University of Ulm
Institute for Micro- and Nanomaterials

Lab „Materials Science“

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Lab C1 & C2: XRD
lab on July, 12th 2007

1 Questions for Preparation

1.1 Interpolar separation

Consider a plane hkl in a crystal lattice

(a) Prove that the reciprocal lattice vector $\vec{G} = h\vec{A} + k\vec{B} + l\vec{C}$ is perpendicular to this plane.

If $\vec{G}$ is perpendicular to the hkl-plane, we can assume, that it is identical to the normal vector $\vec{n}$ of the plane. Every plane can be written in its normal form $E = n_1\vec{A} + n_2\vec{B} + n_3\vec{C}$. Let this plane be our crystal plane so we can write it as $E = h\vec{A} + k\vec{B} + l\vec{C}$, being $\vec{A}$, $\vec{B}$ and $\vec{C}$ the coordinate vectors.

For example we can take a look at the 110-plane displayed in Fig. 1. Its reciprocal vector can be written as $\vec{G} = 1 \cdot \vec{A} + 1 \cdot \vec{B} + 0 \cdot \vec{C} = \vec{A} + \vec{B}$, which is the normal vector of the 110-plane and therefore perpendicular to it.

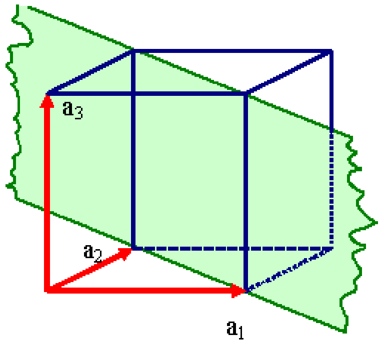


Fig. 1: 110-plane of a cubic crystal

(b) Prove that the distance between two adjacent parallel planes of the lattice is $d(hkl) = 2\pi/|\vec{G}|$
and
(c) Show for a simple cubic lattice that $d = \frac{a}{\sqrt{h^2+k^2+l^2}}$, where a denotes the lattice parameter.

Let's take for example a cubic crystal and determine the distance between two 110-planes. As can be seen easily in Fig. 1 the lattice parameter a (which is a number for the length of the unit cell) is $\sqrt{2}$. The distance between two 110-planes must be 1, because of the packaging of the crystal. That means that the length of the unit cell a is bigger than distance d_{hkl} by the factor of $\sqrt{2}$. So this is exactly what the equation above gives us as a result. (The factor 2π is only a scaling factor which is useful for other calculations)

1.2 Bragg peak positions

The sample that you will measure during this laboratory experiment is primarily made up of nickel. Ni has a face-centered cubic (fcc) lattice with a lattice parameter a of 3.542Å.

(a) Calculate the structure factor F for nickel explicitly - don't just copy the result from a book like Kittel. Take f to be the atomic form factor for nickel. What are hkl values of the first eight allowed Bragg peaks?

The structur factor depends of the type and the arrangement of the atoms in a unit cell. It can be calculated by the formula

$$F = \int_0^a \int_0^b \int_0^c \rho(x, y, z) \exp\left\{2\pi i \left(\frac{hx}{a} + \frac{ky}{b} + \frac{lz}{c}\right)\right\} \text{dxdydz}$$

For a fcc lattice structur follows that F is not zero, if h, k and l are **all** odd or **all** even.

So the first eight peaks for a fcc-material will appear at 111, 200, 220, 222, 113, 133, 333, 400.

(b) Using the results from question 1(c) in combination with the Bragg equation, calculate the 2Θ values of the first six Bragg peaks for Ni. Assume that cobalt radiation with a wavelength of 1.79Å is used in the measurement. Be sure to specify 2Θ in degrees (rather than radians).

With the formula $\Theta = \arcsin\left(\lambda\sqrt{h^2 + k^2 + l^2}\frac{1}{2a}\right)$ the angles for the first six peaks are:

peak	111	200	220	222	113	133
angle in °	26.09	30.52	45.91	61.67	57.38	n.a.

1.3 Diffraction from a one-dimensional crystal

Assume that a one-dimensional crystal is made up of a linear arrangement f identical point scattering centers located at every lattice point $\vec{p}_m = m\vec{a}$, where m is an integer ranging from 1 to M. The total scattered radiation amplitude will be proportional to $F = \sum \exp[-im(\vec{a} \cdot \Delta\vec{k})]$.

(a) Show that the sum over M is

$$F = \frac{1 - \exp[-iM(\vec{a} \cdot \Delta\vec{k})]}{1 - \exp[-i(\vec{a} \cdot \Delta\vec{k})]}$$

by use of the identity

$$\sum_{m=0}^{M-1} = \frac{1 - x^M}{1 - x}$$

It is

$$\sum_{m=0}^{m-1} x^m = \frac{1 - x^M}{1 - x}$$

In our formula for F

$$F = \sum_M \exp\left\{-im(\vec{a}\cdot\Delta\vec{k})\right\}$$

we can set the exp-function as the x, so we can use the sum-identity. This gives us for F the equation

$$F = \frac{1-\exp\left\{-iM(\vec{a}\cdot\Delta\vec{k})\right\}}{1-\exp\left\{-i(\vec{a}\Delta\vec{k})\right\}}$$

(b) The intensity of scattered radiation is proportional to $|F|^2$. Show that

$$|F|^2 = F * F = \frac{\sin^2\frac{1}{2}M(\vec{a}\cdot\Delta\vec{k})}{\sin^2\frac{1}{2}(\vec{a}\cdot\Delta\vec{k})}$$

We can use the following assumption:

$$\begin{aligned}
|F| &= F * F \\
&= \frac{1-\exp\left\{iM(\vec{a}\cdot\vec{\Delta}k)\right\}}{1-\exp\left\{i(\vec{a}\cdot\vec{\Delta}k)\right\}}\cdot\frac{1-\exp\left\{-iM(\vec{a}\cdot\vec{\Delta}k)\right\}}{1-\exp\left\{-i(\vec{a}\cdot\vec{\Delta}k)\right\}}. \\
&= \frac{2-\exp\left\{iM(\vec{a}\cdot\Delta\vec{k})\right\}-\exp\left\{-iM(\vec{a}\cdot\vec{\Delta}k)\right\}}{2-\exp\left\{i(\vec{a}\cdot\Delta\vec{k})\right\}-\exp\left\{-i(\vec{a}\cdot\vec{\Delta}k)\right\}} \\
&= \frac{1-\cos(M(\vec{a}\cdot\Delta\vec{k}))}{1-\cos(\vec{a}\cdot\Delta\vec{k})} \\
&= \frac{1-\cos\left(\frac{1}{2}M(\vec{a}\cdot\Delta\vec{k})\right)}{1-\cos\left(\frac{1}{2}(\vec{a}\cdot\Delta\vec{k})\right)} \\
&= \frac{1-\left[1-2\sin^2\left(\frac{1}{2}M(\vec{a}\cdot\vec{\Delta}k)\right)\right]}{1-\left[1-2\sin^2\left(\frac{1}{2}(\vec{a}\cdot\vec{\Delta}k)\right)\right]} \\
&= \frac{\sin^2\left(\frac{1}{2}M(\vec{a}\cdot\vec{\Delta}k)\right)}{\sin^2\left(\frac{1}{2}(\vec{a}\cdot\vec{\Delta}k)\right)}
\end{aligned}$$

(c) We know that a diffraction maximum appears when $\vec{a} \cdot \Delta\vec{k} = 2\pi h$, where h is the an integer. What's the result for $|F|^2$ if $\vec{a} \cdot \Delta\vec{k} = 2\pi h$?

