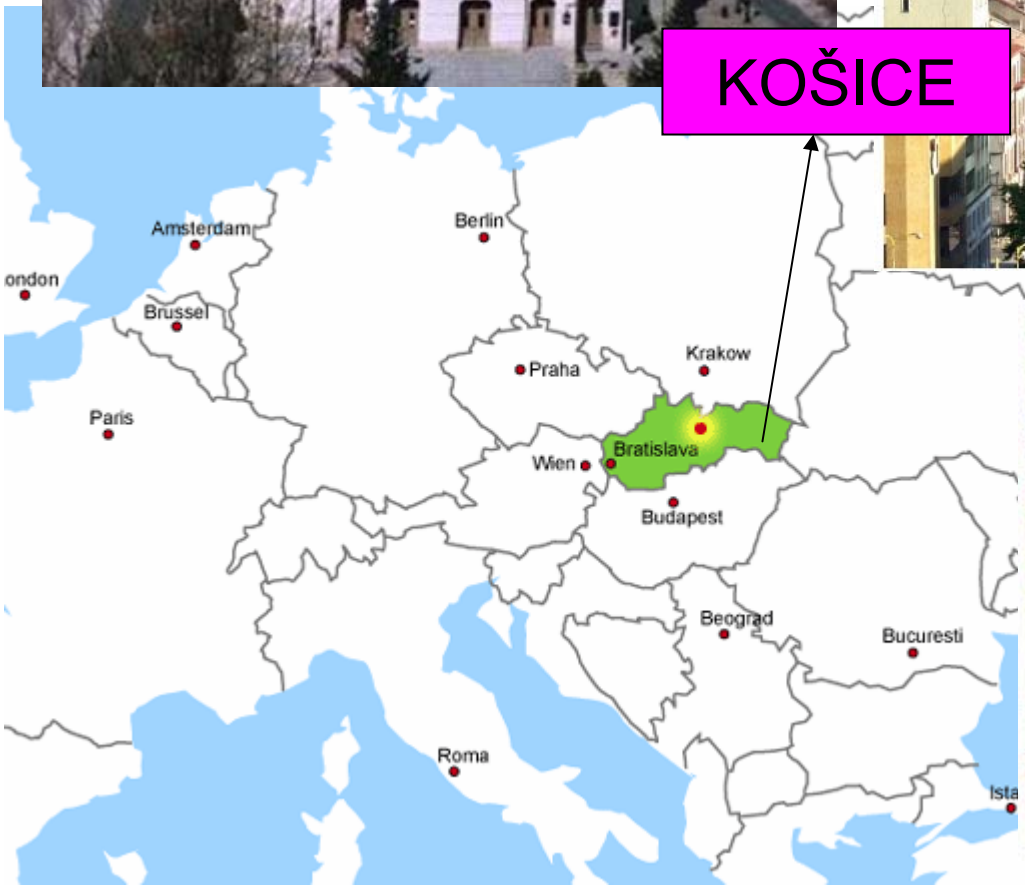
KOŠICE




my faculty

# Thermal stability of metallic microwires studied by hard X-ray diffraction

Jana Gamcová
supervisors: Štefan Michalik
**Jozef Bednarčík**

# Thermal stability of metallic microwires studied by hard X-ray diffraction

- characterize the structure of as-prepared metallic glasses and follow evolution of its structure during thermal loading using in-situ hard X-ray diffraction

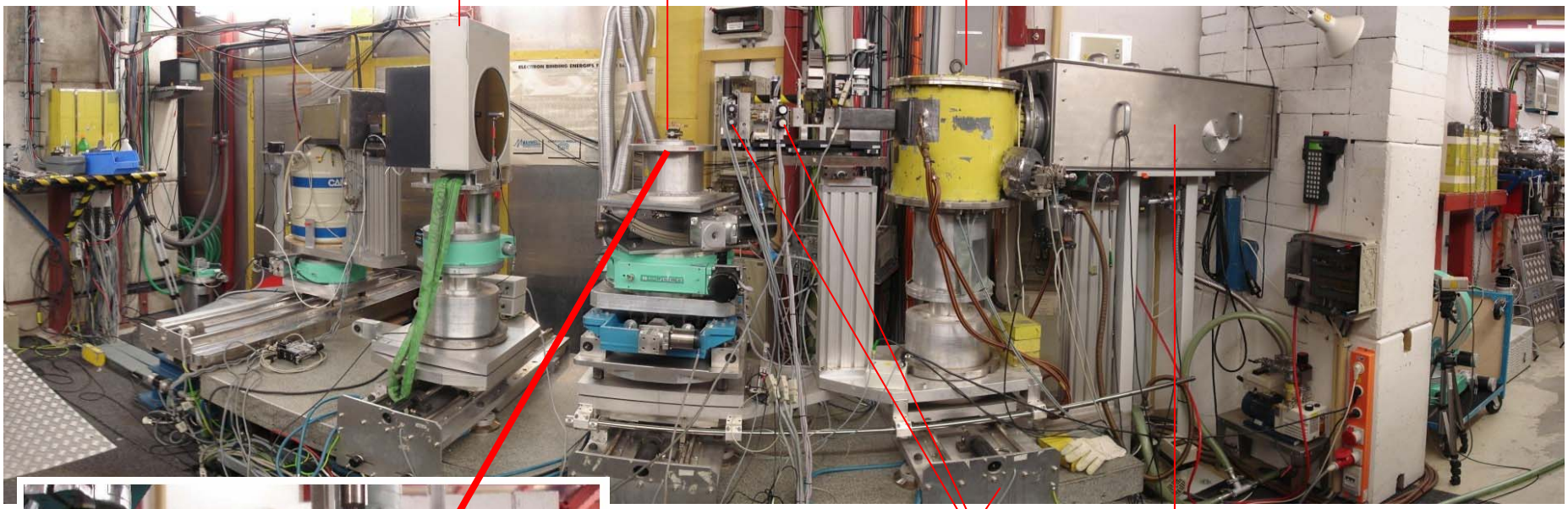
- the study of the glass-transition and at the early stages of crystallization

