D_H + \eta_M) * (OSC + \omega_M)}{4\pi} * m^{hkl}$$

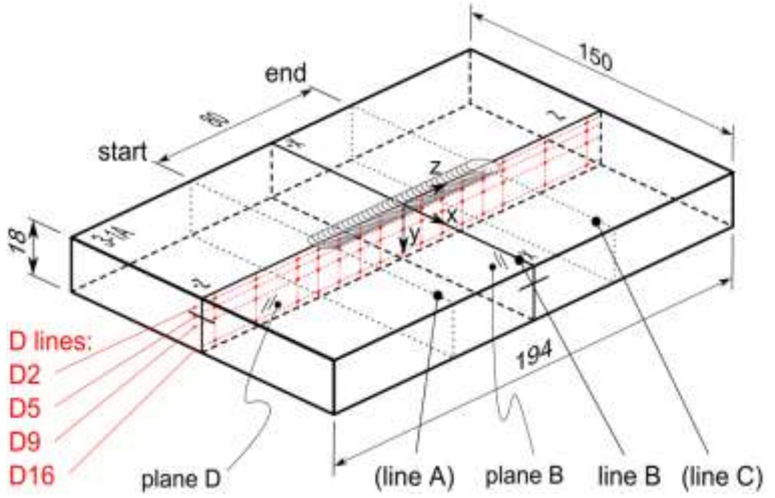
$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right)$$

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

The D lines were ‘single shot’

The D9 line is completely within the parent material of the specimen: Should show up the least grain size effect

Position	Reference	Grain size estimation S_G	Main sample
Parent	Black	90µm	D9
Weld Bottom	Green	180µm	D5
Weld Top	Red	260µm	D2



Applying the model to main sample

Position	Reference	Grain size estimation S_G	Main sample
Parent	Black	90µm	D9
Weld Bottom	Green	180µm	D5
Weld Top	Red	260µm	D2

hkl	E_{hkl} [GPa]	ν_{hkl}
311	183.6	0.306
222	209.4	0.278

$u(2\theta)$ $u(2\theta_0)$

$u(\varepsilon)$

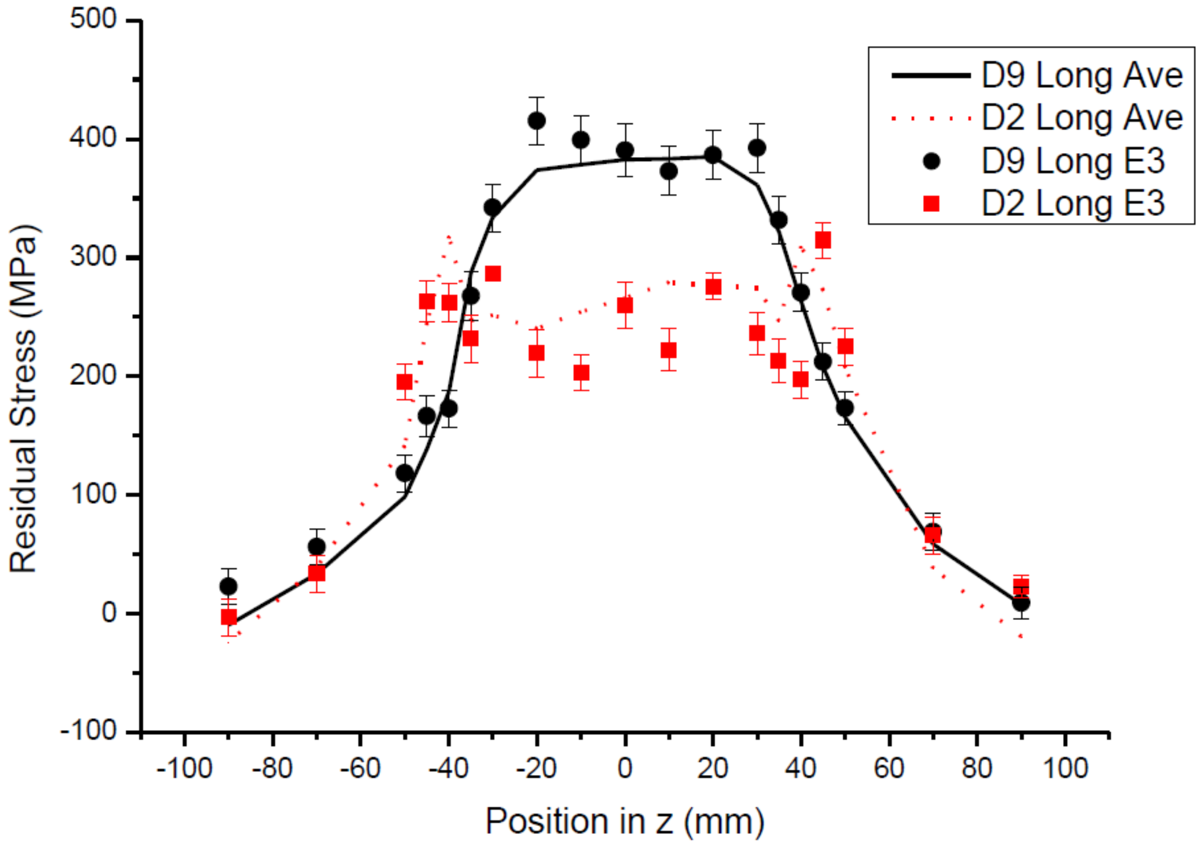
$$u(\varepsilon) = \frac{1}{tan\theta_0}\left[u(\theta_{fitting})^2 + u(\theta_{grain})^2 + u(\theta_{0-fitting})^2 + u(\theta_{0-grain})^2\right]^{\frac{1}{2}}$$

