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Mössbauer Spectroscopy  
in  
Materials Science

# Effect of external magnetic field annealing on magnetic texture of Mo containing NANOPERM-type alloys

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**Acknowledgement :** This work was supported by FR/SK/23, FR/SL/FEISTU/04 and APVT-20-008404 and VEGA 1/1014/04 and Deutscher Akademischer Austauschdienst

## Introduction

In this work, we concentrate on the study of the effect of applied magnetic field on magnetic microstructure

- External magnetic fields are known to modify microstructure of materials during their solidification and/or crystallisation. In an external magnetic field strong particle to particle interactions lead to a highly anisotropic microstructure. If the alloy is in ferromagnetic state, strong interactions between magnetic moments of particles and external magnetic field as well as particle-to-particle couplings are expected.
- Therefore, we focused on magnetic texture of ribbon-shaped precursors of Mo containing alloys of similar composition, namely  $\text{Fe}_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  and  $(\text{Fe}_{0.5}\text{Co}_{0.5})_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$ .
- Several techniques were applied :
  - **Magnetic measurements** follow the changes of saturation magnetization and coercive force
  - **Mössbauer experiments** provide information about the orientation of magnetization of the individual particles with respect to an external magnetic field
  - **MOKE experiments** follow the changes of magnetization at the very surface of ribbon-shaped specimen

## Experimental details

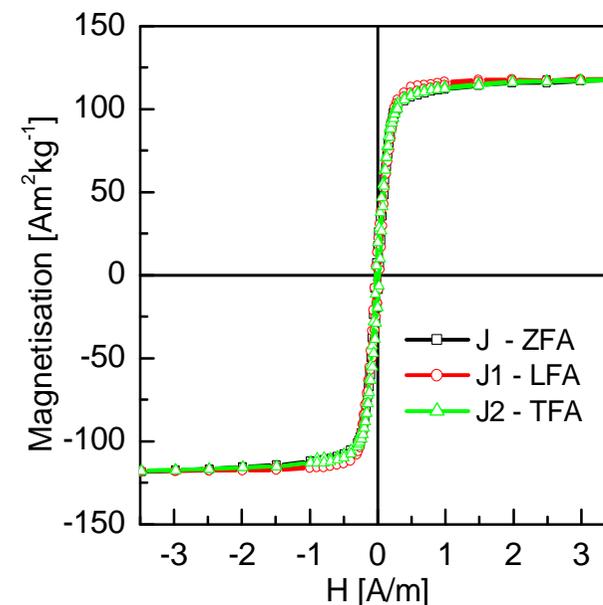
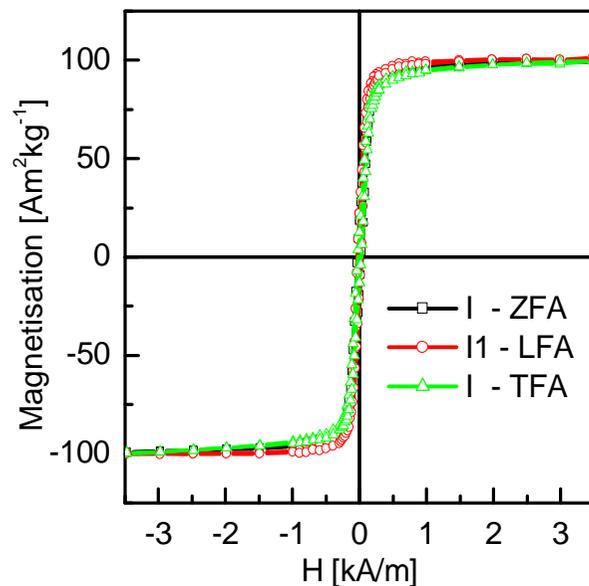
Amorphous precursor of the nominal composition of  $\text{Fe}_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  and  $(\text{Fe}_{0.5}\text{Co}_{0.5})_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  were prepared by rapid quenching on a rotating wheel.