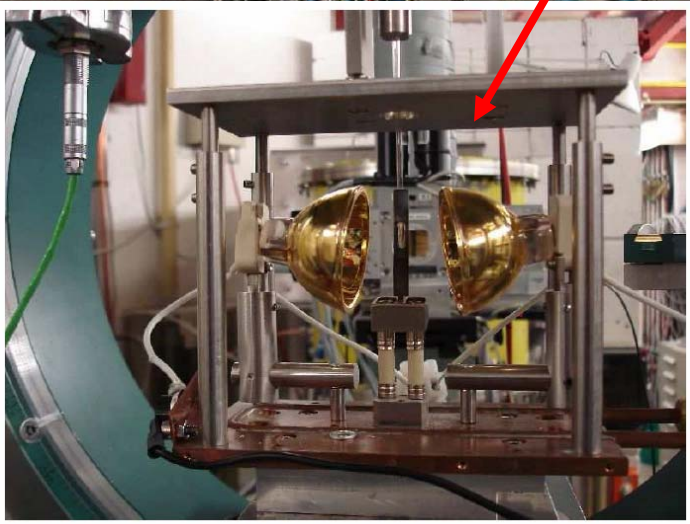
# BW 5

- BW5 is dedicated to X-ray scattering experiments at energies between 60 and 150 keV. They are high energy.

- The large penetration depth at these energies of typically several mm to cm allows the investigation of bulk materials and complex sample environments.

# BW 5



# Our aims:

- characterize a structure of my samples by hard X-ray scattering experiments
- characterize the structure of amorphous samples during thermal loading by in-situ hard X-ray diffraction using mirror infrared furnace
- determinate phases
- determinate grain size

# Conditions of our experiment

- measured by in-situ X-ray diffraction method in transmission mode

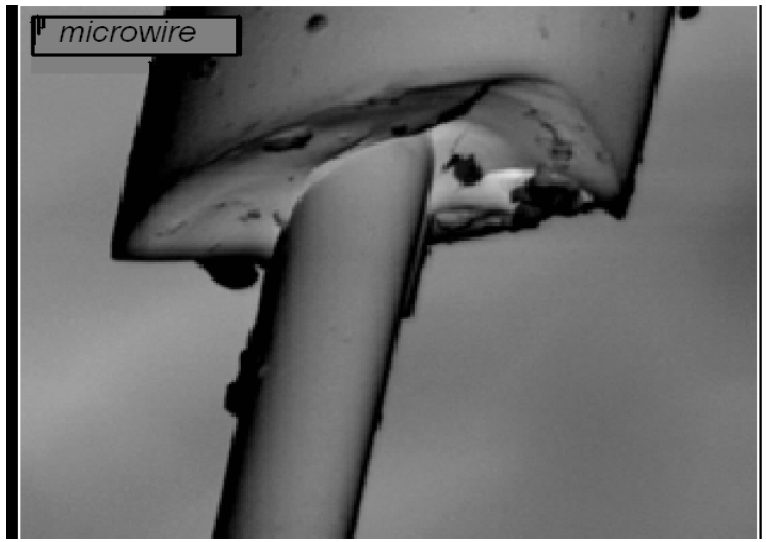
- wavelength:  0.124Å (E=100keV)
- beam size: 1x1mm
- illumination time: 30sec.
- constant rate heating 5K/min
- sample to detector distance: 998.2mm