$u(\sigma)$

$$u(\sigma_{xx}) \cong \left[\left(\frac{E_{hkl}(1-\nu_{hkl})}{(1+\nu_{hkl})(1-2\nu_{hkl})}u(\varepsilon_{xx})\right)^2 + \left(\frac{\nu_{hkl}E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\right)^2\left(u(\varepsilon_{yy})^2 + u(\varepsilon_{zz})^2\right)\right]^{\frac{1}{2}}$$

(Assuming $u(\varepsilon_{xx}) = u(\varepsilon_{yy}) = u(\varepsilon_{zz})$)

‘Actual Uncertainties’ Comparison to Robust Average



$u(\sigma)$

For the D2 line the measurement points between z = -40 and 40 mm are within the weld material.

The scatter of the data is clearly more than that of the D9 line (which is completely in parent material).

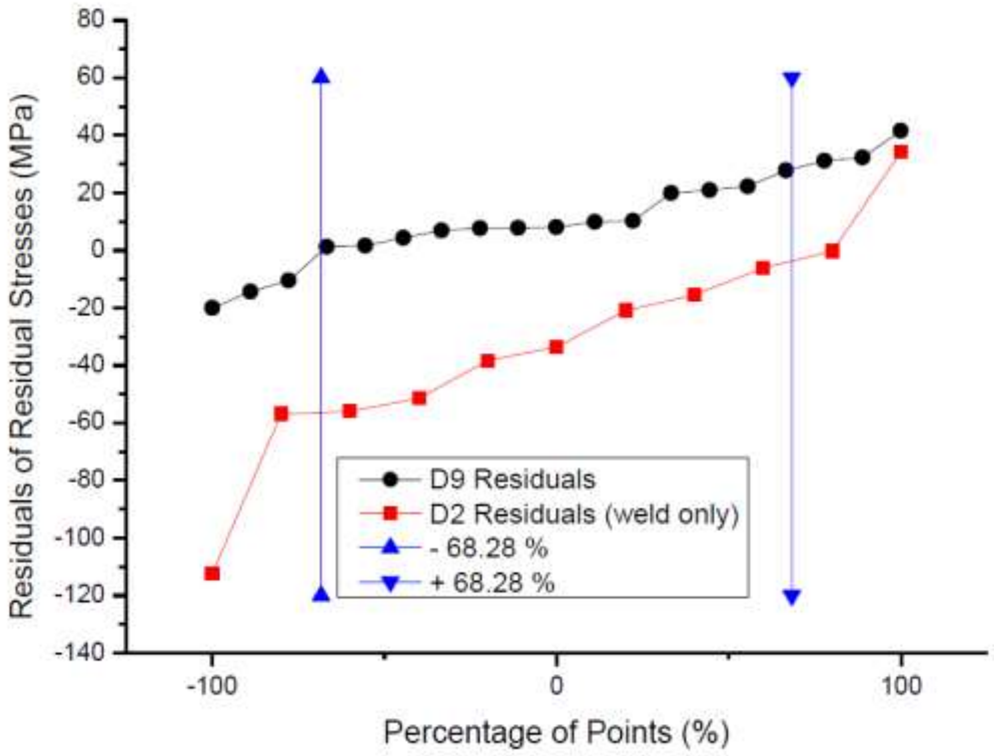
Many measurements were made in NeT-TG4 and this gave a good opportunity to calculate a robust average of all the measurements.