If we set in $2\pi h$ for $\vec{a} \cdot \Delta\vec{k}$ we'll get for the function $|F|^2$

$$\begin{aligned} |F|^2 &= \frac{\sin^2\left(\frac{1}{2}M2\pi h\right)}{\sin^2\left(\frac{1}{2}2\pi h\right)} \\ &= \frac{\sin^2(M2\pi h)}{\sin^2(\pi h)} \end{aligned}$$

As the $\sin^2$-function has a zero every $2n\pi$-times, and since we know that h and M are integers, we can assume that $|F|^2$ is zero at the diffraction maximum.

(d) We change $\Delta\vec{k}$ slightly and define a quantity ϵ such that when $\vec{a} \cdot \Delta\vec{k} = 2\pi h + \epsilon$ is substituted into $\sin\frac{1}{2}M(\vec{a} \cdot \Delta\vec{k})$, the function $|F|^2$ has its first value of zero. Show that $\epsilon = \frac{2\pi}{M}$, which means that the width of the diffraction maximum is proportional to $\frac{1}{M}$ and can therefore be extremly narrow for macroscopic (i.e. large) values of M.

If we set in $2\pi h + \epsilon$ in the equation for $|F|^2$ we'll get:

$$|F|^2 = \frac{\sin^2\left(\frac{1}{2}M(2\pi h + \epsilon)\right)}{\sin^2\left(\frac{1}{2}(2\pi h + \epsilon)\right)}$$

Now we have to look at a diffraction maximum ($|F|^2$ has to be zero, so we consider only the nominator), we take the one at 2π, doing so it follows:

$$\begin{aligned} \sin^2\left(\frac{1}{2}M(2\pi h + \epsilon)\right) &= 0 \\ \Leftrightarrow \quad M(2\pi h + \epsilon) &= 2\pi \\ \Leftrightarrow \quad \epsilon &= \frac{2\pi}{M} - 2\pi h \end{aligned}$$

We now should only consider the first value of zero, this is equal to setting h to zero and it follows for ϵ:

$$\epsilon = \frac{2\pi}{M}$$

1.4 Distribution of lattice parameters

Assume that a given material contains regions with local lattice parameters a that differ from the overall average lattice parameter a_0 by the (varying) quantity $\delta a = a - a_0$. This distribution of local lattice parameter values leads to a broadening $\delta(2\theta)$ of Bragg peaks.

(a) With the help of the Bragg equation, show that

$$|\delta(2\Theta_0)| = 2\tan\Theta_0 \left|\frac{\delta a}{a_0}\right|$$

where $2\Theta_0$ denotes the position of a Bragg peak of a material with uniform lattice parameter a_0.

It is

$$2\frac{a_0}{\sqrt{h^2+k^2+l^2}}\sin\Theta_0 = \lambda$$

$$\Leftrightarrow \quad \sin(\Theta_0) = \frac{\lambda\sqrt{h^2+k^2+l^2}}{2a_0}$$

and

$$2\frac{\delta a}{\sqrt{h^2+k^2+l^2}}\sin\delta\Theta_0 = \lambda$$

$$\Leftrightarrow \quad \sin(\delta\Theta_0) = \frac{\lambda\sqrt{h^2+k^2+l^2}}{2\delta a}$$

Dividing both equation leads us to:

$$\frac{\sin(\delta\Theta)}{\sin(\Theta_0)} = \frac{a_0}{\delta a}$$

Now by applying some addition theorems and some magic, it turns out that

$$2\tan(\Theta_0)\left|\frac{\delta a}{a_0}\right| = |\delta(2\Theta_0)|$$

(b) The above equation for $|\delta(2\Theta)|$ takes on a simpler form when a change of variable is performed from Θ to $s = 2(\sin\Theta)/\lambda$. Show that in this case

$$|\delta s| = s_0\left|\frac{\delta a}{a_0}\right|$$

If we put in $s = 2(\sin(\Theta)/\lambda$ into the given formula $|\delta s| = s_0|\delta a/a_0|$ then we get:

$$\left|\frac{\delta 2\sin(\Theta)}{\lambda}\right| = \frac{2\sin(\Theta_0)}{\lambda}\left|\frac{\delta a}{a_0}\right|$$
$$\Leftrightarrow \quad \frac{sin\delta\Theta}{\sin\Theta} = \left|\frac{\delta a}{a_0}\right|$$

This is the same result as in task 1.4a.

2 Carrying out the Experiment

In the experiment we performed different XRD-measurements with a $Ni_{0.98}Zr_{0.2}$-sample, one at room temperature (25°), five measurements at 750°and one at 300°. The goal was to find out the lattice parameter, to say something about the thermal expansion coefficient and to do different calculations on the broadening of the peaks, to get informations about the grain size and the microstrain.

2.1 Lattice parameter of $Ni_{0.98}Zr_{0.02}$

Principle

The first task was to find out the lattice parameter of $Ni_{0.98}Zr_{0.02}$ at room temperature, at 750°and at 300°. Because it is not possible to mount the sample correctly relative to the x-ray tube and detector, we have to correct the error from this misplacement. We know that the sample misplacement can be written as

$$a = a_0 - \left(\frac{a_0\tilde{d}}{r}\right)\frac{\cos^2\Theta}{\sin\Theta}$$

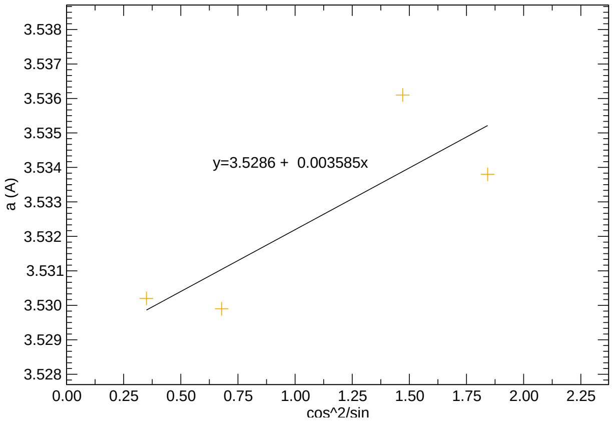
with a being the measured value for the lattice parameter, a_0 the real value of the lattice parameter, $\frac{a_0\tilde{d}}{r}$ the term for the misplacement ($\tilde{d}$ is the misplacement and r the radius of the measuring circle) as well as θ being the measured peak angle.