# Set of investigated samples:

we were measuring: $Ni_{75,5}Si_{7,5}B_{15}$,

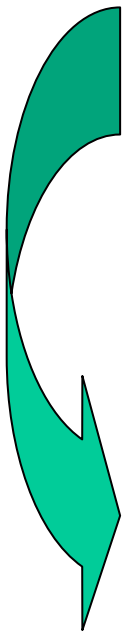
$Fe_{38,75}Ni_{38,8}Si_{7,5}B_{15}$,

$Fe_{49,6}Ni_{27,9}Si_{7,5}B_{15}$




metal core – 15μm
glass cover – 7μm

# Characterize a structure of samples…

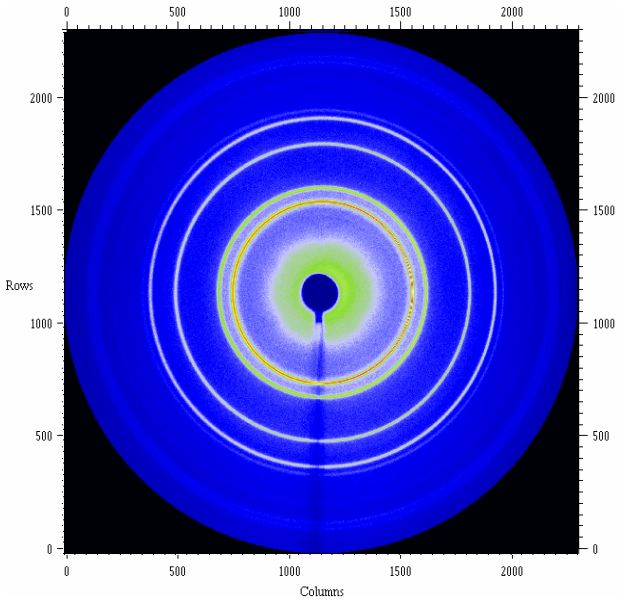


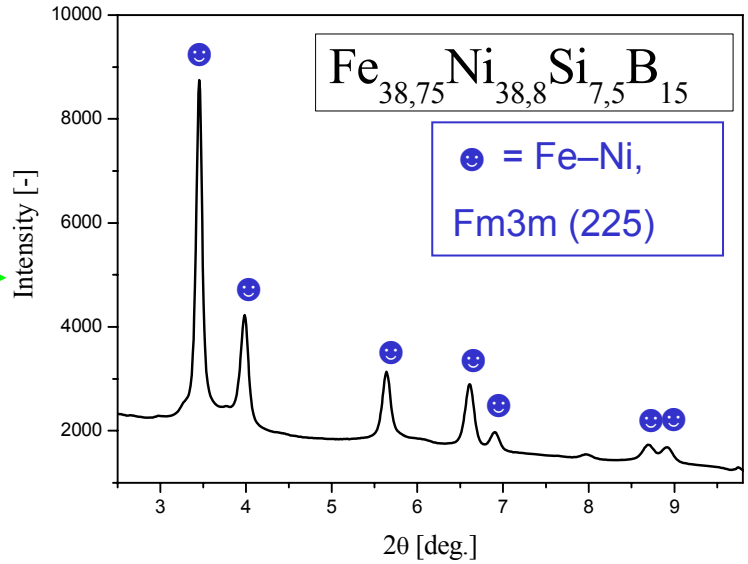
$Ni_{75,5}Si_{7,5}B_{15}$ ..... polycrystalline

$Fe_{38,75}Ni_{38,8}Si_{7,5}B_{15}$ ..... polycrystalline

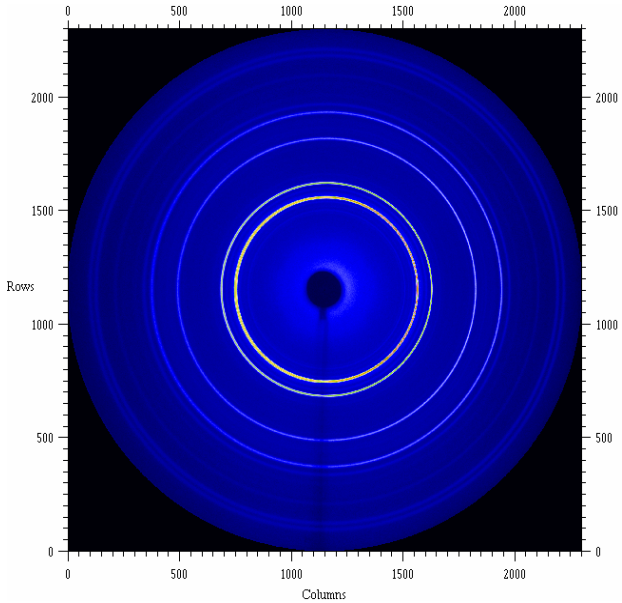
$Fe_{49,6}Ni_{27,9}Si_{7,5}B_{15}$ ..... !!!!! amorphous !!!!!

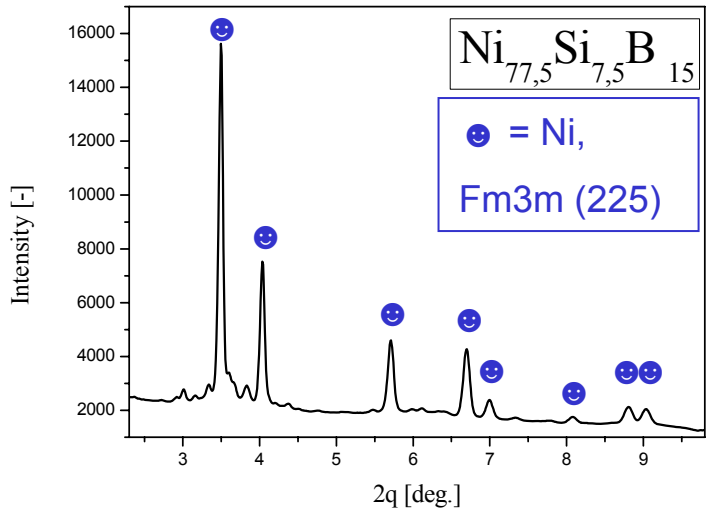
next step: phase analysis
determination of grain size