- Samples with the thickness of about 20  $\mu\text{m}$  and width of 2-3 mm were inspected.
- First stages of crystallisation were covered by :
  - isothermal one-hour annealing at 510°C (sample I) and 550°C (sample J) in a vacuum in the presence of external longitudinal (index 1, LFA) and transverse (index 2, TFA) magnetic fields, for the first alloy.
  - isothermal one hour annealing at 440°C and 470°C in a vacuum in the presence of transverse magnetic field, for the second alloy.
- Measurements of Mössbauer spectra were carried out, at room (RT: 300 K) and liquid nitrogen (LNT: 77K) temperatures, in transmission geometry with a  $^{57}\text{Co}(\text{Rh})$  source.
  - Spectral parameters were refined by the help of the NORMOS fitting software.
- Magnetic measurements were performed by using vibrating sample magnetometer. Hysteresis loops were measured at room temperature.
- Magneto Optical Kerr Effect (MOKE) was performed using He-Ne laser ( $\lambda=632,8$  nm) at room temperature.

## Magnetic measurements

From hysteresis loops of  $\text{Fe}_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  alloy annealed at  $510^\circ\text{C}$  and  $550^\circ\text{C}$  in longitudinal and transverse field:

- an increase in magnetic induction is observed with rising annealing temperature.
  - LFA causes a slight increase in saturation induction whereas the TFA exhibits an opposite tendency as compared with the ZFA hysteresis loop. One can speculate that the external field forces the magnetic moments of the crystalline grains to become aligned more normal to the plane of the ribbon.



## Magnetic measurements

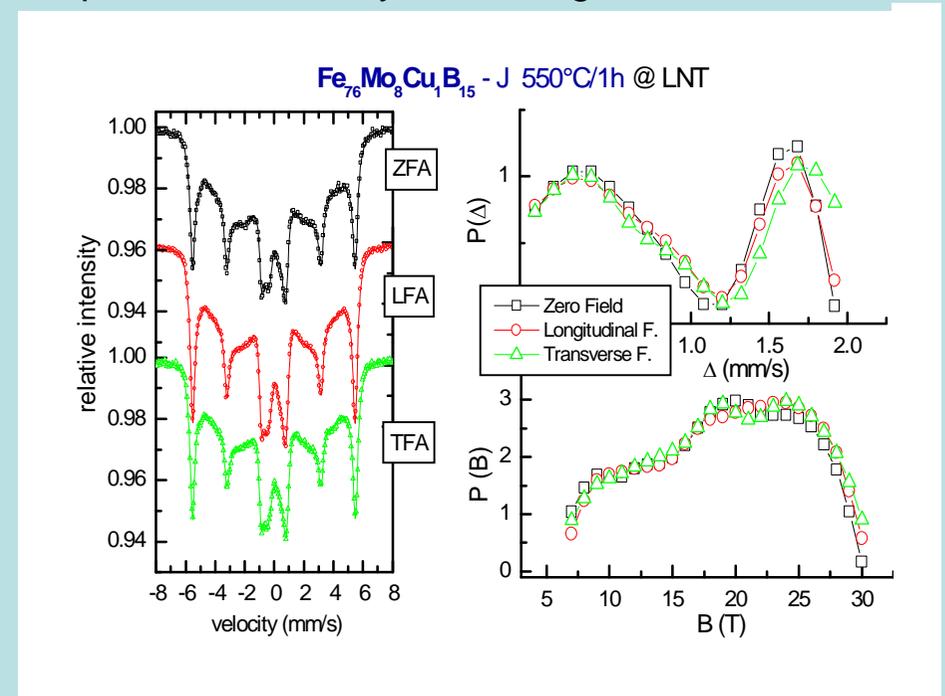
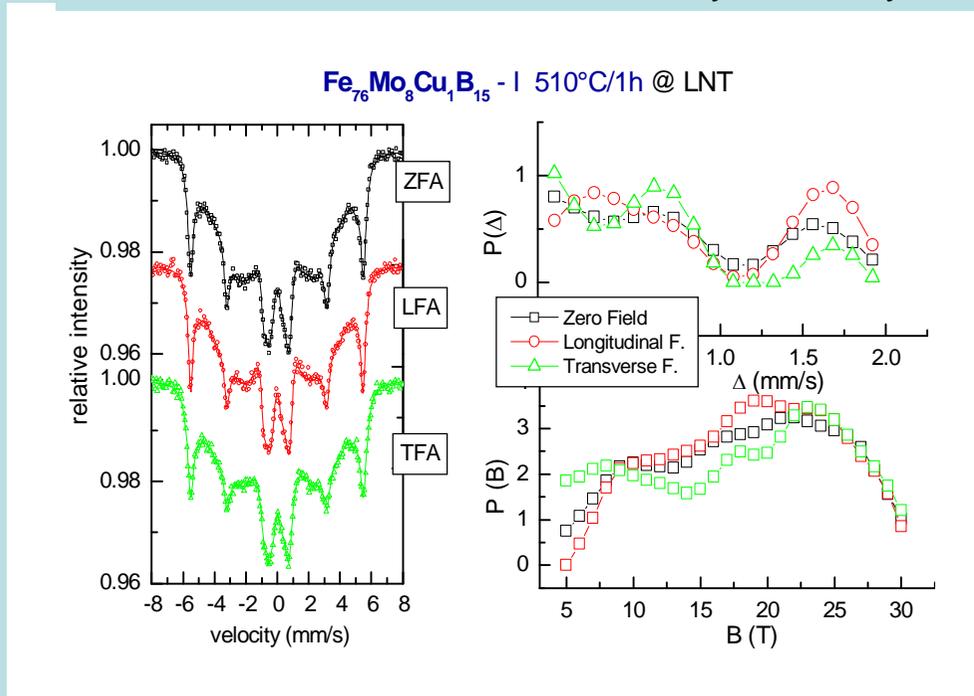
- Table lists the derived values of saturation induction  $B_s$  and coercive field  $H_C$  values.
  - The in-field annealing decreases  $H_C$  in sample I which has lower crystalline fraction. In sample J, the decrease in  $H_C$  is found only for LFA. TFA has practically no effect upon coercive field.

