Precision investigation of modern crystalline materials by neutron diffraction method

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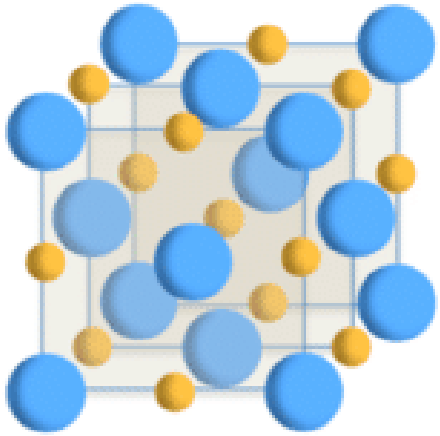
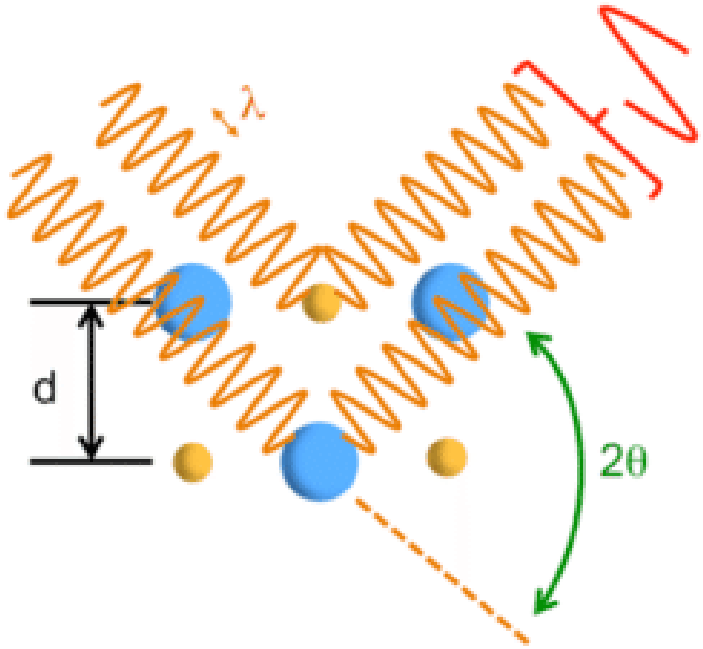
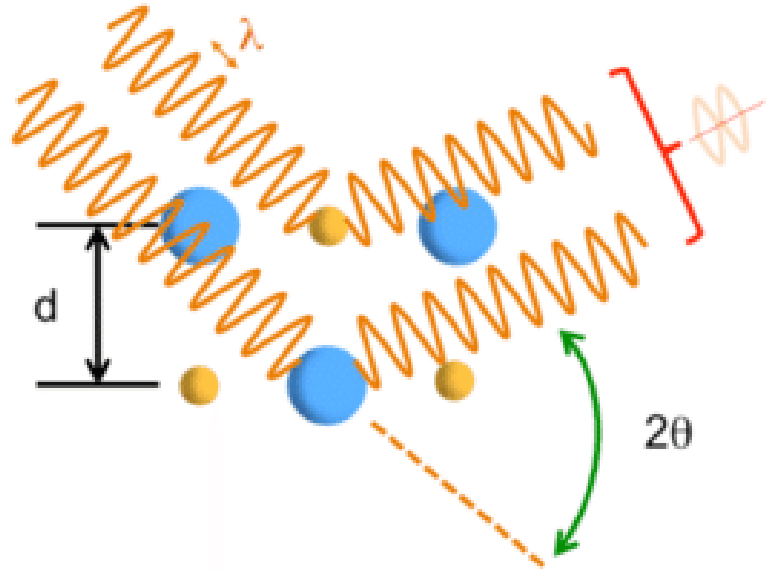
Diffraction in crystals