grain size:
cca 11nm




grain size:
cca 30nm

# Size Broadening

**Scherrer formula:**

$$D_v = \frac{K\lambda}{\beta \cos\theta}$$

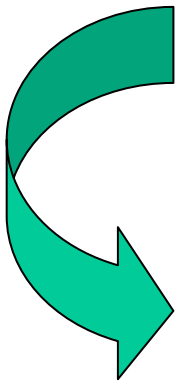
- $D_v$ = volume weighted crystallite size
- K = Scherrer constant, somewhat arbitrary value that falls in the range 0.87-1.0 ( I assume K=1)
- $\lambda$ = the wavelength of the radiation
- $\beta$ = the Full Wide at Half Maximum (FWHM). It is in radians $2\theta$

# Characterize a structure of samples…

$Ni_{75,5}Si_{7,5}B_{15}$ ..... polycrystalline

$Fe_{38,75}Ni_{38,8}Si_{7,5}B_{15}$ ..... polycrystalline

$Fe_{49,6}Ni_{27,9}Si_{7,5}B_{15}$ ..... !!!!! amorphous !!!!!



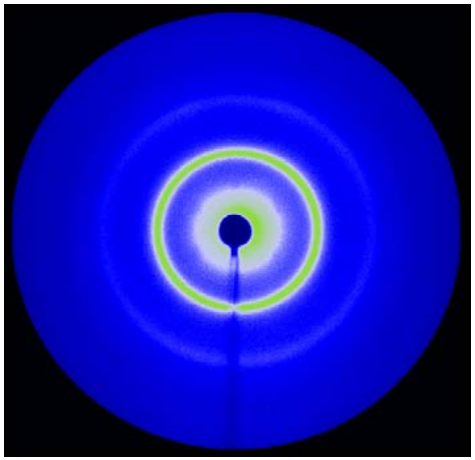
next step: characterize of structure during thermal loading using mirror infrared furnace

determinate phases

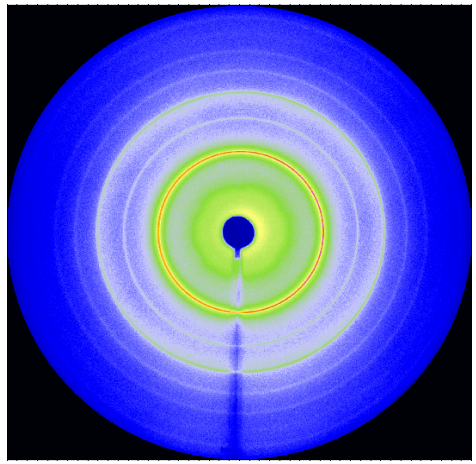
determination size of grain

# $Fe_{49.6}Ni_{27.9}Si_{7.5}B_{15}$

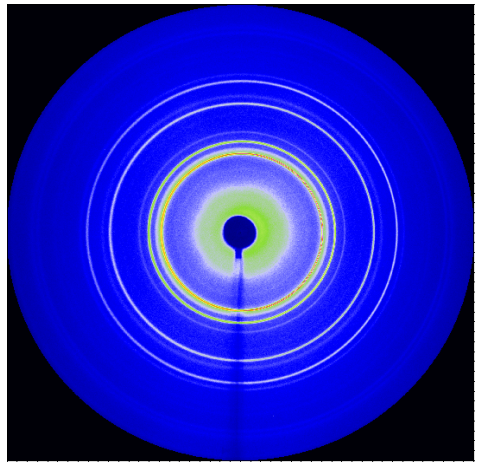
## … thermal loading …



initial stage
(amorphous)