One can take away this robust average from each data set and study the residuals to calculate the actual systematic offsets and random uncertainties (which contains the grain size contribution as well as fitting uncertainties).

It should be noted that the robust average contained measurements on other nominally the same NeT-TG4 specimens, i.e. the 1-1A and 2-1A specimens as well as the 3-1A.

Analyzing the residuals

Longitudinal direction



$u(\sigma)$

In order to discriminate against outliers in the data, after taking away the appropriate robust average from each data set, the residuals were arranged equidistantly in magnitude order .

Subsequently after scaling the abscissa: -100% to 100%, a linear fit was made between ± 68.28%, corresponding to ±1 standard deviation (between the two vertical lines in the figure).

This linear fit gives simultaneously the systematic offset and the total random uncertainty from the gradient.

Of the total 19 points in each direction of the D9 line, 13 points lie within the first standard deviation.

For the D5 and D2 lines, the measurement points between z = -40 and 40 mm are within the weld material and only these were considered to estimate the systematic and
random uncertainties. This meant that out of a total number of points of 11 in the weld region, 7 points laid within the first standard deviation .

Comparison of Model with Actual Uncertainties

20 years NeT The European Network on Neutron Techniques Standardization for Structural Integrity 2002 - 2022

Parent

Sample	*P*	$u(\sigma_{grain})$ D9 Model [MPa]	$u(\sigma_{model})$ Including fitting uncertainty of 15 [MPa]	$u(\sigma_{actual})$ Random uncertainty from R-fits [MPa]
Black coupon {2 2 2}	0.00377	28.7	32.4	31.0
Black coupon {3 1 1}	0.01131	15.3	21.5	19.7
FRM II (a) D9	0.04691	13.4	20.1	15.8
FRM II (b) D9	0.07065	7.6	16.8	22.8
HZB E3 D9	0.08601	4.3	15.6	14.8