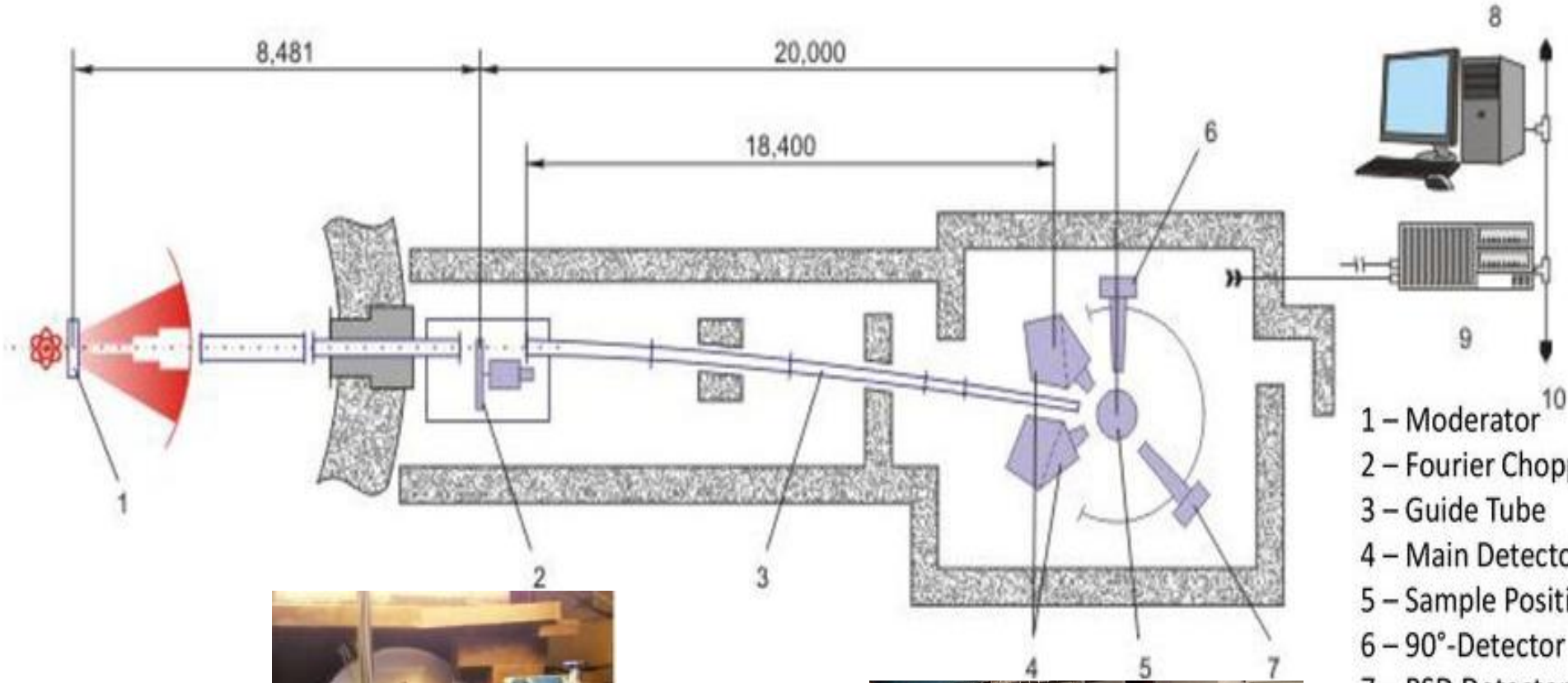




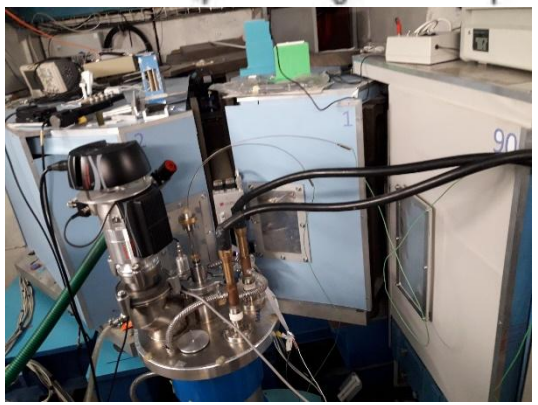


$n\lambda = 2d \operatorname{sen}\theta$ (Bragg´s Law)

High Resolution Fourier Difractometer

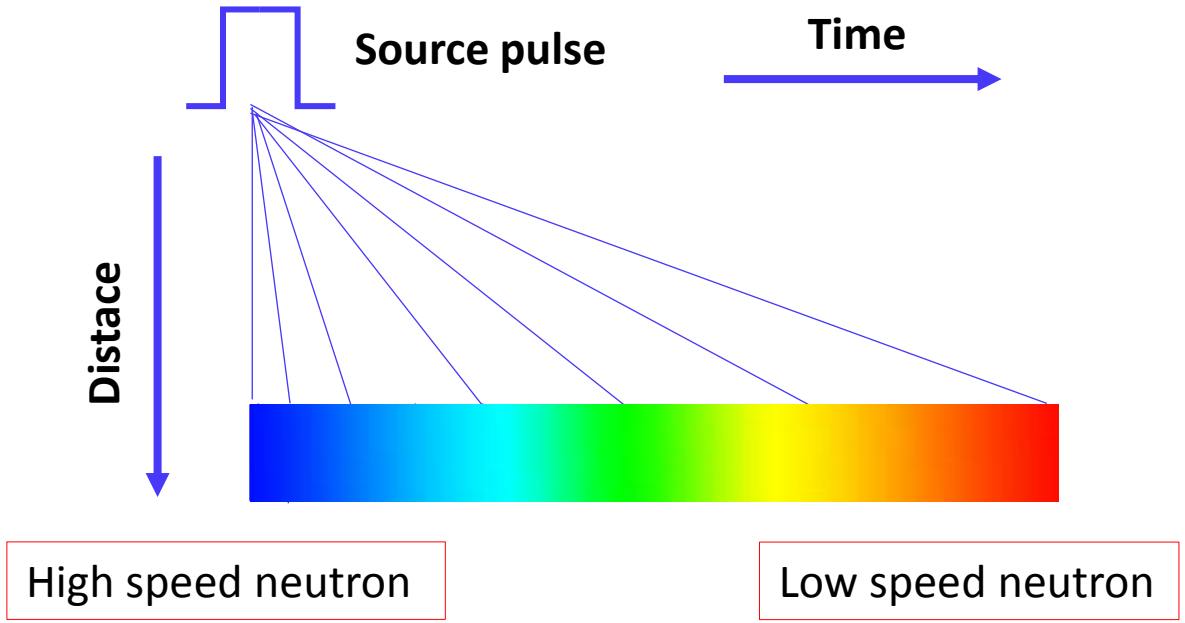


1 – Moderator
2 – Fourier Chopper
3 – Guide Tube
4 – Main Detector
5 – Sample Position
6 – 90°-Detector
7 – PSD Detector
8 –Control and Operative
9 – Data Acquisition
10 –Ethernet Data Transfer