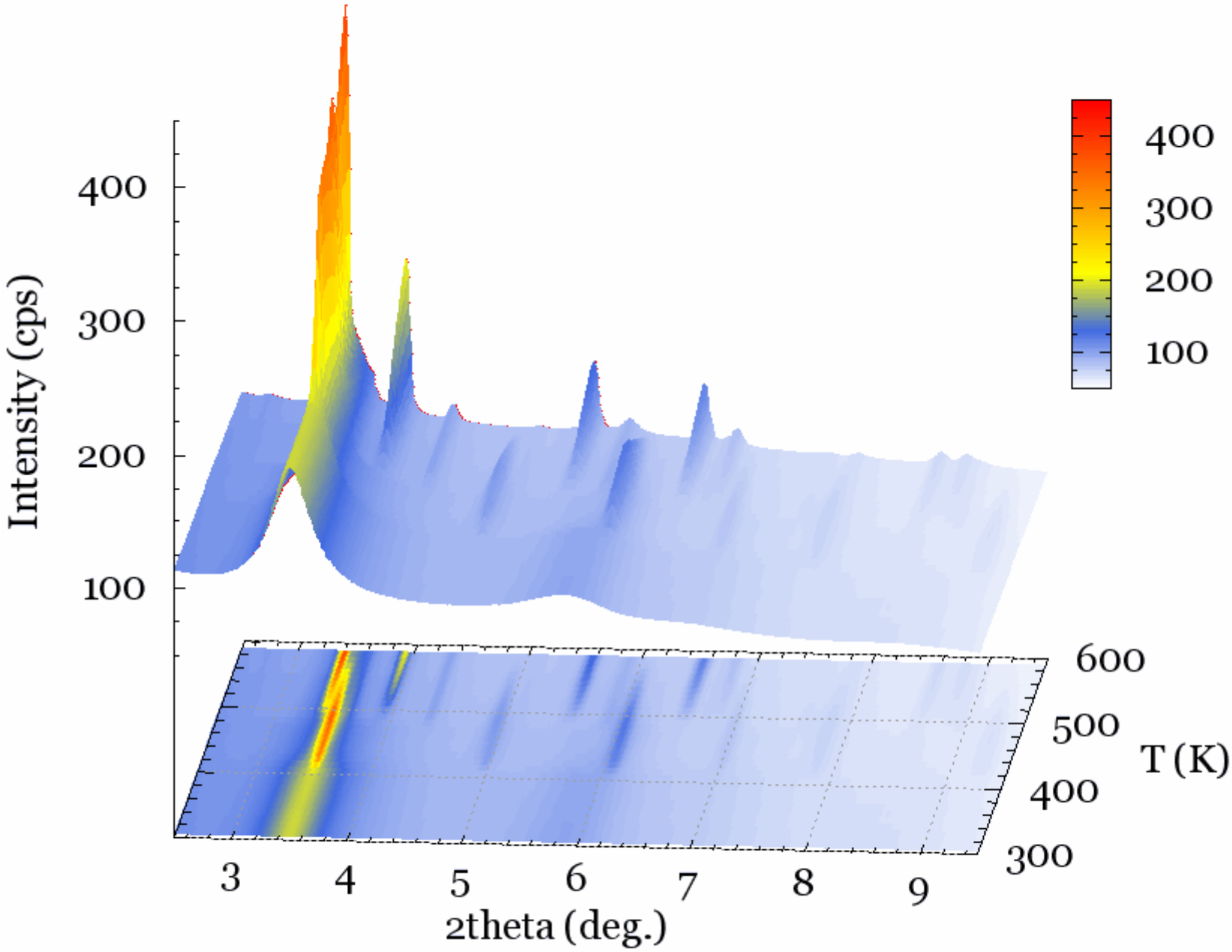
first crystallization



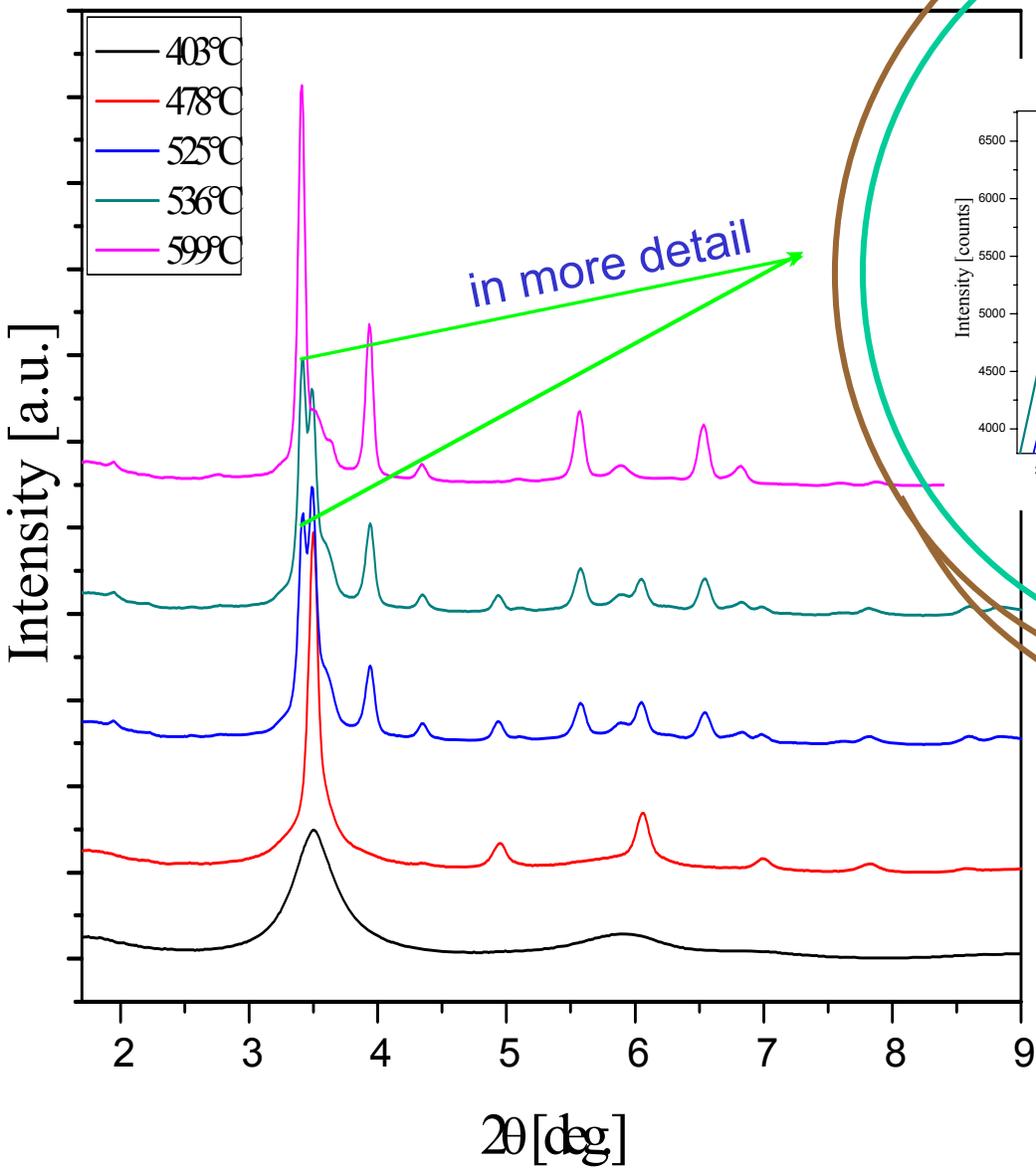
final stage

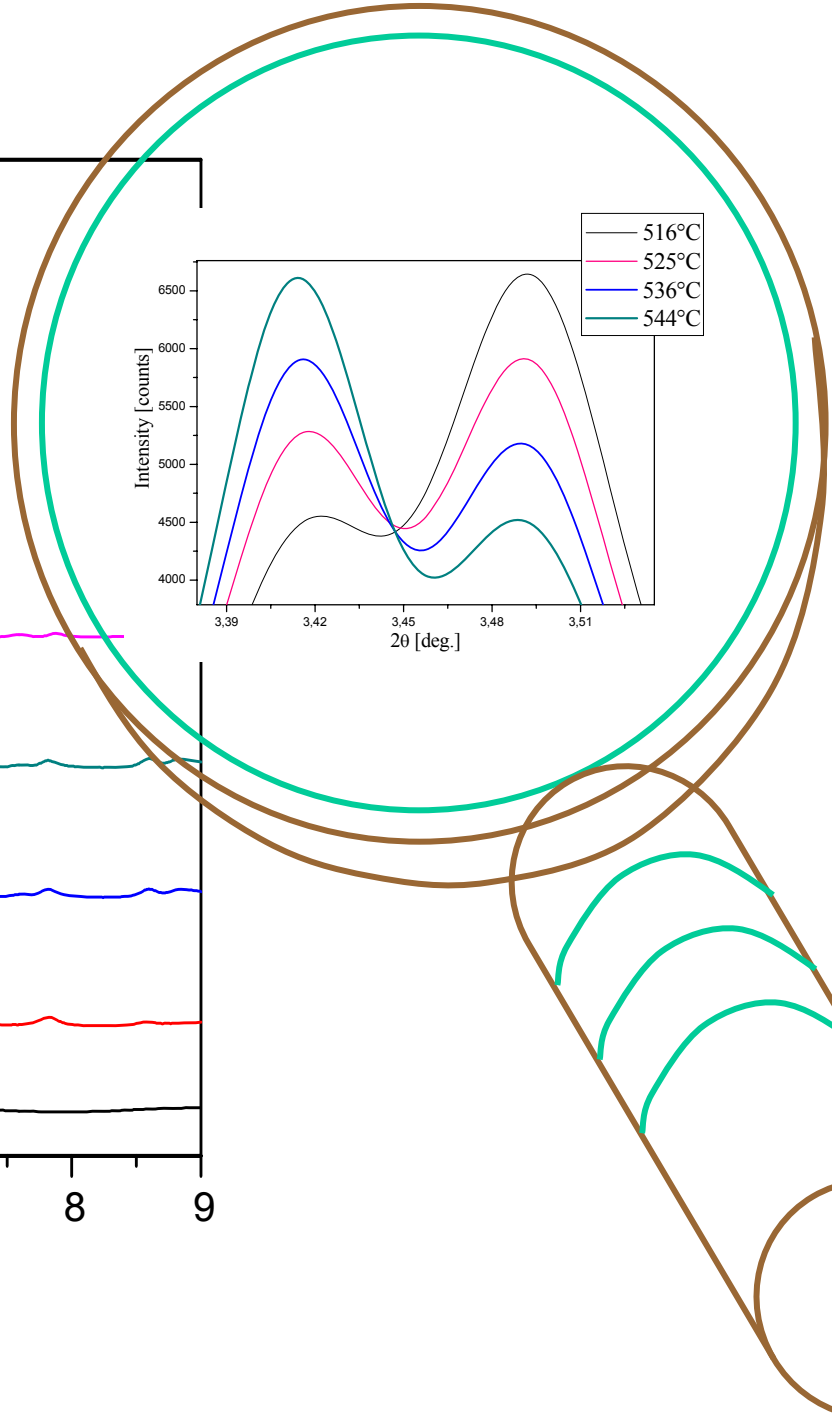
increase temperatures: 34 – 608 °C