Weld Bottom

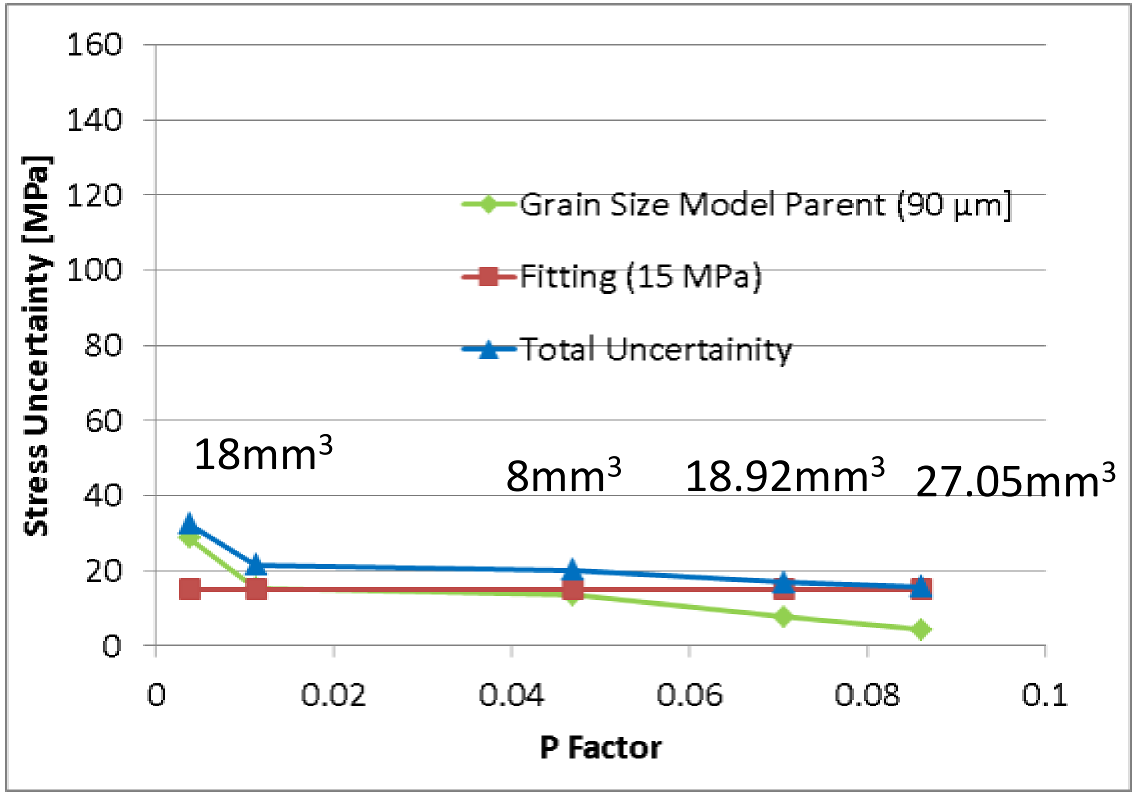
Sample	*P*	D5 Model [MPa]	Including fitting uncertainty of 15 [MPa]	Random uncertainty from R-fits [MPa]
Green coupon {2 2 2}	0.00377	84.0	85.4	95.8
Green coupon {3 1 1}	0.01131	43.4	45.9	40.7
FRM II (a) D5	0.04691	37.9	40.8	52.5
FRM II (b) D5	0.07065	21.5	26.2	29.1
HZB E3 D5	0.08601	12.2	19.3	18.2

Weld Top

Sample	*P*	D2 Model [MPa]	Including fitting uncertainty of 15 [MPa]	Random uncertainty from R-fits [MPa]
Red coupon {2 2 2}	0.00377	145.9	146.7	141.6
Red coupon {3 1 1}	0.01131	75.3	76.8	76.9
FRM II (a) D2	0.04691	65.9	67.5	49.7
FRM II (b) D2	0.07065	37.4	40.2	39.2
HZB D2	0.08601	21.1	25.9	33.9

Parent

Sample	*P*	D9 Model [MPa] $u(\sigma_{grain})$	Including fitting uncertainty of 15 [MPa] $u(\sigma_{model})$	Random uncertainty from R-fits [MPa] $u(\sigma_{actual})$
Black coupon {2 2 2}	0.00377	28.7	32.4	31.0
Black coupon {3 1 1}	0.01131	15.3	21.5	19.7
FRM II (a) D9	0.04691	13.4	20.1	15.8
FRM II (b) D9	0.07065	7.6	16.8	22.8
HZB E3 D9	0.08601	4.3	15.6	14.8