By plotting the measured value a against the angle-term $\frac{\cos^2\Theta}{\sin\Theta}$ we'll get the real value of a at the intercept point with the y-axis (because here we have $a = a_0 + 0 = a_0$).

Room-Temperature Measurement

The values we got for the room-temperature-measurement can be seen in the table below.

Plotting this values gives us an intercept point at 3.5286, this is a_0 of $Ni_{0.98}Zr_{0.2}$ at room temperature (see Fig. 2).

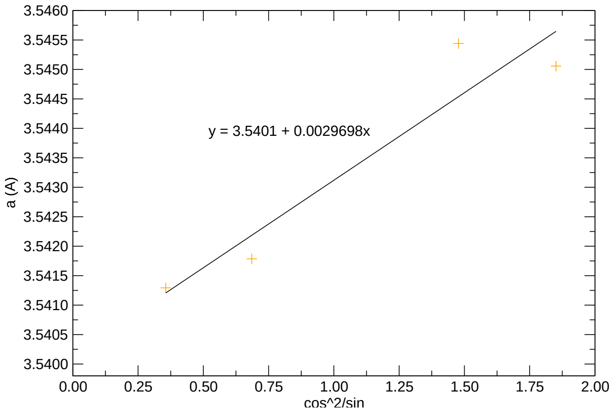


hkl	$2\Theta/^\circ$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	52.0075	2.04025	3.5338	1.8424
200	60.7849	1.76807	3.5361	1.4707
220	91.5715	1.24802	3.5299	0.6785
113	114.3616	1.06440	2.5302	0.3495

Fig. 2: Determination of a_0 at room temperature (25°)

300° Measurement

The same measurement we performed now at 300°. The values and the plot we got can be seen below. The value resulting for a_0 at 300° is 3.5401.



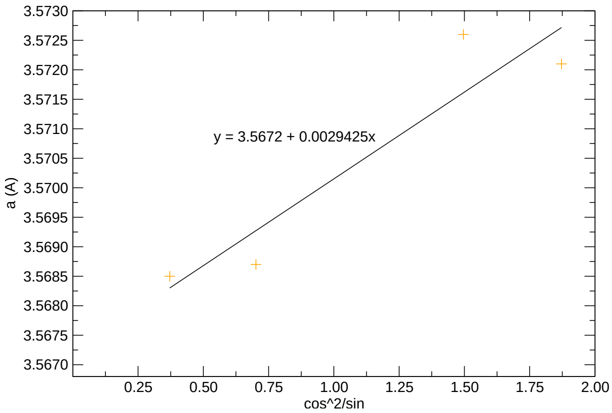
hkl	$2\Theta/^\circ$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.8302	2.04674	3.5451	1.8511
200	60.6090	1.77272	3.5454	1.4772
220	91.1784	1.25221	3.5418	0.6856
113	113.8085	1.06774	3.5413	0.3559

Fig. 3: Determination of a_0 at 300°

750° Measurement

At 750° we performed 6 measurements, because we need the values at this temperature for further tasks. That means that we have six measurements for the determination of a_0. So we plotted every measurement values and built in the end an average over the a_0-values.

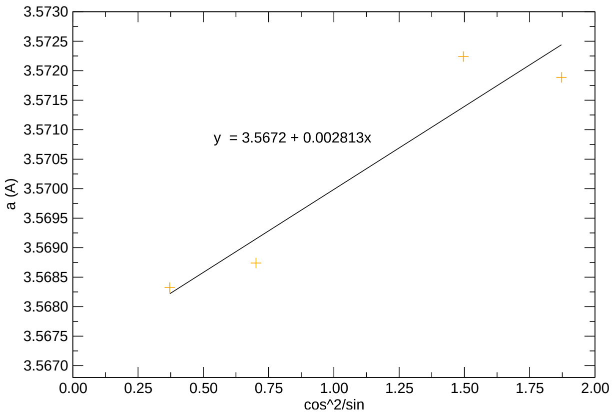
The values we have measured as well as the plots can be seen below.



hkl	$2\Theta/°$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4095	2.06234	3.5721	1.8718
200	60.1008	1.78629	3.5726	1.4962
220	90.3008	1.26171	3.5687	0.7016
113	112.4787	1.07595	3.5685	0.3715

Fig. 4: Determination of a_0 at 750°, First Measurement

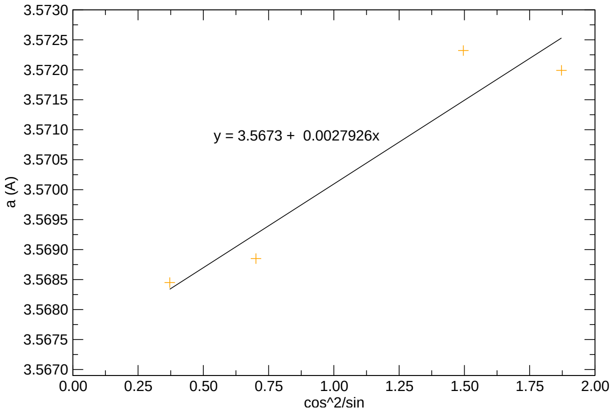
The first measurement gives us a value of 3.5672 for a_0.



hkl	$2\Theta/°$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4123	2.06223	3.5719	1.8717
200	60.1070	1.78612	3.5722	1.4960
220	90.2980	1.26174	3.5687	0.7016
113	112.4872	1.07589	3.5683	0.3714

Fig. 5: Determination of a_0 at 750°, Second Measurement

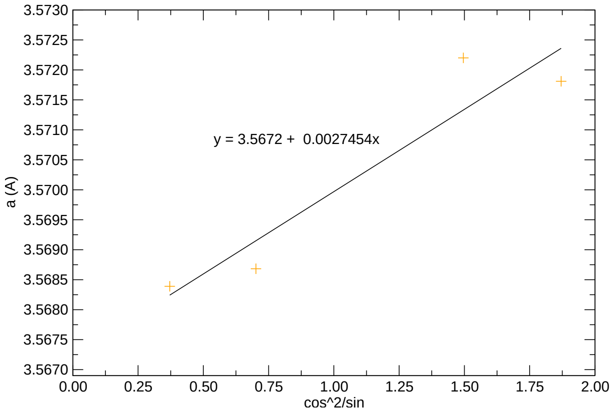
For the second measurement we get a value for a_0 of 3.5672.



hkl	$2\Theta/°$	$d/Å$	$a/Å$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4107	2.06229	3.5720	1.8718
200	60.1056	1.78616	3.5723	1.4960
220	90.2945	1.26178	3.5689	0.7017
113	112.4813	1.07593	3.5685	0.3714