High Resolution Fourier Difractometer

Time-of-flight techinque at pulsed neutron source

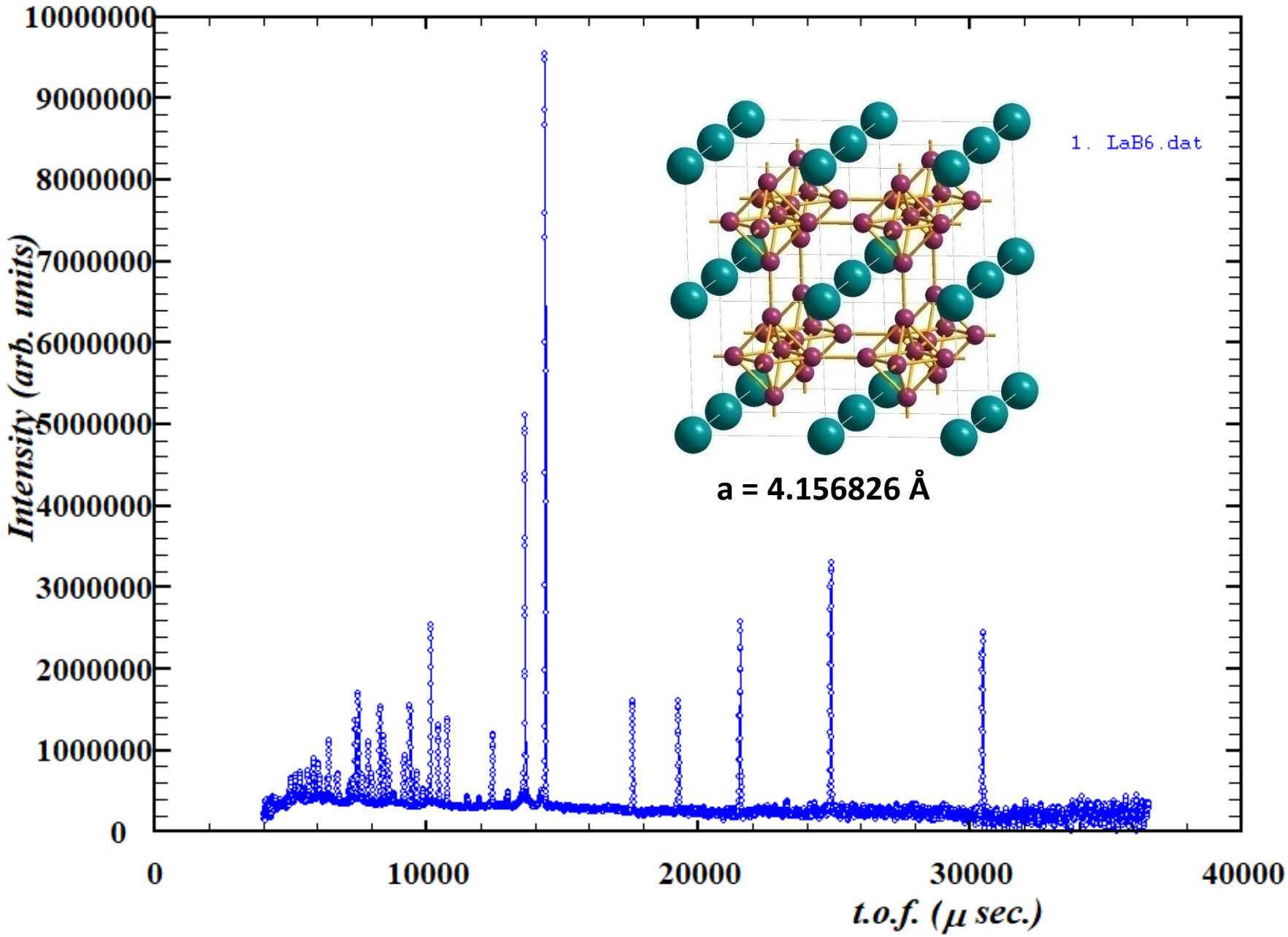


- ✓ Termal neutron travel a distance **L**
- ✓ At speed **v=(h/m λ)**
- ✓ Arrive to the detector at time **t=L/v**