$$u(\sigma) \approx (u(\sigma_{fitting})^2 + u(\sigma_{grain})^2)^{1/2}$$

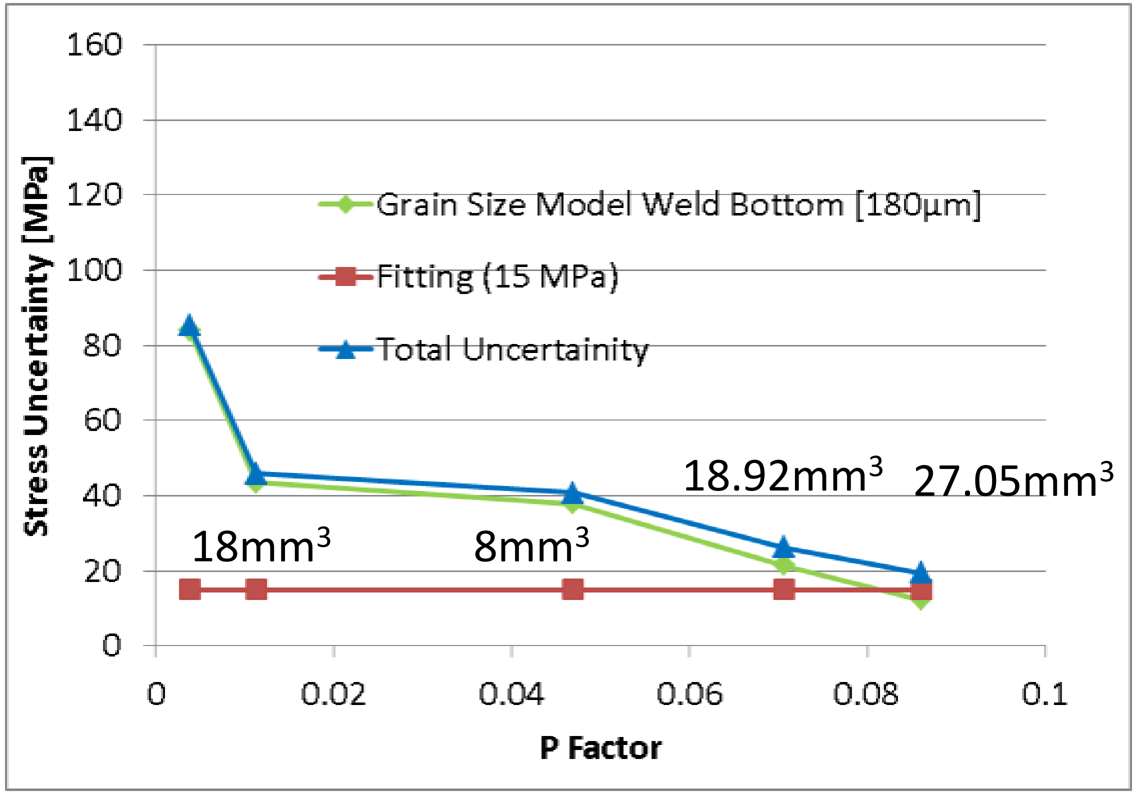
=15 MPa

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right) \qquad S_G = 90\mu m$$

Weld Bottom

Sample	*P*	D5 Model [MPa] $u(\sigma_{grain})$	Including fitting uncertainty of 15 [MPa] $u(\sigma_{model})$	Random uncertainty from R-fits [MPa] $u(\sigma_{actual})$
Green coupon {2 2 2}	0.00377	84.0	85.4	95.8
Green coupon {3 1 1}	0.01131	43.4	45.9	40.7
FRM II (a) D5	0.04691	37.9	40.8	52.5
FRM II (b) D5	0.07065	21.5	26.2	29.1
HZB E3 D5	0.08601	12.2	19.3	18.2



$$u(\sigma) \approx \left(u(\approx_{fitting})^2 + u(\approx_{grain})^2\right)^{1/2}$$