Fig. 6: Determination of a_0 at 750°, Third Measurement

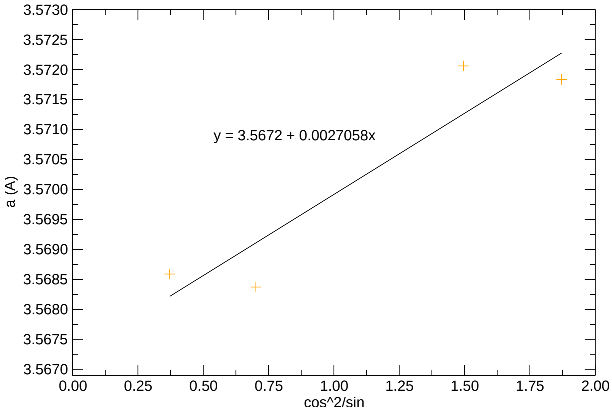
In this measurement we got a value of 3.5673 for a_0.



hkl	$2\Theta/°$	$d/Å$	$a/Å$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4133	2.06219	3.5718	1.8707
200	60.1079	1.78610	3.5722	1.4959
220	90.3003	1.26172	3.5687	0.7016
113	112.4849	1.07591	3.5684	0.3714

Fig. 7: Determination of a_0 at 750°, Fourth Measurement

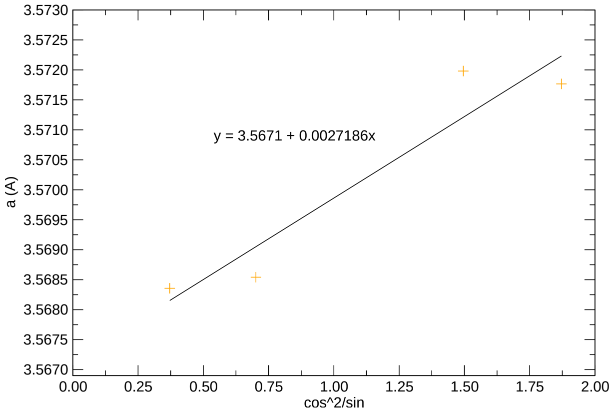
The value for a_0 in the fourth measurement again is 3.5672.



hkl	$2\Theta/°$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4126	2.06222	3.5718	1.8717
200	60.1104	1.78603	3.5721	1.4959
220	90.3104	1.26161	3.5684	0.7014
113	112.4741	1.07597	3.5686	0.3715

Fig. 8: Determination of a_0 at 750°, Fifth Measurement

The fifth measurement again resulted in a value for a_0 of 3.5672.



hkl	$2\Theta/°$	$d/\text{Å}$	$a/\text{Å}$	$\frac{\cos^2\Theta}{\sin\Theta}$
111	51.4142	2.06216	3.5718	1.8716
200	60.1121	1.78599	3.5720	1.4958
220	90.3045	1.26167	3.5685	0.7015
113	112.4864	1.07590	3.5684	0.3714

Fig. 9: Determination of a_0 at 750°, Sixth Measurement

In the last measurement a_0 turned out to be 3.5671. That means in average a_0 at 750°is 3.5672.

We can see, that in all measurement we nearly got the same value for a_0.

2.2 Determination of the thermal expansion coefficient of $Ni_{0.98}Zr_{0.2}$

From the measured values above we can calculate the thermal expansion coefficient ($\Delta a/°K$) for $Ni_{0.98}Zr_{0.2}$.

Δa/Å	ΔT/°K	$\Delta a/\Delta T$/Å/K
0.0115	275	$4.1818 \cdot 10^{-5}$
0.02276	450	$5.0577 \cdot 10^{-5}$
0.03426	725	$4.7255 \cdot 10^{-5}$

That means, the average thermal expansion is $4.6550 \cdot 10^{-5}$Å/K.

2.3 Broadening: Grain Growth and Microstrain

Principle

In the ideal case we would expect δ-impulses for the bragg peaks. In real measurements we can't get δ-impulses, what we get instead are broadened impulses, which can be assumed as having lorentzian or gaussian shape. Due to the fact that this broadening occurs due to different mechanisms, we are able to get information concerning e.g. the grain size and growth as well as about the microstrain.

One of these mechanism is instrumental broadening, that means broadening that comes from instrumental effects e.g. from the fact, that a x-ray diffractometer has a limited resolution and that the x-rays are slightly dispersive as well as from the fact that there is x-ray scattering at various depth of the sample.

To get an idea how big this effect is and to correct the other broadened peaks by this factor we first make a "calibration" measurement with LaB_6 which has very big grains and nearly no microstrain, that means the broadening of the bragg peaks in this material is only due to instrumental broadening, therefore we can use the LaB_6-peaks to correct our sample broadened bragg peaks.

The values we got for this first measurement are a broadening $\delta(2\Theta)_{\text{inst}}$ of 0.00971, which gives us the transformed variable δs_{inst} as $\delta s_{\text{inst}} = 9.4678 \cdot 10^{-5}$ and δs^2_{inst} as $\delta s^2_{\text{inst}} = 8.9636963 \cdot 10^{-9}$. The transformed variable s is defined as $s = 2(sin\theta)/\lambda$ and e is an abbrevation for $e = \frac{1}{2}|\delta a/a_0|$.

Beside the instrumental broadening there are two other broadening mechanisms: the microstrain broadening and the size-broadening. The first broadening is due to microstrain between different layers of the sample, the latter results from the finite size of the sample.

By doing more than one measurement we are able to determine the size of this two broadening effects and so we can get the values for the chrystallite size <$\mathrm{D_{xray}}$> and the microstrain e by plotting δs_{corr} over s_0 if we assume that the broadenings are lorentzian shapedor by plotting δs^2_{corr} over s_0^2 if we assume that the broadenings are gaussian shaped, where δ_{corr} is the measured broadening corrected by the instrumental broadening.

From the plots we can determine the microstrain e from the slope and the grain size $<D_{\text{xray}}>$ from the intercept with the δs_{corr}-axis.

Determination of crystallite size <$\mathrm{D_{xray}}$> and microstrain e at 25°

With the values from every measurement we did a plot under the assumption that all broadenings are lorentzian shaped and another one under the assumption, that alle broadenings are gaussian shaped. In fig. 10 and fig. 11 you can see both plots and in the tables aside the values we measured and calculated.