sample	$M_s$ ( $\text{Am}^2\text{kg}^{-1}$ )			$H_C$ (A/m)		
	ZFA	LFA	TFA	ZFA	LFA	TFA
I	100.23	95.54	95.53	33.5	29.4	25.7
J	112.38	115.73	114.16	29.3	25.5	29.6

**Coercive force  $H_C$  and saturation induction  $B_s$**  derived from hysteresis loops of  **$\text{Fe}_{76}\text{M}_{08}\text{Cu}_1\text{B}_{15}$**  alloy annealed at  $510^\circ\text{C}$  (I) and  $550^\circ\text{C}$  (J) in zero (ZFA), longitudinal (LFA) and transverse (TFA) magnetic field.

# Mössbauer measurements

- The spin texture was determined from Mössbauer measurements using the fact that the ratio  $A_{23} = I_{2,5} / I_{3,4}$  (intensities of the spectral lines 2, 5, and 3, 4) depends upon the angle  $\Theta$  between the magnetisation direction and  $\gamma$ -rays which propagate perpendicular to the plane of the ribbon-shaped sample.
- $A_{23}$  values can vary between 0 and 4 which correspond to the orientation of spins perpendicular to and parallel with the ribbon plane, respectively.
- Deviations in line intensities namely of the crystalline component are clearly seen in figures



Mössbauer spectra and hyperfine field distributions of **Fe<sub>76</sub>Mo<sub>8</sub>Cu<sub>1</sub>B<sub>15</sub>** alloy, annealed @ 510°C and 550°C in external longitudinal (LFA) and transverse (TFA) magnetic fields, recorded @ 77 K

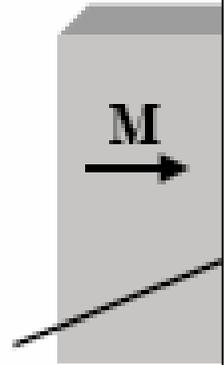
# Mössbauer measurements

- Average hyperfine parameters did not change within the experimental error. Lower values of parameters at RT are due to temperature dependence of hyperfine parameters.
- Magnetic moments in the sample J rotate out of the ribbon plane probably due to stronger particle-to-particle magnetic interactions because of higher amount of nanocrystalline grains.
- In sample I, a tendency of the moments to turn back to the ribbon plane is observed after TFA. It should be noted, however, that at RT the repositioning of the magnetization is alike in both samples, i.e. regardless the intergrain coupling.
- The effects of thermal fluctuations are perhaps already demonstrated.