**what is happening??**

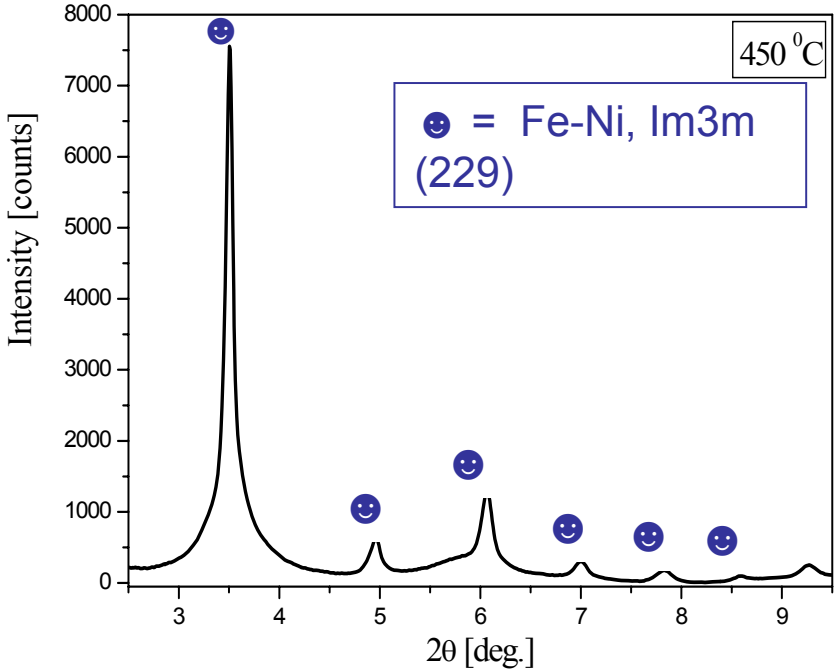

Intensity (cps)
400
300
200
100
400
300
200
100
600
500
400
300
T (K)
3
4
5
6
7
8
9
2theta (deg.)