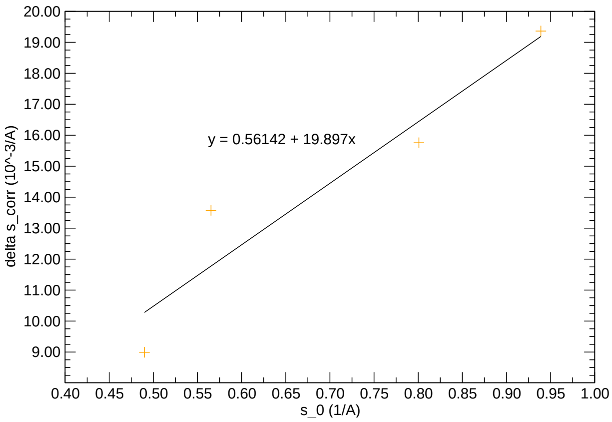


Fig. 10: 25°: Lorentzian Plot

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0/\frac{1}{Å}$	$(\delta s)_{\text{corr}}/\frac{1}{Å}$
111	26.0038	0.4629	0.4899	$8.9332{\cdot}10^{-3}$
200	30.3925	0.7010	0.5653	$13.575{\cdot}10^{-3}$
220	45.7858	0.8129	0.8008	$15.758{\cdot}10^{-3}$
113	57.1808	0.9979	0.9389	$19.363{\cdot}10^{-3}$

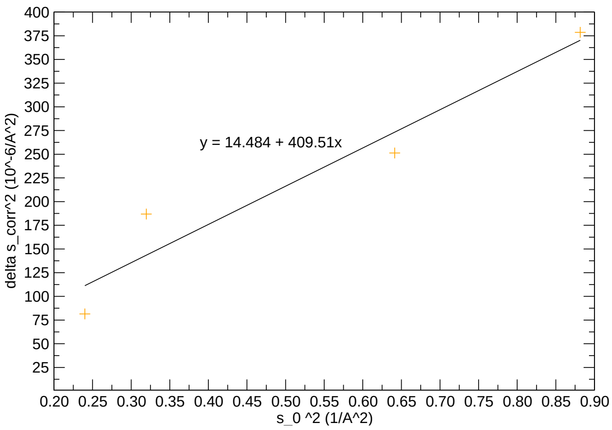


Fig. 11: 25°: Gaussian Plot

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{\text{corr}^2}/\frac{1}{Å^2}$
111	26.0038	0.4629	0.2399	$81.492{\cdot}10^{-6}$
200	30.3925	0.7010	0.3195	$186.85{\cdot}10^{-6}$
220	45.7858	0.8129	0.6413	$251.31{\cdot}10^{-6}$
113	57.1808	0.9979	0.8817	$378.86{\cdot}10^{-6}$

From the slope of the lorentzian plot we can calculate the microstrain by the formula slope $= e/2$. So it is: $e = 0.994\%$. For the gaussion plot e can be calulated as $e = \sqrt{\text{slope}/4}$, so e is 1.01181%. Building the average we get for the microstrain in the 25° measurement the value of $e = 1.00033\%$.

The grainsize $<D_{\text{xray}}>$ can be calculated by $<D_{\text{xray}}>= 1.2/(\text{y} - \text{intercept})$ for the lorentzian plot and by $<D_{\text{xray}}>= \sqrt{1.2/(\text{y} - \text{intercept})}$ for the gaussian plot. Doing so, we get $<D_{\text{xray}}>= 21.37nm$ and $<D_{\text{xray}}>= 28.78nm$, the average is $<D_{\text{xray}}>= 25.07nm$.

Determination of crystallite size $<D_{\text{xray}}>$ and microstrain e at 300°

We repeated the measurement at 300° - in fig. 12 and fig. 13 you can see the resulting plots and values.

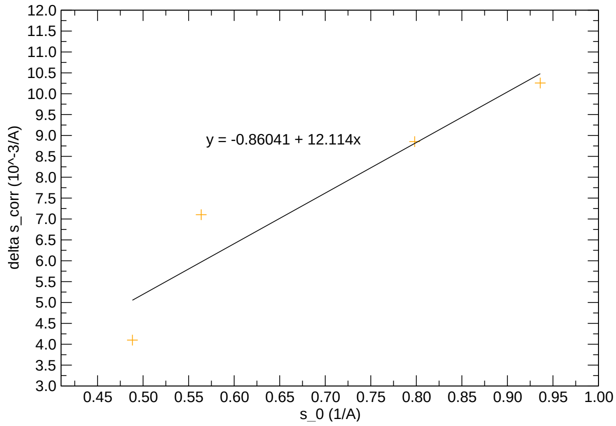


Fig. 12: 300°: Lorentzian Plot

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0/\frac{1}{Å}$	$(\delta s)_{\text{corr}}/\frac{1}{Å}$
111	25.9151	0.2151	0.4883	$4.0989{\cdot}10^{-3}$
200	30.3045	0.3691	0.5638	$7.1031{\cdot}10^{-3}$
220	45.5892	0.4589	0.7981	$8.8532{\cdot}10^{-3}$
113	56.9043	0.5309	0.9360	$10.257{\cdot}10^{-3}$

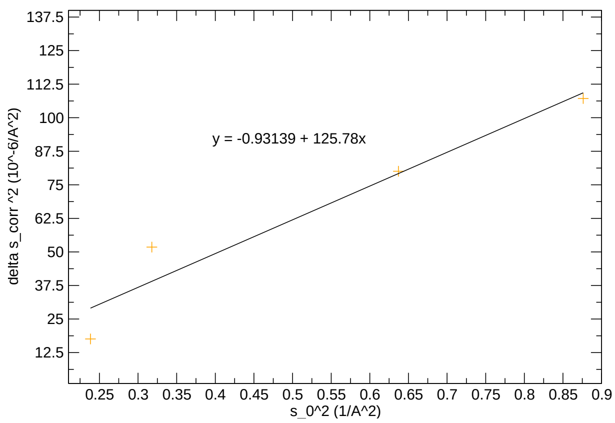


Fig. 13: 300°: Gaussian Plot

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{\text{corr}^2}/\frac{1}{Å^2}$
111	25.9151	0.2151	0.2384	$17.577{\cdot}10^{-6}$
200	30.3045	0.3691	0.3179	$51.798{\cdot}10^{-6}$
220	45.5892	0.4589	0.6370	$80.055{\cdot}10^{-6}$
113	56.9043	0.5309	0.8762	$107.15{\cdot}10^{-6}$

For the microstrain at 300° follows with the formula mentioned above as an average $e = 0.58323\%$. We aren't able to calculate a value for the grain size $< D_{\text{x-ray}} >$ for the 300° measurement, because we have a negative intercept point!

Determination of crystallite size $<D_{\text{xray}}>$ and microstrain e at 750°

For 750° we performed the measurement six times. Unfortunately it turned out that some values are not really useful, it is not quite clear why we got those bad results.

Below you can see the plots and value tables for every measurement (fig. 14 to fig. 25).

As can be seen very easily we can't determine the value for $<D_{\text{x-ray}}>$ for every measurement, because we have negative intercept points.

In the following table we have listed all values we could calculate:

	$< D_{\text{x-ray,Lorentz}} >$ in nm	e_{Lorentz} in %	$<D_{\text{x-ray,Gauss}}>$ in nm	e_{Gauss} in %
1	30.384	0.202	18.710	0.267
2	-	0.586	-	0.558
3	169.861	0.488	28.058	0.475
4	2293.7	0.544	37.6	0.519
5	-	0.609	-	0.559
6	145.48	0.437	32.747	0.446

	$<D_{\text{x-ray}}>$ in nm (aver.)	e in % (aver.)
1	24.5	0.235
2	-	0.572
3	-	0.482
4	-	0.532
5	-	0.585
6	-	0.441

If we don't care about the values from measurements two, five and six we can see, that the grain size $<D_{\text{x-ray}}>$ is getting bigger by the time as well as the microstrain is decreasing. This is what we would expect.