77K	I - 510°C/1h			J - 550°C/1h			I - 510°C/1h			J - 550°C/1h		
RT	ZFA	LFA	TFA									
$\langle B_{am} \rangle$ (T) $\pm$ 0.1	16.5	16.7	16.6	16.1	16.4	16.4	11.4	11.5	11.6	12.9	12.3	12.2
$\langle \sigma_{am} \rangle$ (T) $\pm$ 0.1	6	5.6	6.6	5.3	5.3	5.4	5.4	5.5	5.5	5.6	5.2	5.0
$\langle \delta_{am} \rangle$ (mm/s) $\pm$ 0.01	0.1	0.10	0.09	0.09	0.09	0.09	0.00	0.00	0.00	0.01	0.01	0.02
$B_{cr}$ (T) $\pm$ 0.1	34.1	34.1	34.1	34.1	34.1	34.2	32.7	32.6	32.7	32.8	32.7	32.7
$B_{if}$ (T) $\pm$ 0.1	31.1	31.1	31.1	31	31.0	31.1	30.7	30.7	30.7	31.0	30.0	30.0
$A_{am}$ (%) $\pm$ 2.5	70	72	66	61	62	63	78	80	78	71	67	66
$A_{cr+if}$ (%) $\pm$ 2.5	31	28	34	39	38	37	22	20	22	29	34	35
A23	1.96	1.4	1.7	1.5	1.3	1.1	1.62	1.28	1.60	1.97	1.3	1.7
$\Theta$ (deg)	54.2	45.4	50.4	48.2	45.1	41.9	49.4	44.1	49.1	54.3	45.1	50.6

Average **hyperfine parameters** derived from Mössbauer spectra of  $Fe_{76}Mo_8Cu_1B_{15}$  alloy annealed at 510°C and 550°C, in zero ZFA, longitudinal (LFA) and transverse (TFA) external magnetic fields, measured at room temperature and 77K.

## MOKE measurements



$$E_s(\phi) = K_0 + \frac{K_1^{eff}}{4} \sin^2[2(\phi - \phi_1)] + K_u \cos^2(\phi - \phi_u)$$