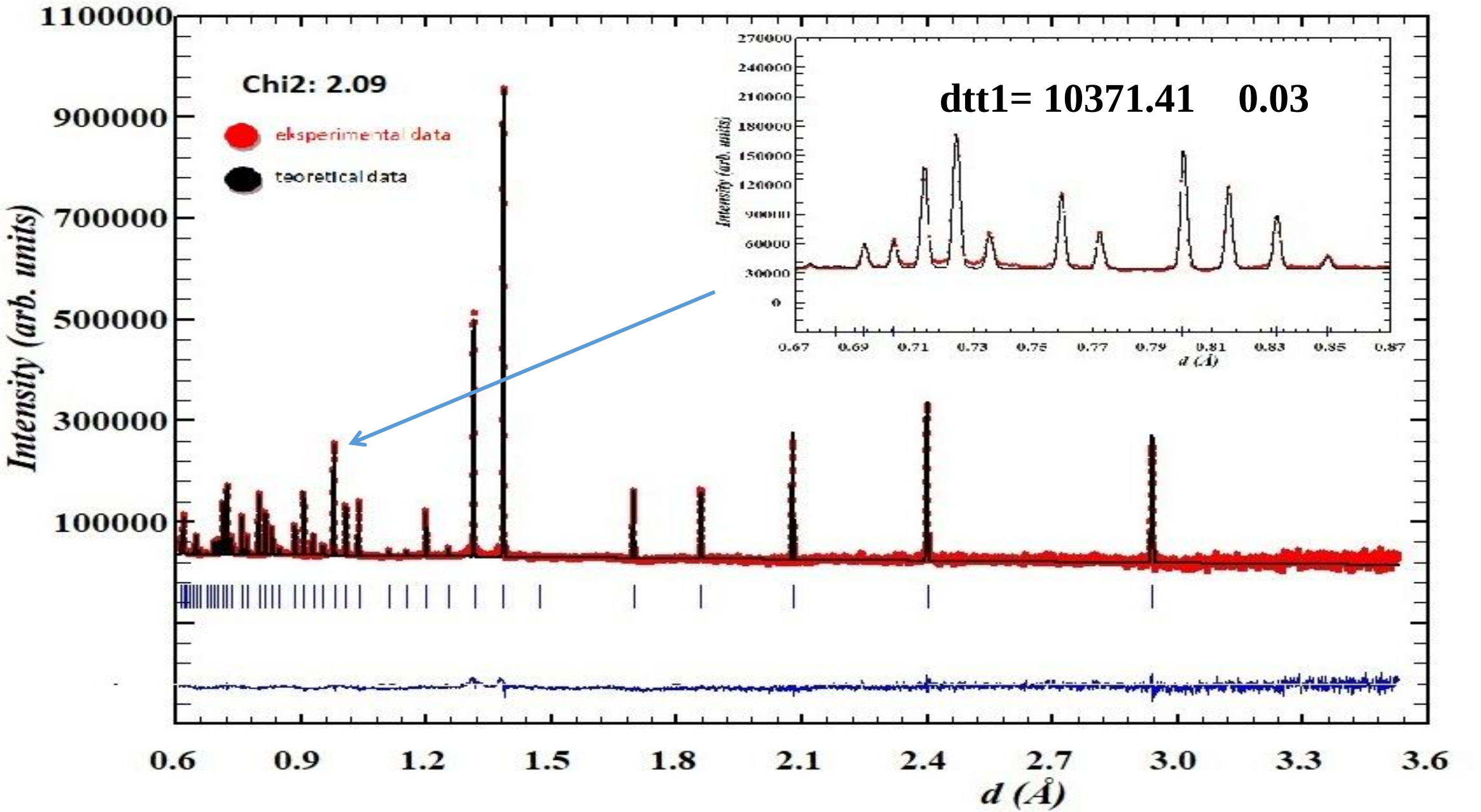
Wavelength is

$$\lambda = (h/m)t/L$$

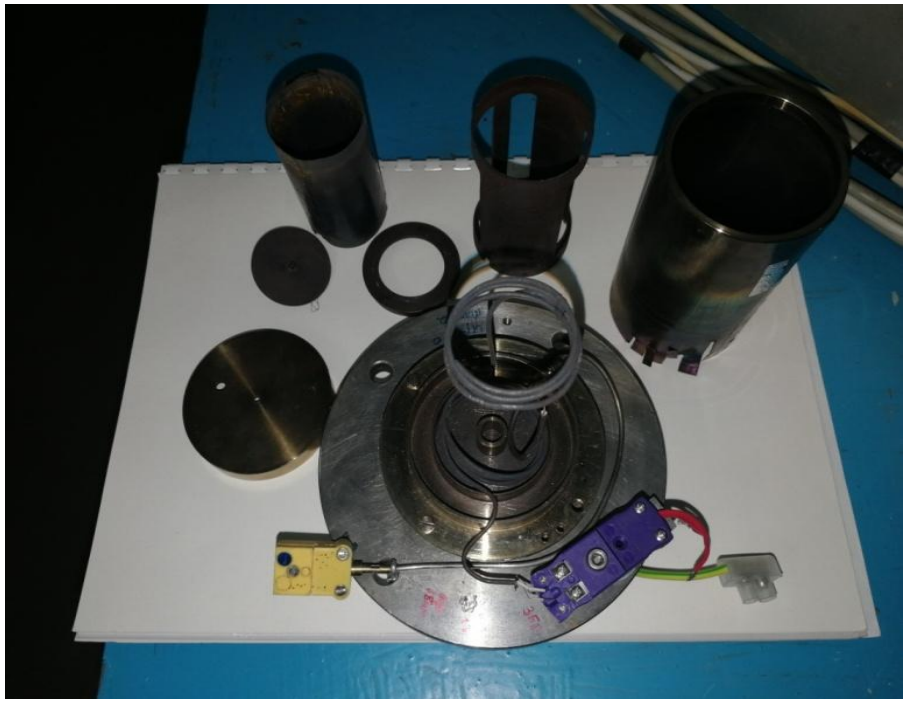
$$n\lambda = 2d\ \text{sen}\theta \quad \text{(Bragg´s Law)}$$


10000000
9000000
8000000
7000000
6000000
5000000
4000000
3000000
2000000
1000000
0
Intensity (arb. units)
0
10000
20000
30000
40000
t.o.f. (μ sec.)
1. LaB6.dat
a = 4.156826 Å

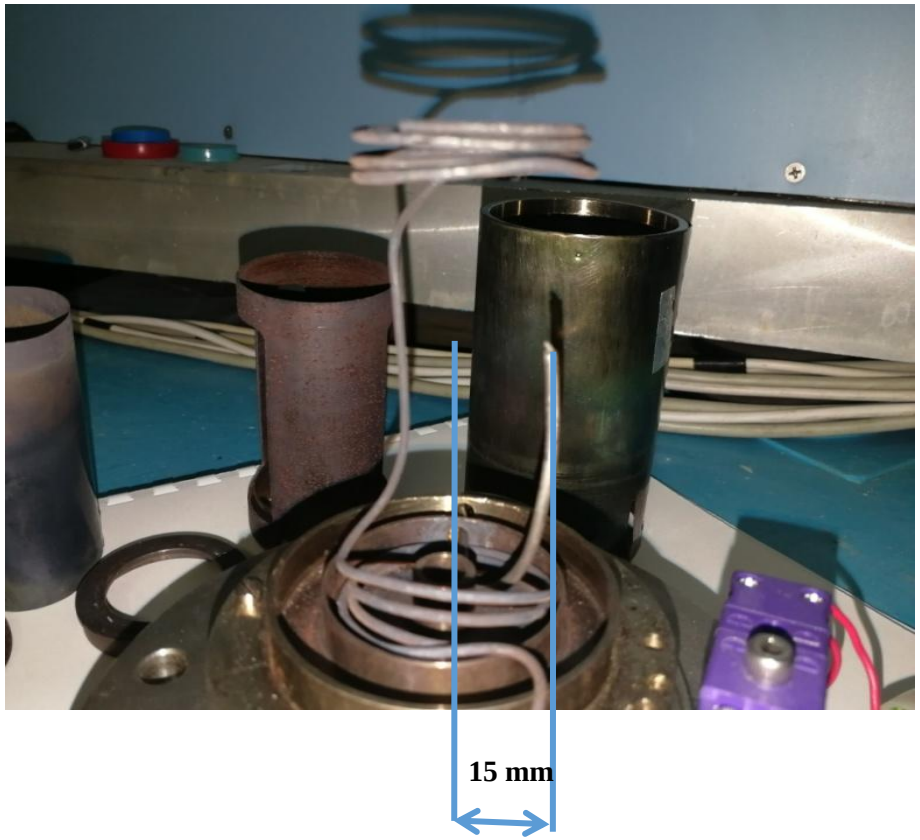
LaB6 , T=293 K, HRFD-Dubna



Furnace



- Vanadium wrap;
- Thermopare type K;
- Tmax=500 C;
- Vmax=20 C/min.