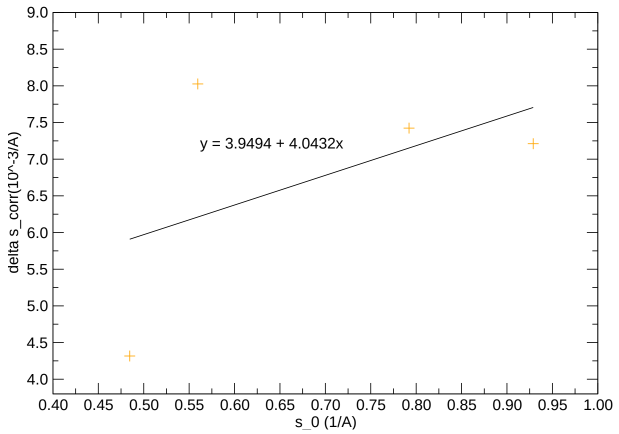


Fig. 14: 750°: Lorentzian Plot (first measurement)

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0/\frac{1}{\text{Å}}$	$(\delta s)_{\text{corr}}/\frac{1}{\text{Å}}$
111	25.7048	0.2262	0.4846	$4.3164 \cdot 10^{-3}$
200	30.0504	0.4165	0.5595	$8.0264 \cdot 10^{-3}$
220	45.1504	0.3856	0.7921	$7.4238 \cdot 10^{-3}$
113	56.2394	0.3747	0.9289	$7.2113 \cdot 10^{-3}$

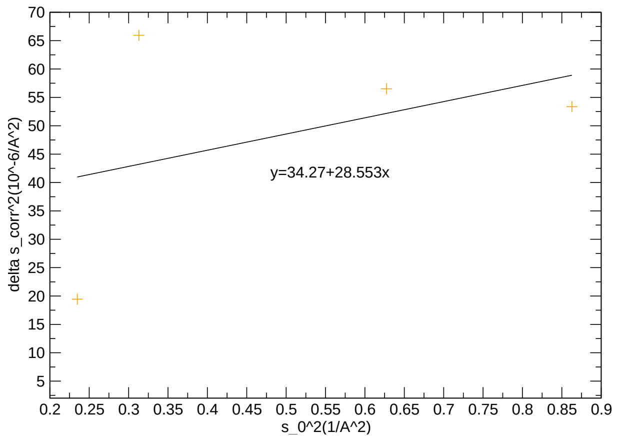


Fig. 15: 750°: Gaussian Plot (first measurement)

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0^2/\frac{1}{\text{Å}^2}$	$(\delta s)_{\text{corr}^2}/\frac{1}{\text{Å}^2}$
111	25.7048	0.2262	0.2349	$19.449 \cdot 10^{-6}$
200	30.0504	0.4165	0.3131	$65.943 \cdot 10^{-6}$
220	45.1504	0.3856	0.6275	$56.519 \cdot 10^{-6}$
113	56.2394	0.3747	0.8629	$53.368 \cdot 10^{-6}$

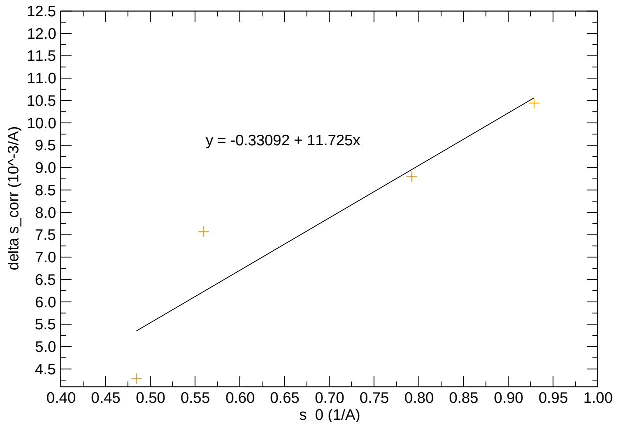
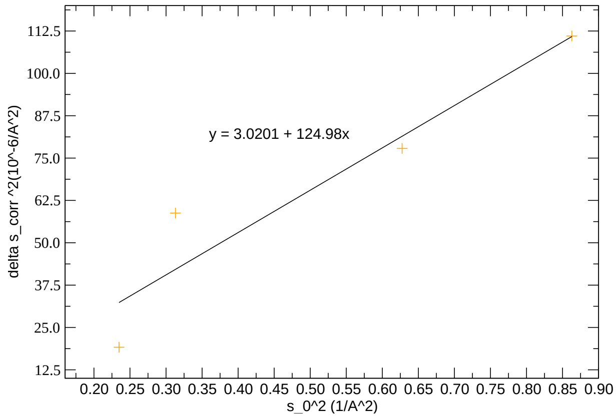


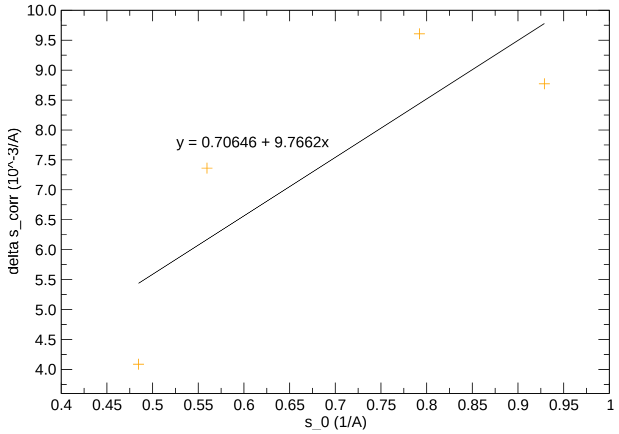
Fig. 16: 750°: Lorentzian Plot (second measurement)

hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0/\frac{1}{\text{Å}}$	$(\delta s)_{\text{corr}}/\frac{1}{\text{Å}}$
111	25.7062	0.2246	0.4846	$4.2842 \cdot 10^{-3}$
200	30.0535	0.3931	0.5596	$7.5720 \cdot 10^{-3}$
220	45.1490	0.4525	0.7921	$8.7294 \cdot 10^{-3}$
113	56.2436	0.5404	0.9289	$10.443 \cdot 10^{-3}$