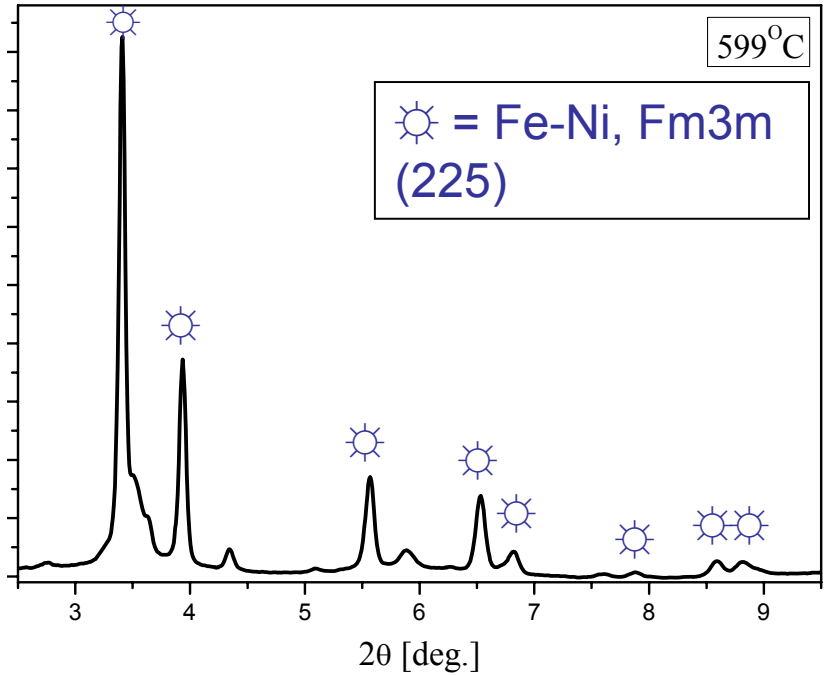
# $Fe_{49.6}Ni_{27.9}Si_{7.5}B_{15}$

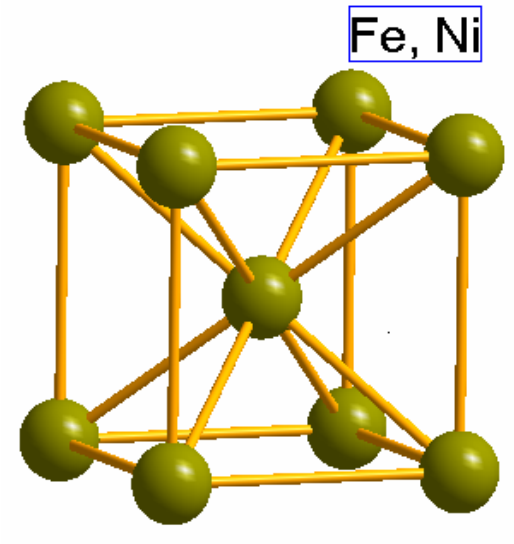


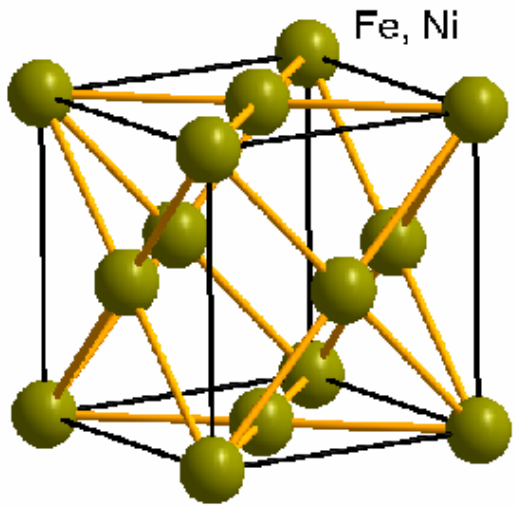



# $Fe_{49.6}Ni_{27.9}Si_{7.5}B_{15}$

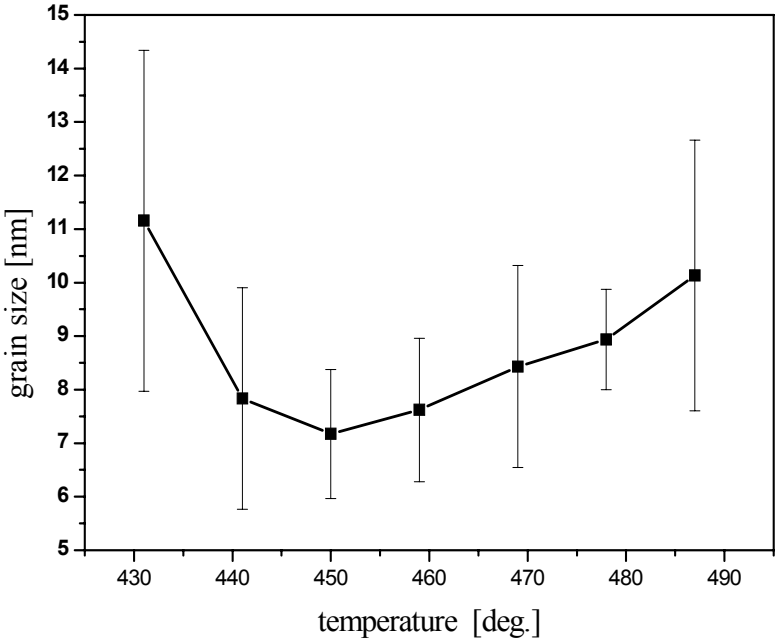


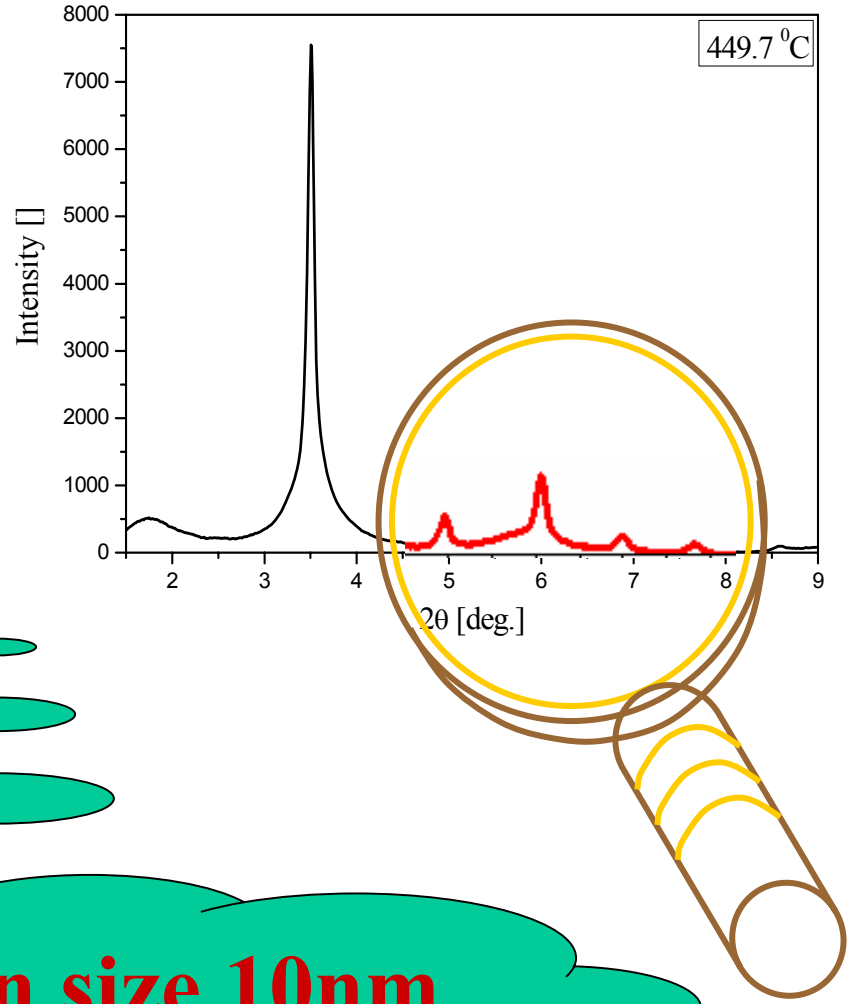



# $Fe_{49.6}Ni_{27.9}Si_{7.5}B_{15}$




**average grain size 10nm**

# Acknowledge...

... **DESY** for opportunity to participate in Summer Student Program 2009

... my supervisors **Dr. Jozef Bednarčík** and Štefan Michalik for their care

... **all of you** for your attention