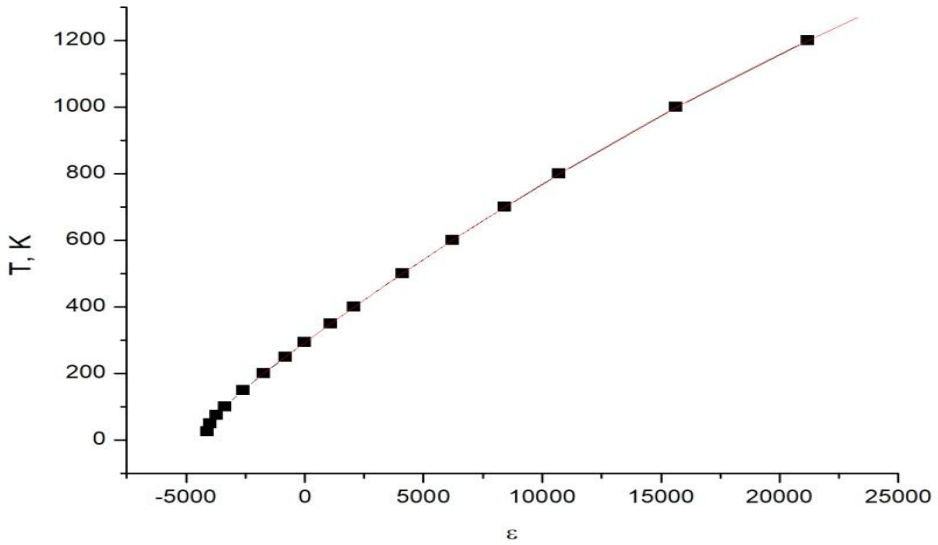


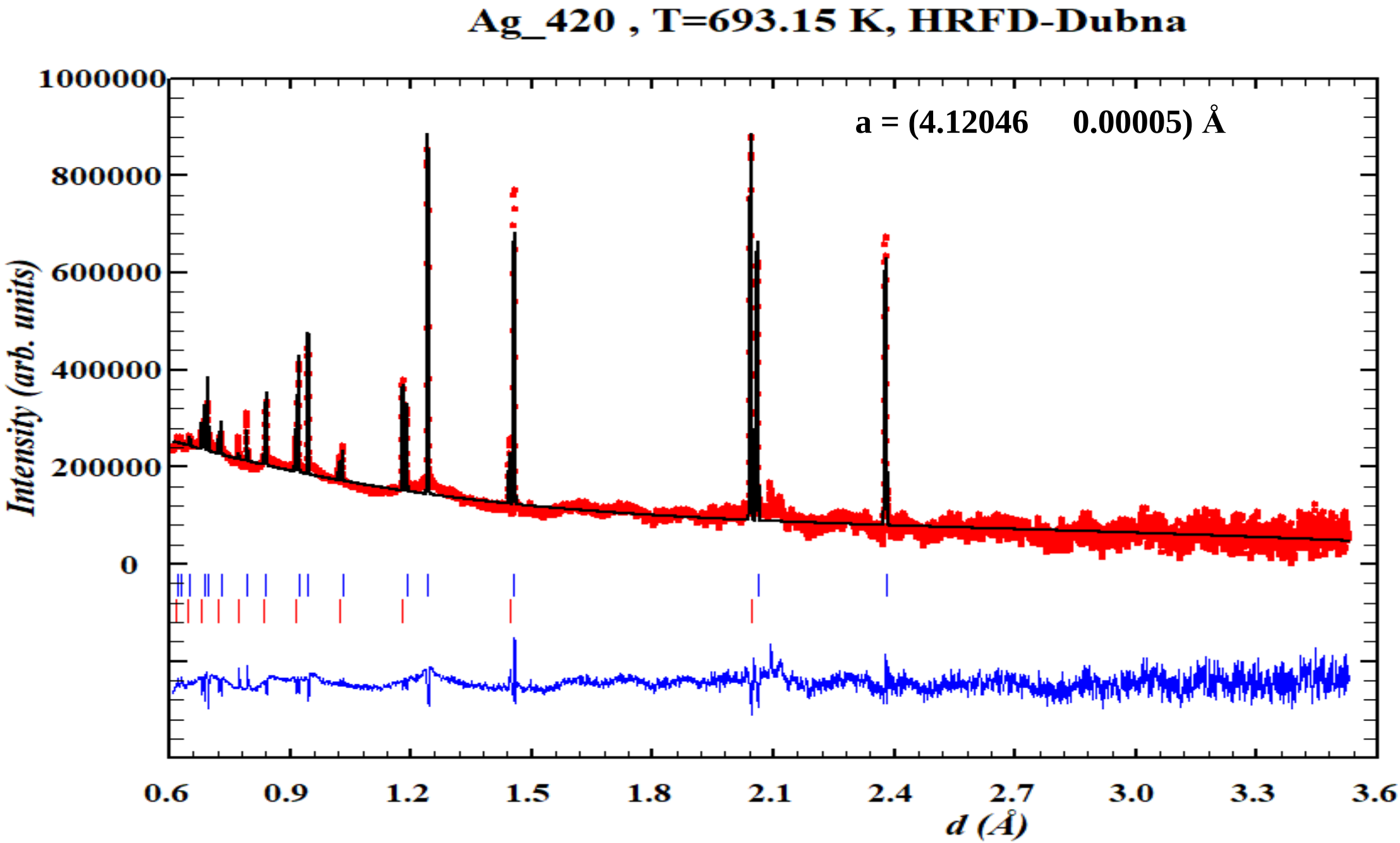
Temperature calibration

- Collect sequence of diffraction patterns for silver powder.
- Refine unit cell dimensions as a function of temperature
- Calculate ε, $\delta\varepsilon$
- Calculate real T, δT using polynomial coefficients