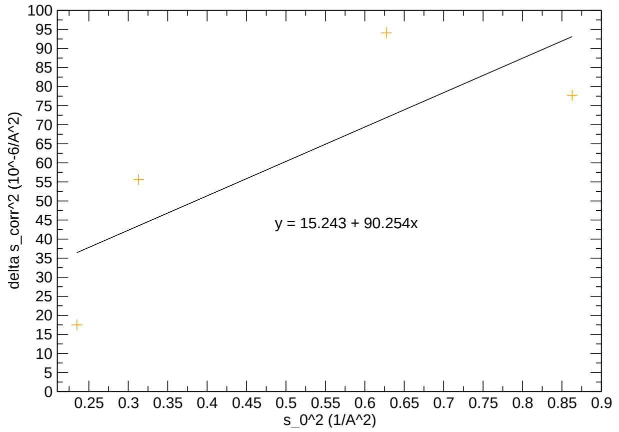
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{corr^2}/\frac{1}{Å^2}$
111	25.7062	0.2246	0.2348	$19.165 \cdot 10^{-6}$
200	30.0535	0.3931	0.3131	$58.769 \cdot 10^{-6}$
220	45.1490	0.4525	0.6274	$77.856 \cdot 10^{-6}$
113	56.2436	0.5404	0.8629	$111.04 \cdot 10^{-6}$

Fig. 17: 750°: Gaussian Plot (second measurement)



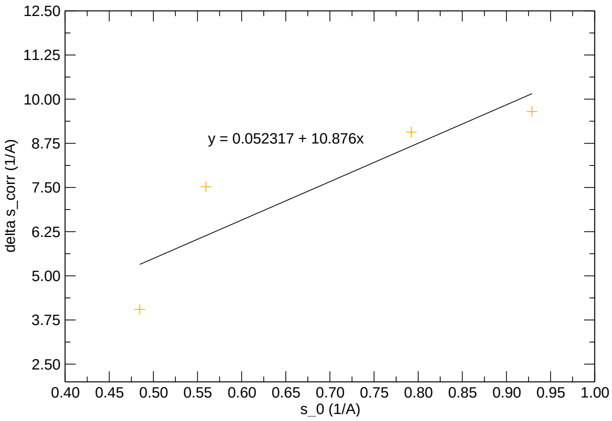
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0/\frac{1}{Å}$	$(\delta s)_{corr}/\frac{1}{Å}$
111	25.7054	0.2146	0.4846	$4.0892 \cdot 10^{-3}$
200	30.0528	0.3825	0.5596	$7.3633 \cdot 10^{-3}$
220	45.1473	0.4975	0.7921	$9.6069 \cdot 10^{-3}$
113	56.2407	0.4547	0.9289	$8.7713 \cdot 10^{-3}$

Fig. 18: 750°: Lorentzian Plot (third measurement)



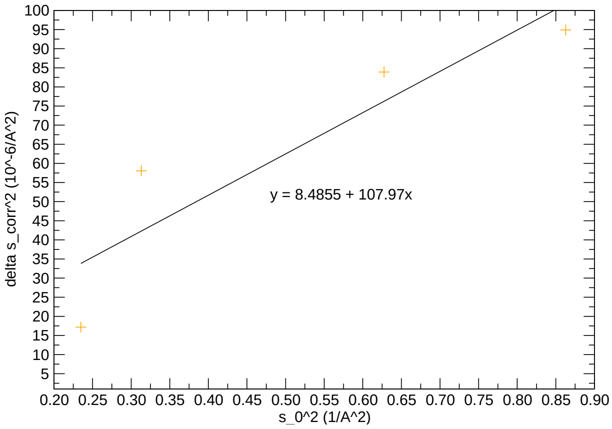
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{corr^2}/\frac{1}{Å^2}$
111	25.7054	0.2146	0.2349	$17.496 \cdot 10^{-6}$
200	30.0528	0.3825	0.3131	$55.614 \cdot 10^{-6}$
220	45.1473	0.4975	0.6274	$94.111 \cdot 10^{-6}$
113	56.2407	0.4547	0.8629	$77.709 \cdot 10^{-6}$

Fig. 19: 750°: Gaussian Plot (third measurement)



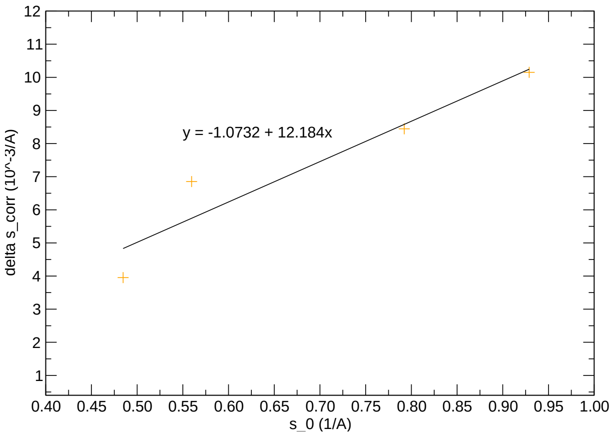
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0/\frac{1}{Å}$	$(\delta s)_{corr}/\frac{1}{Å}$
111	25.7066	0.2126	0.4847	$4.0512{\cdot}10^{-3}$
200	30.0539	0.3908	0.5596	$7.5120{\cdot}10^{-3}$
220	45.1501	0.4697	0.7921	$9.0647{\cdot}10^{-3}$
113	56.2425	0.4996	0.9289	$9.6478{\cdot}10^{-3}$

Fig. 20: 750°: Lorentzian Plot (fourth measurement)



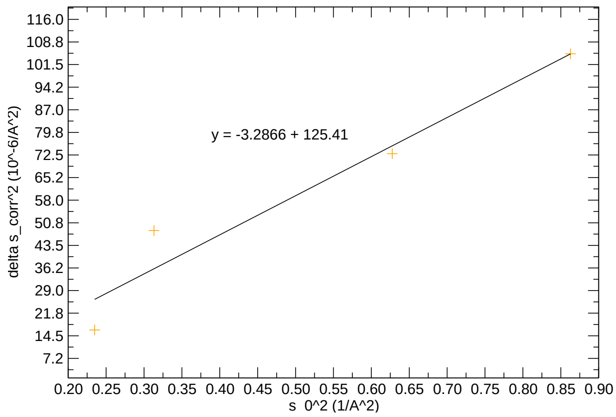
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0/\frac{1}{Å}$	$(\delta s)_{corr}/\frac{1}{Å}$
111	25.7066	0.2126	0.2349	$17.179{\cdot}10^{-6}$
200	30.0539	0.3908	0.3131	$58.054{\cdot}10^{-6}$
220	45.1501	0.4697	0.6274	$83.886{\cdot}10^{-6}$
113	56.2425	0.4996	0.8629	$94.908{\cdot}10^{-6}$

Fig. 21: 750°: Gaussian Plot (fourth measurement)