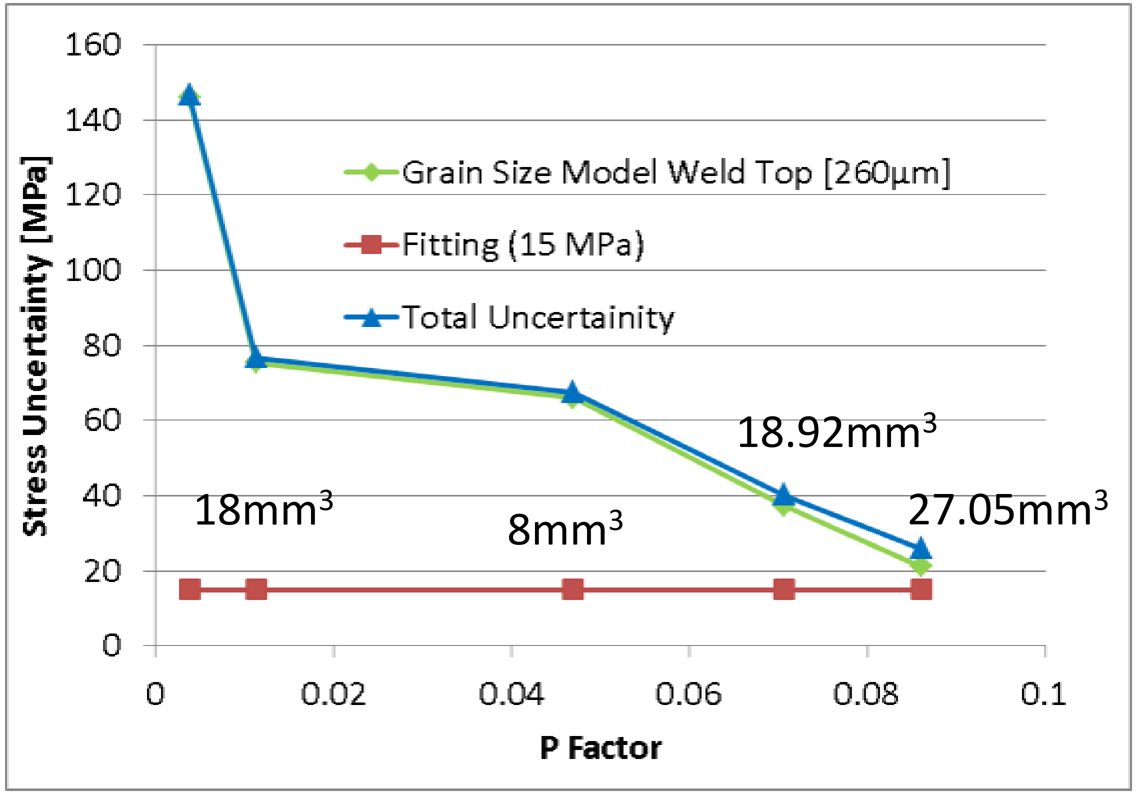
=15 MPa

$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right) \qquad S_G = 180\mu m$$

Weld Top

Sample	*P*	D2 Model [MPa] $u(\sigma_{grain})$	Including fitting uncertainty of 15 [MPa] $u(\sigma_{model})$	Random uncertainty from R-fits [MPa] $u(\sigma_{actual})$
Red coupon {2 2 2}	0.00377	145.9	146.7	141.6
Red coupon {3 1 1}	0.01131	75.3	76.8	76.9
FRM II (a) D2	0.04691	65.9	67.5	49.7
FRM II (b) D2	0.07065	37.4	40.2	39.2
HZB D2	0.08601	21.1	25.9	33.9



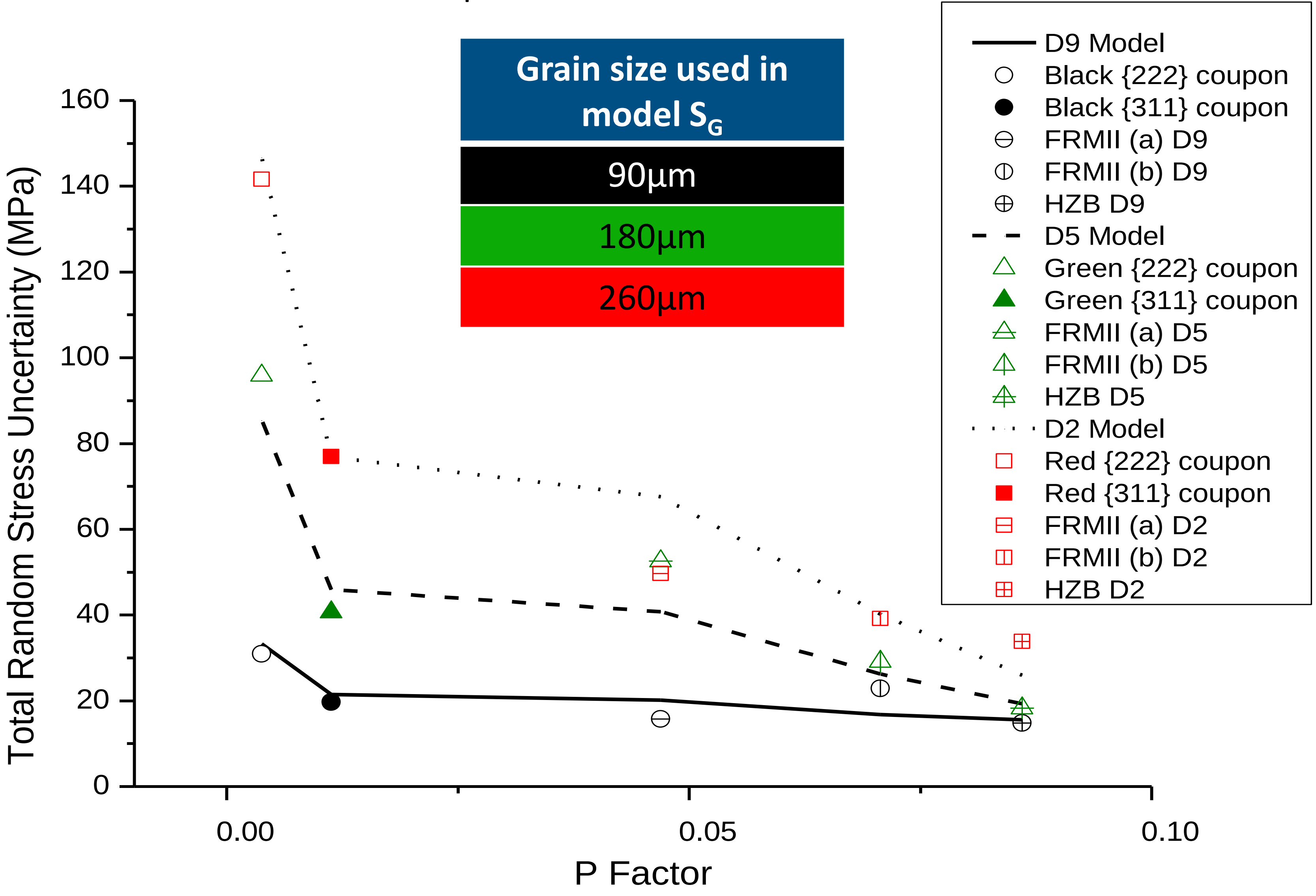
$$u(\sigma) \approx (u(\sigma_{fitting})^2 + u(\sigma_{grain})^2)^{1/2}$$