$$\varepsilon = \frac{a - a_{20}}{a_{20}}$$

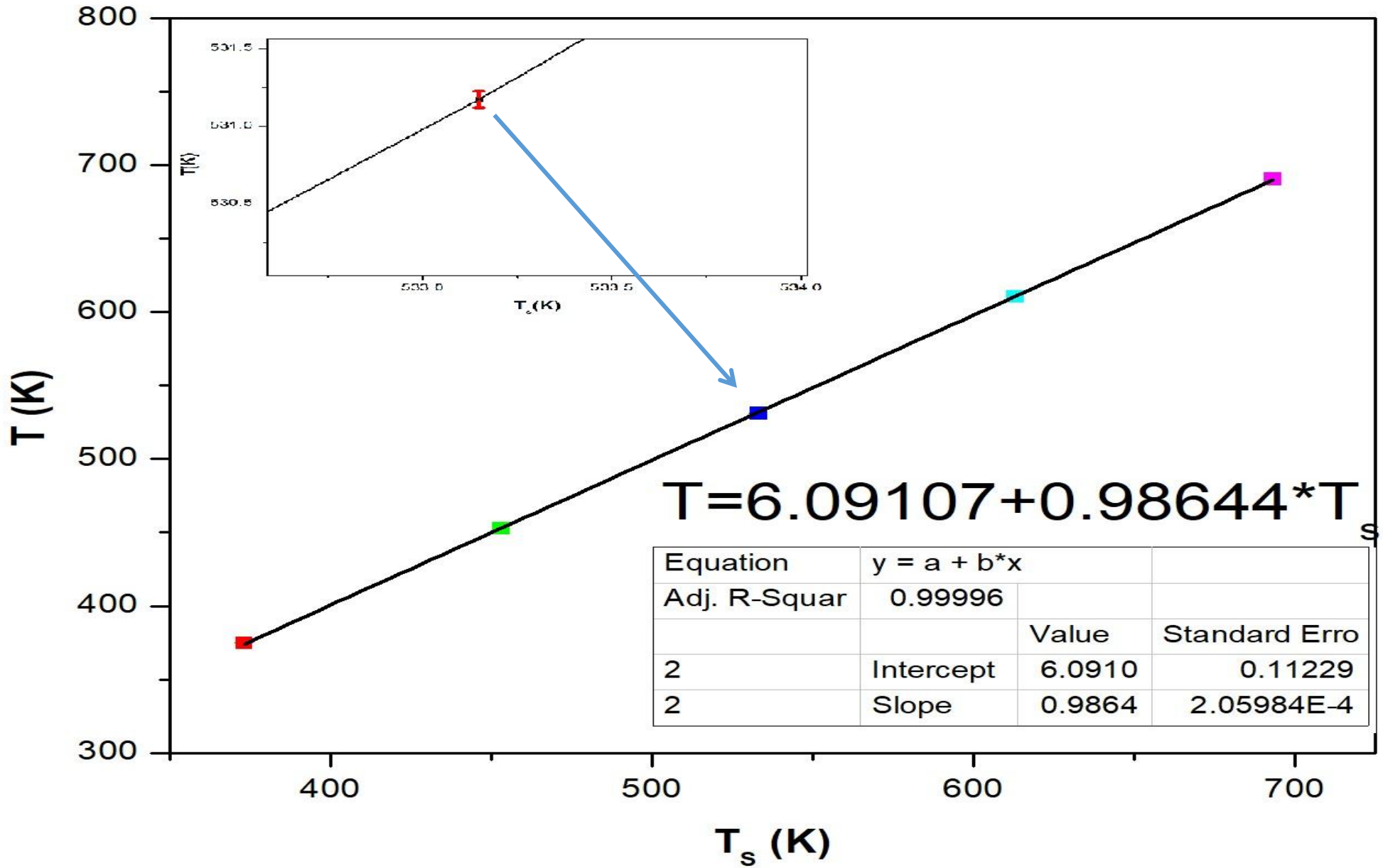
$$T = 293.21939 + 0.05216 \cdot \varepsilon - 4.63693 \cdot 10^{-7} \cdot \varepsilon^2 + 1.07258 \cdot 10^{-12} \cdot \varepsilon^3$$



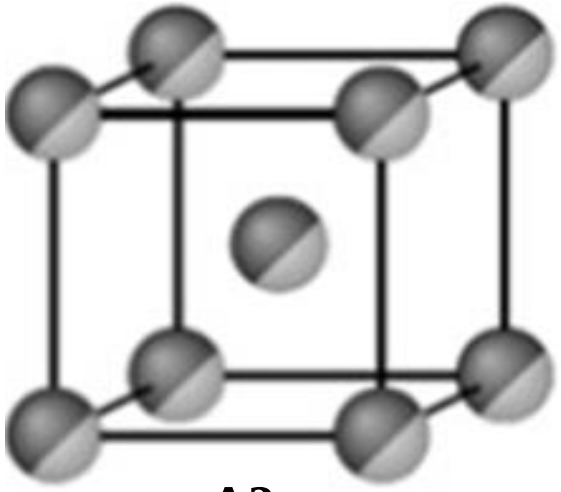

Ag_420 , T=693.15 K, HRFD-Dubna
a = (4.12046 0.00005) Å
Intensity (arb. units)
1000000
800000
600000
400000
200000
0
0.6
0.9
1.2
1.5
1.8
2.1
2.4
2.7
3.0
3.3
3.6
d (Å)

$$T = 293.21939 + 0.05216 \cdot \varepsilon - 4.63693 \cdot 10^{-7} \cdot \varepsilon^2 + 1.07258 \cdot 10^{-12} \cdot \varepsilon^3$$

Sample	Ts (K)	a (Å)	δa (Å)	T (K)	δT (K)
Ag_100	393.15	4.092607	0.00003	357.2300008	0.0521460
Ag_180	473.15	4.099796	0.00003	445.5357127	0.0521398
Ag_260	533.15	4.106391	0.00004	524.0738368	0.0521189
Ag_340	613.15	4.113296	0.00003	603.7998881	0.0521181
Ag_420	693.15	4.12046	0.00005	683.8419137	0.0520712


T=6.09107+0.98644*T
S
Equation y = a + b*x
Adj. R-Squar 0.99996
Value Standard Erro
2 Intercept 6.0910 0.11229
2 Slope 0.9864 2.05984E-4
T (K)
T_S (K)
800
700
600
500
400
300
400
500
600
700
T(K)
T_s(K)