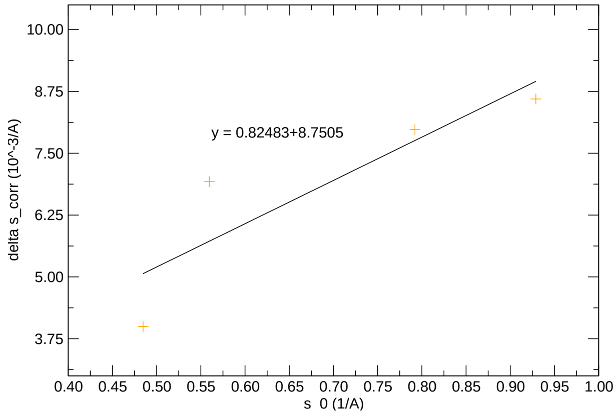
hkl	$\Theta_0/^\circ$	$\delta\Theta/^\circ$	$s_0/\frac{1}{Å}$	$(\delta s)_{corr}/\frac{1}{Å}$
111	25.7063	0.2076	0.4846	$3.9527{\cdot}10^{-3}$
200	30.0552	0.2563	0.5596	$6.8525{\cdot}10^{-3}$
220	45.1552	0.4379	0.7922	$8.4446{\cdot}10^{-3}$
113	56.2371	0.5254	0.9288	$10.151{\cdot}10^{-3}$

Fig. 22: 750°: Laurentzian Plot (fifth measurement)



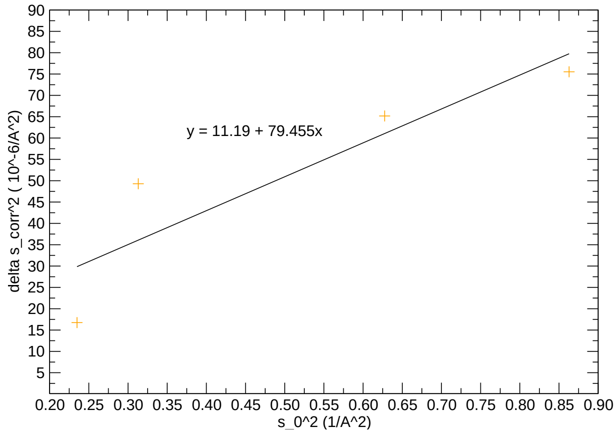
hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{corr^2}/\frac{1}{Å^2}$
111	25.7063	0.2076	0.2349	$16.372\cdot10^{-6}$
200	30.0552	0.2563	0.3131	$48.254\cdot10^{-6}$
220	45.1552	0.4379	0.6276	$72.911\cdot10^{-6}$
113	56.2371	0.5254	0.8628	$104.96\cdot10^{-6}$

Fig. 23: 750°: Gaussian Plot (fifth measurement)



hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0/\frac{1}{Å}$	$(\delta s)_{corr}/\frac{1}{Å}$
111	25.7071	0.2098	0.4847	$3.9966\cdot10^{-3}$
200	30.0561	0.3600	0.5596	$6.9256\cdot10^{-3}$
220	45.1526	0.4140	0.7922	$7.9786\cdot10^{-3}$
113	56.2432	0.4457	0.9289	$8.5968\cdot10^{-3}$

Fig. 24: 750°: Laurentzian Plot (sixth measurement)



hkl	$\Theta_0/°$	$\delta\Theta/°$	$s_0^2/\frac{1}{Å^2}$	$(\delta s)_{corr^2}/\frac{1}{Å^2}$
111	25.7071	0.2098	0.2349	$16.729\cdot10^{-6}$
200	30.0561	0.3600	0.3132	$49.275\cdot10^{-6}$
220	45.1526	0.4140	0.6275	$65.169\cdot10^{-6}$
113	56.2432	0.4457	0.8629	$75.532\cdot10^{-6}$

Fig. 25: 750°: Gaussian Plot (sixth measurement)

Evaluation of crystallite size $<\mathrm{D_{xray}}>$ over the time t

The last task was to plot the grain sizes $<D_{\text{x-ray}}>$ which we got in the task before over the time.

Doing so, we get the following plot (fig. 26):

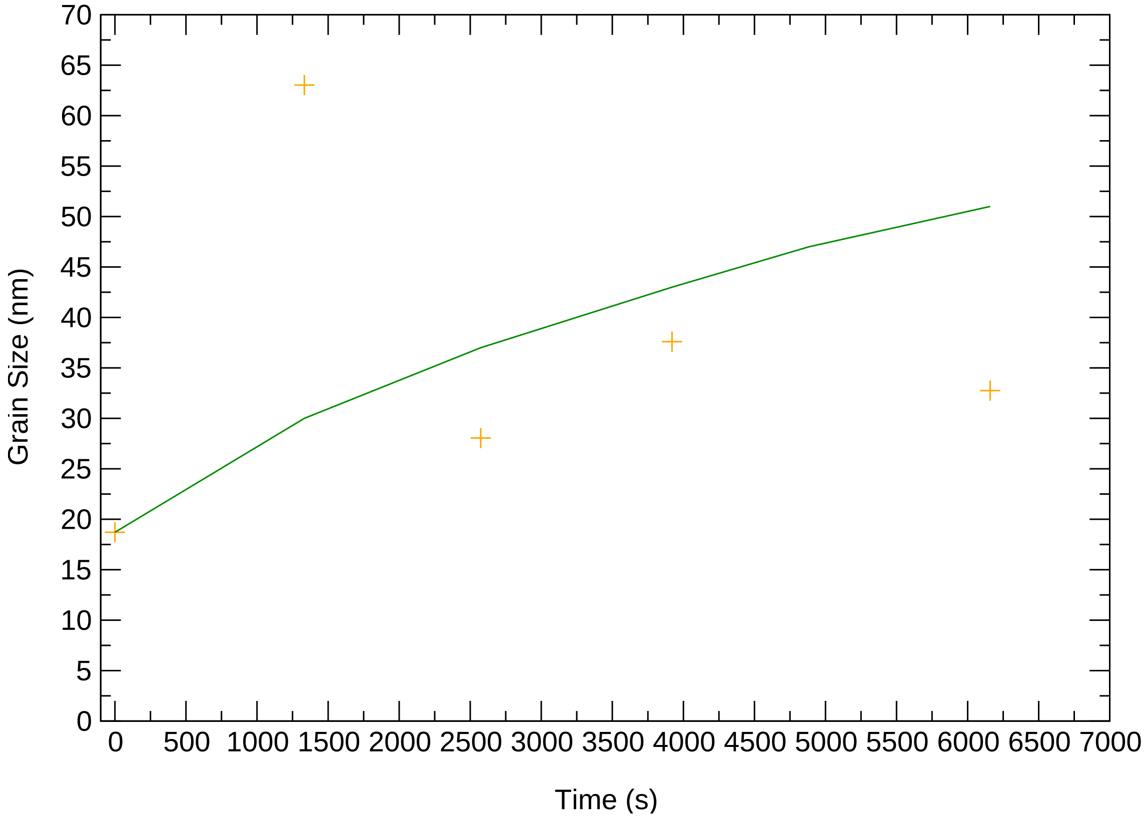


Fig. 26: Grain Size over time

The orange crosses are the values we measured, the green line is the function we would expect due to the formula $<D_{\text{x-ray}}(t)>^2 = kt - <D_{\text{x-ray}}(0)>^2$, with being $<D_{\text{x-ray}}(0)>$ the grain size at the beginning. We can see again, that some values are very far away from the desired curve. The values fitting best are the values of measurement one, three and four.