=15 MPa

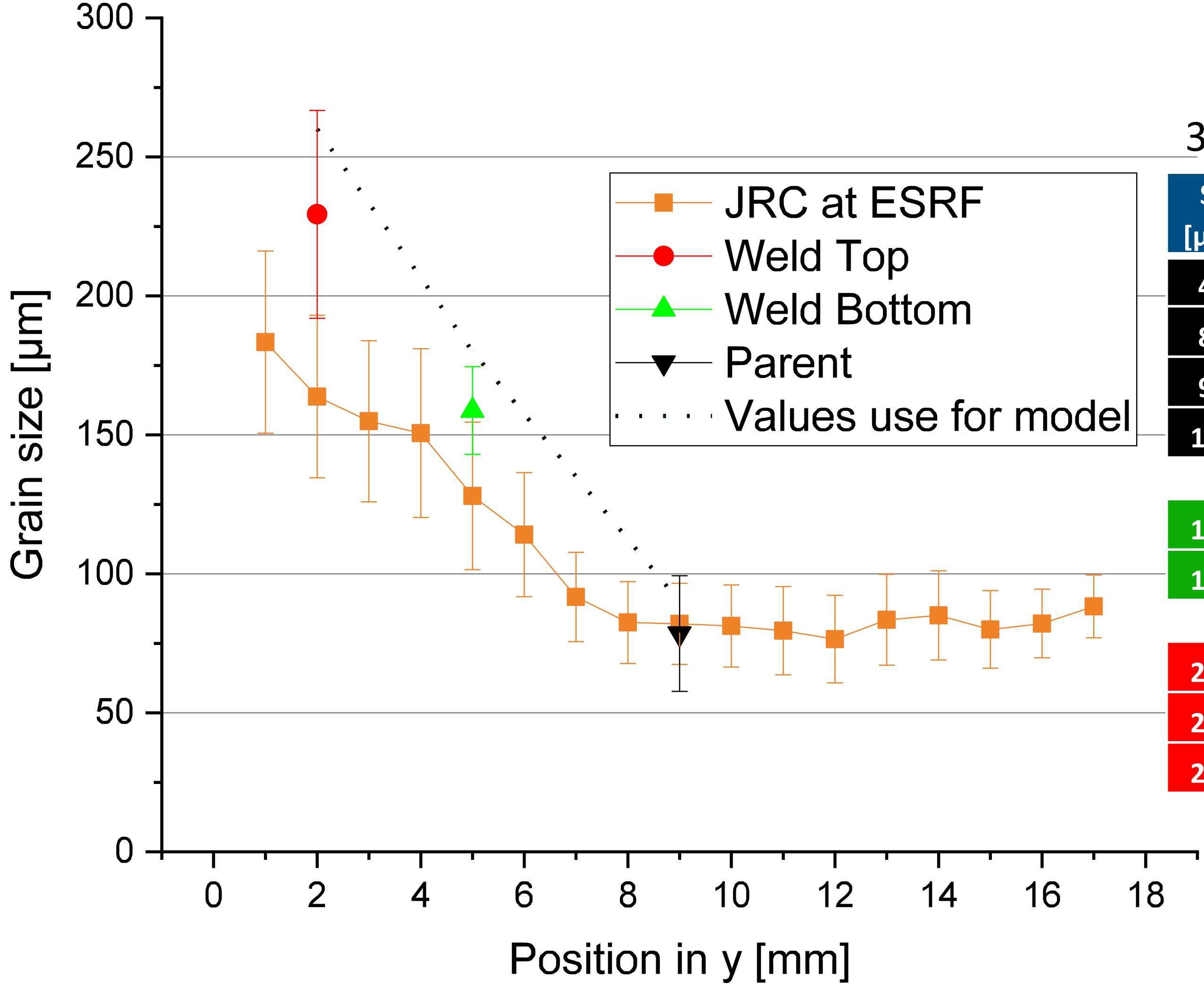
$$u(2\theta_{grain}) \approx \frac{0.5 * SD_{Gauss}}{(N_{DG})^{1/2}}$$