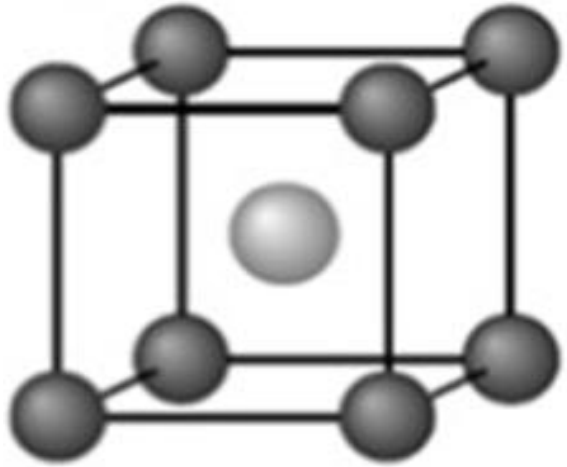
Fe-Al alloy



A2

Space group: I m 3 m

hkl : h+k+l = 2n
0kl : k+l = 2n
hhl : l = 2n
h00 : h = 2n

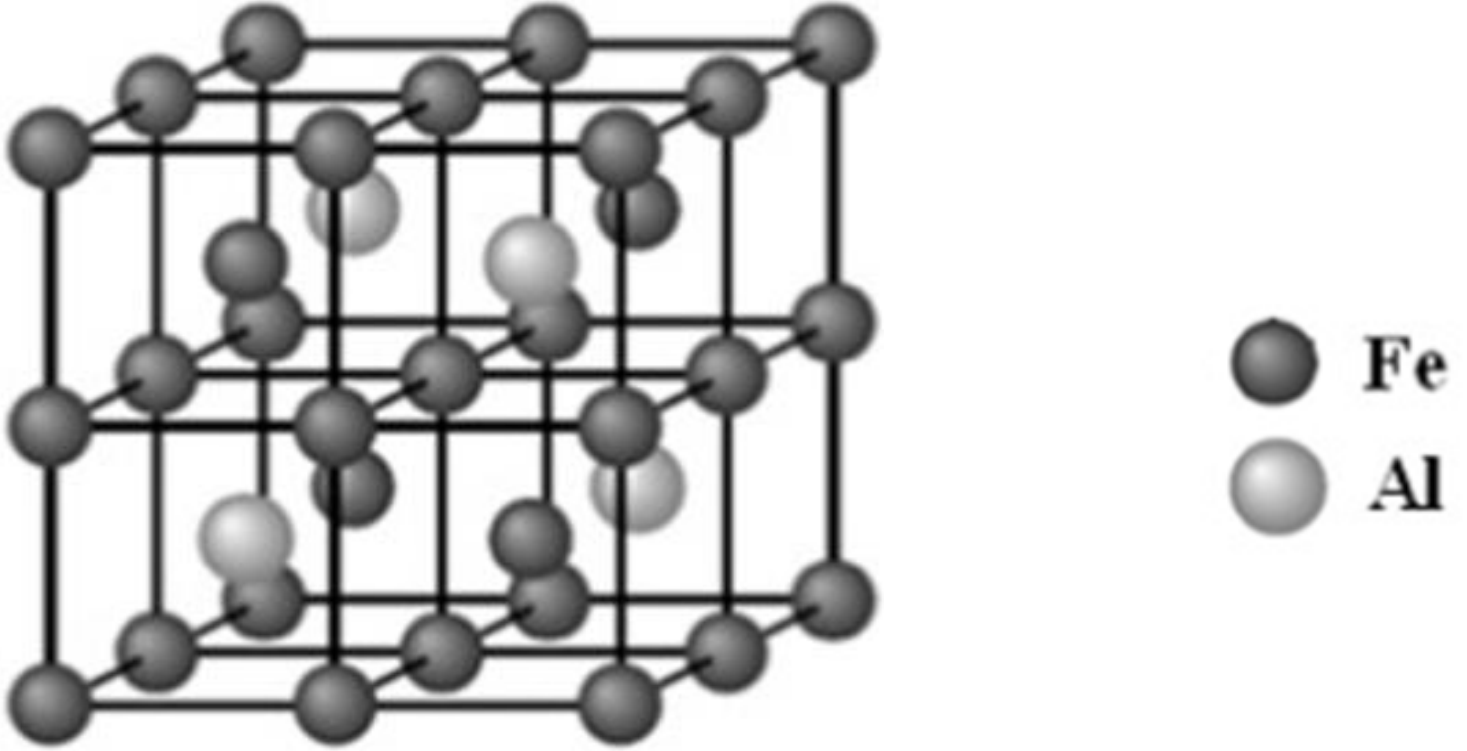


B2

Space group: P m 3 m

h, k, l no restriction

For these two structures although they have similar geometric arrangements, neither the lattice, the basis nor the crystal structure are the same. A2 structures have the same element in the corners of the unit cell as well as the center of the body. B2 has different elements at the corners of the unit cell than the one in the center of the body.