where

$K_0$  - isotropic contribution

$K_1^{eff}$  - crystalline anisotropy constant

$\phi_1$  - rotation of crystalline anisotropy axis

$K_u$  - uniaxial anisotropy constant

$\phi_u$  - rotation of uniaxial anisotropy axis

- incident angle of light
- 360 ° in-plane rotation of sample

Brockmann et al.: J. Appl. Phys., Vol 81, #8, 1997

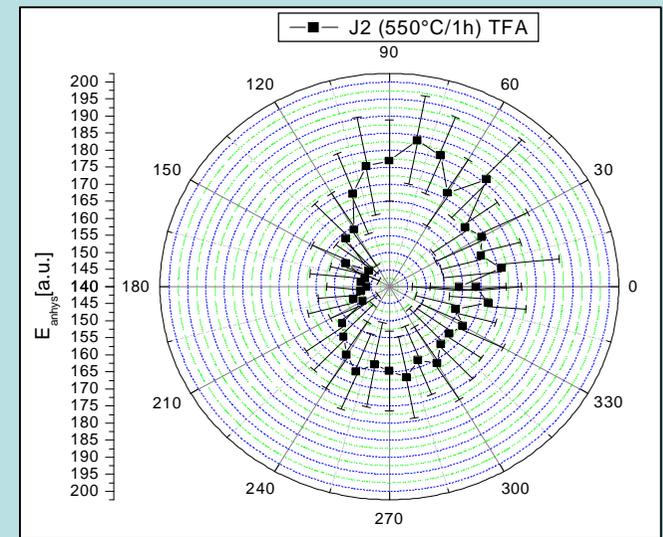
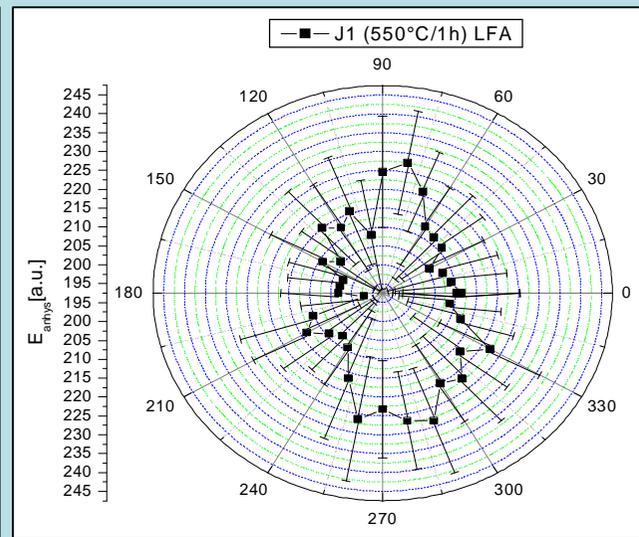
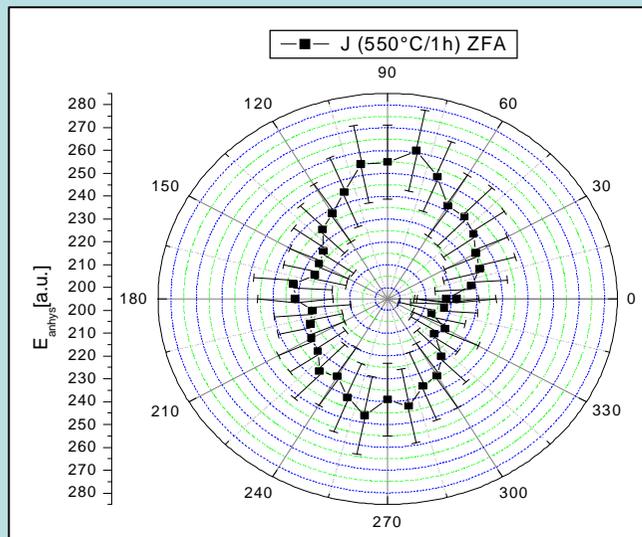