$$N_{DG} \approx \left(P * \frac{gv}{(S_G)^3}\right) \qquad S_G = 260\mu m$$

$u(\sigma_{model})$ Compared to $u(\sigma_{actual})$



Grain size



311	222
S_G [µm]	S_G [µm]
42	69
84	63
91	76
109	94
137	165
159	174
227	175
221	210
276	267

JRC at ESRF (ID15a, spiral slit set up). High energy synchrotron X-rays (using 5 peaks)

Grain size estimation from the microscopic studies at the Open University in the parent material region provided values of 75 ±12 µm, over grains without twins and 67±10 µm, over grains with twins, each average calculated over 20 values.

This agrees well with the JRC/ESRF estimation 83 ± 4 µm (from 11 values, y=8 to 17mm, in Figure).

A separate study in the parent material of the TG4 specimen made by JRC (made at a different location in the specimen) gave a result of 92 ± 9 µm (with a range of values 78-106 µm). This agrees well with the grain size estimations from reference specimens. This indicates that the grain size does vary slightly from place to place in the parent material.

Uncertainty in the uncertainties

The peak fitting uncertainty is often **not enough** to describe completely the true random uncertainty of a neutron strain measurement and resultant stress determinations.

$$\varepsilon = \frac{Sin\theta_0}{Sin\theta} - 1 \longrightarrow u(\varepsilon) = \frac{1}{tan\theta_0}\left[u(\theta_{fitting})^2 + u(\theta_{0-fitting})^2\right]^{\frac{1}{2}}$$

The traditional way

Detecting not enough diffracting grains also contributes to the random uncertainty.

A simple model is needed to estimate the **extra random uncertainty contribution due to the so-called grain size statistics.**

$$u(\varepsilon) = \frac{1}{tan\theta_0}\left[u(\theta_{fitting})^2 + u(\theta_{grain})^2 + u(\theta_{0-fitting})^2 + u(\theta_{0-grain})^2\right]^{\frac{1}{2}}$$

The way we should do it

Either by multiple measurement or ‘modeling’

Remarks and Conclusions

Single shot measurement of ‘main sample’: Only gives fitting uncertainty, however is the normal measurement practice

Fitting uncertainty is time dependent whereas the uncertainty due to grain size is dependent on the number of detected diffracting grains

Propagation of only the fitting uncertainty of Bragg peaks is not enough with ‘large grains’

Multiple Measurement of ‘main sample’: Time constraints, Expensive Beam Time, not the normal practice

Multiple Measurement of ‘Representative References’: Can be quick and give information about grain-size uncertainty in the ‘main sample’ and used to estimate the extra uncertainty due to grain size.

Also a priori knowledge of grain size can also be directly used in the model to estimate the extra uncertainty needed to add to the fitting uncertainty.

Thank you