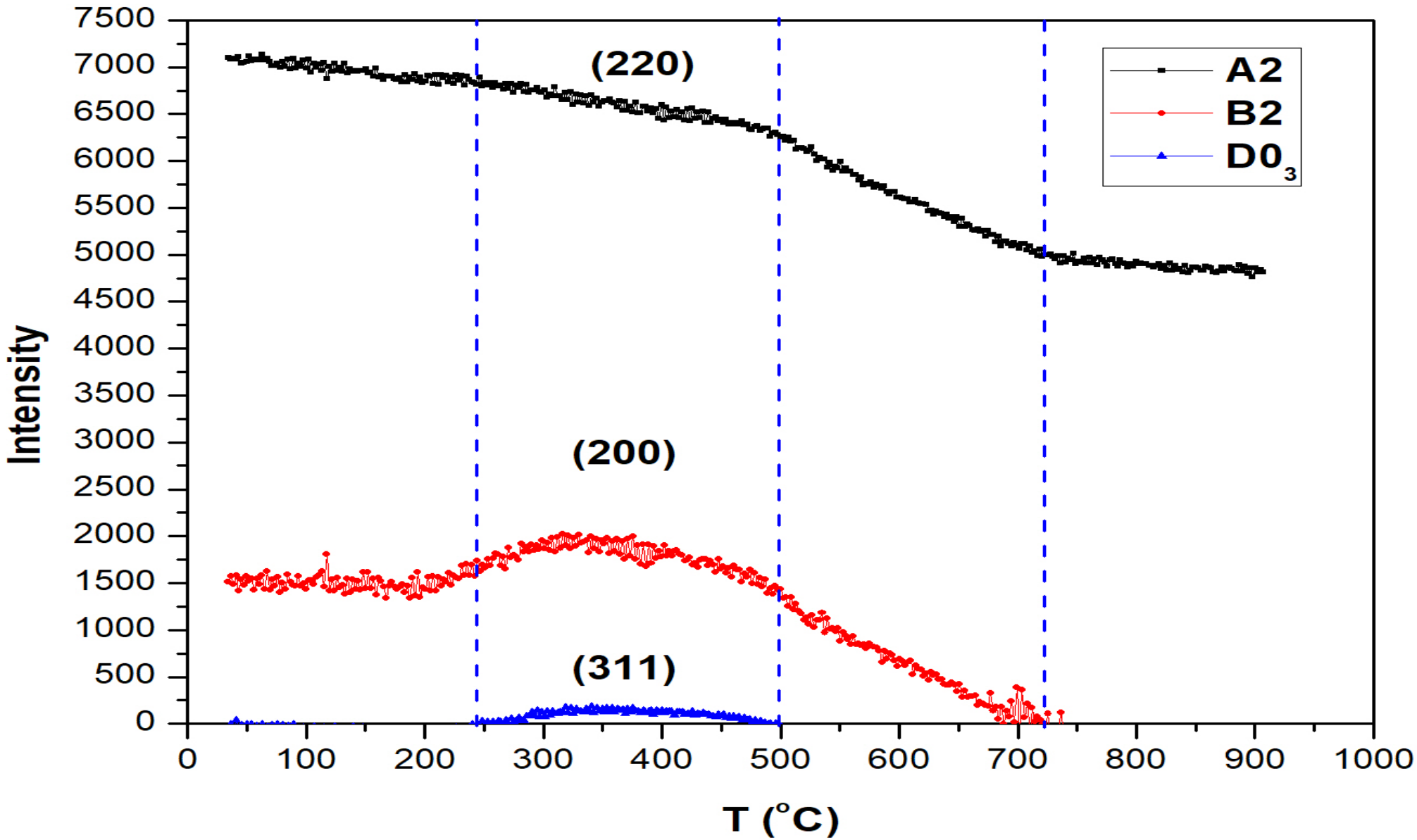


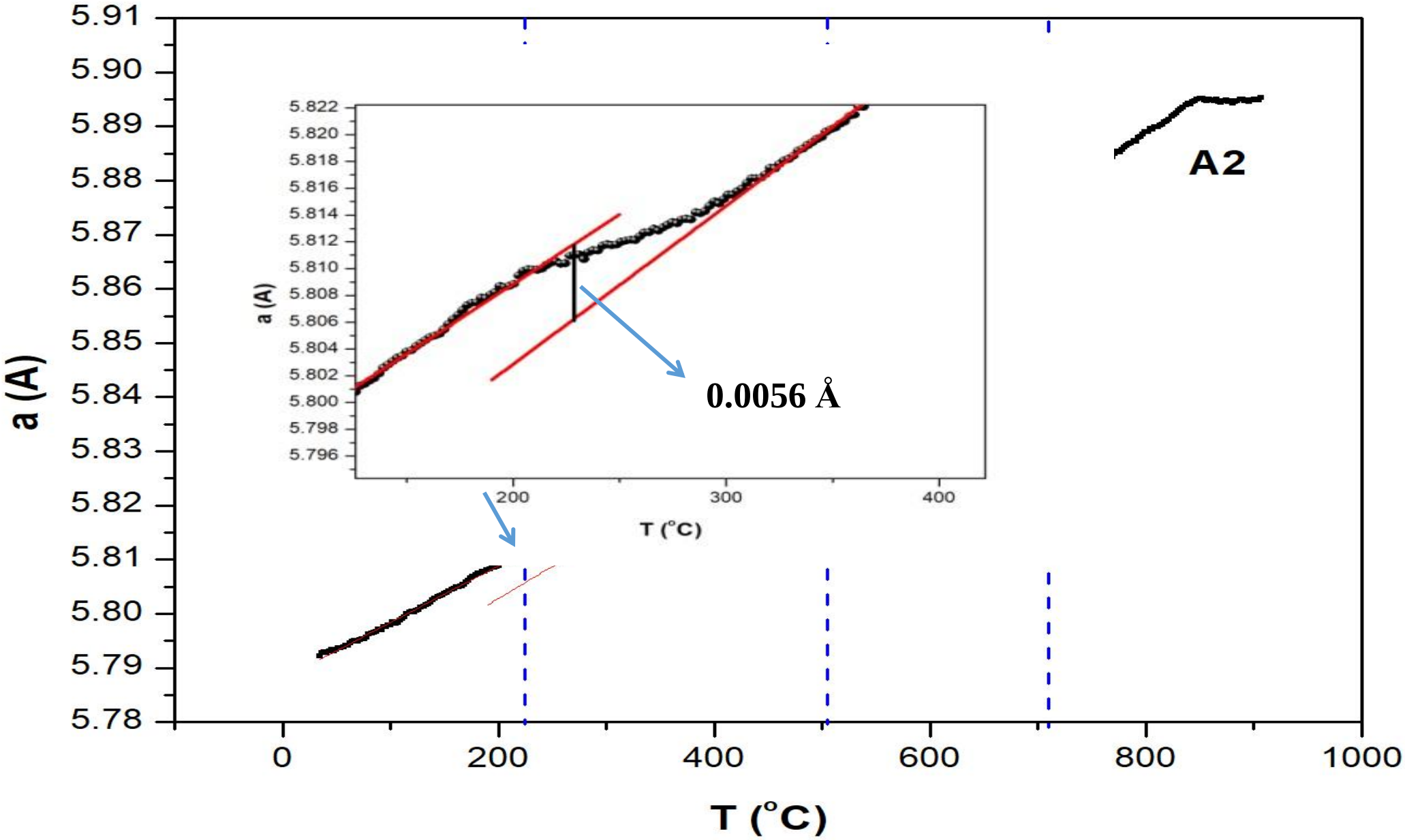
$D0_3$

Space group: F m 3 m

hkl : h+k,h+l,k+l = 2n
0kl : k, l = 2n
hhl : h+l = 2n
h00 : h = 2n

The D03 crystal structure can be thought of as 8 subunits with Fe on the cube corners and Fe and Al alternating in the body centers.


(220)
(200)
(311)
A2
B2
D0 3
Intensity
T (°C)
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
0
100
200
300
400
500
600
700
800
900
1000


a (A)
T (°C)
A2
0.0056 Å

Conclusion

- Introduced to neutron diffraction;
- Learning to work at Fullprof and Fityk;
- Calculated parameters experimental station using standard sample with those programs;
- Did calibration furnace;
- Used data from FeAl and saw that parameters unit cell changed with order and disorder structure (D03-order; B2,A2-disorder).

Thank you for your attention !!!