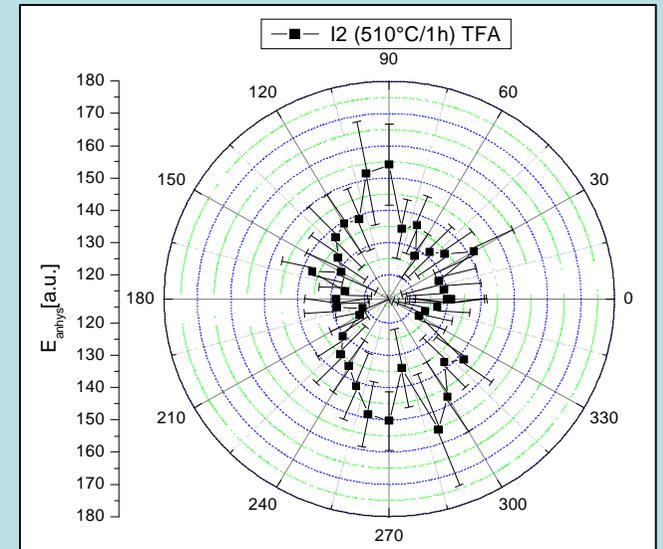
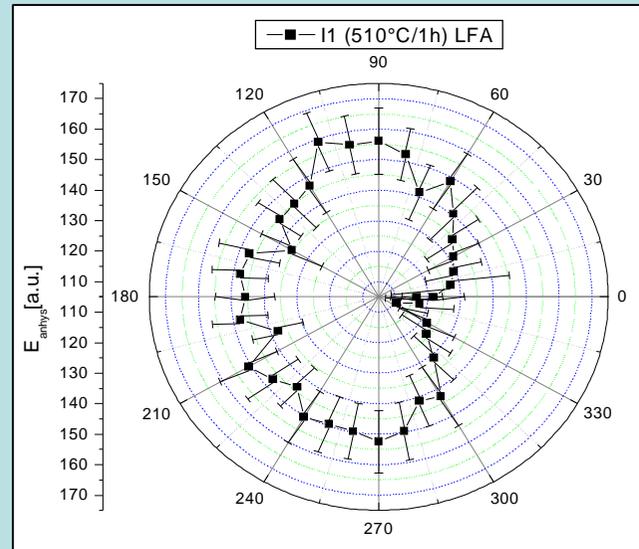
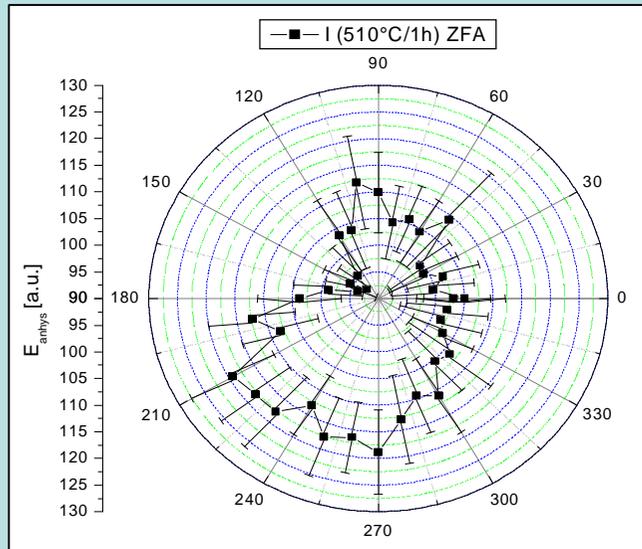
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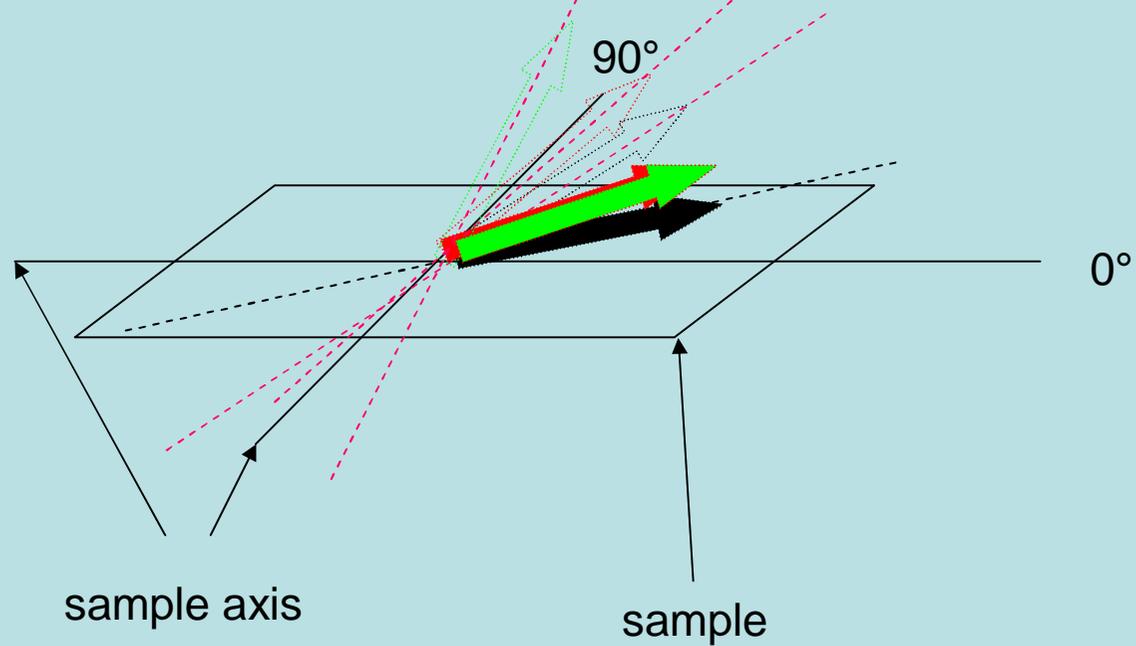
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# MOKE measurements



# MOKE measurements

RT	I - 510°C/1h			J - 550°C/1h		
	ZFA	LFA	TFA	ZFA	LFA	TFA
$\phi_1$ (deg) +/-1	53	57	55	51	46	37
$\phi_{yu}$ (deg) +/- 1	70	82	98	80	98	80



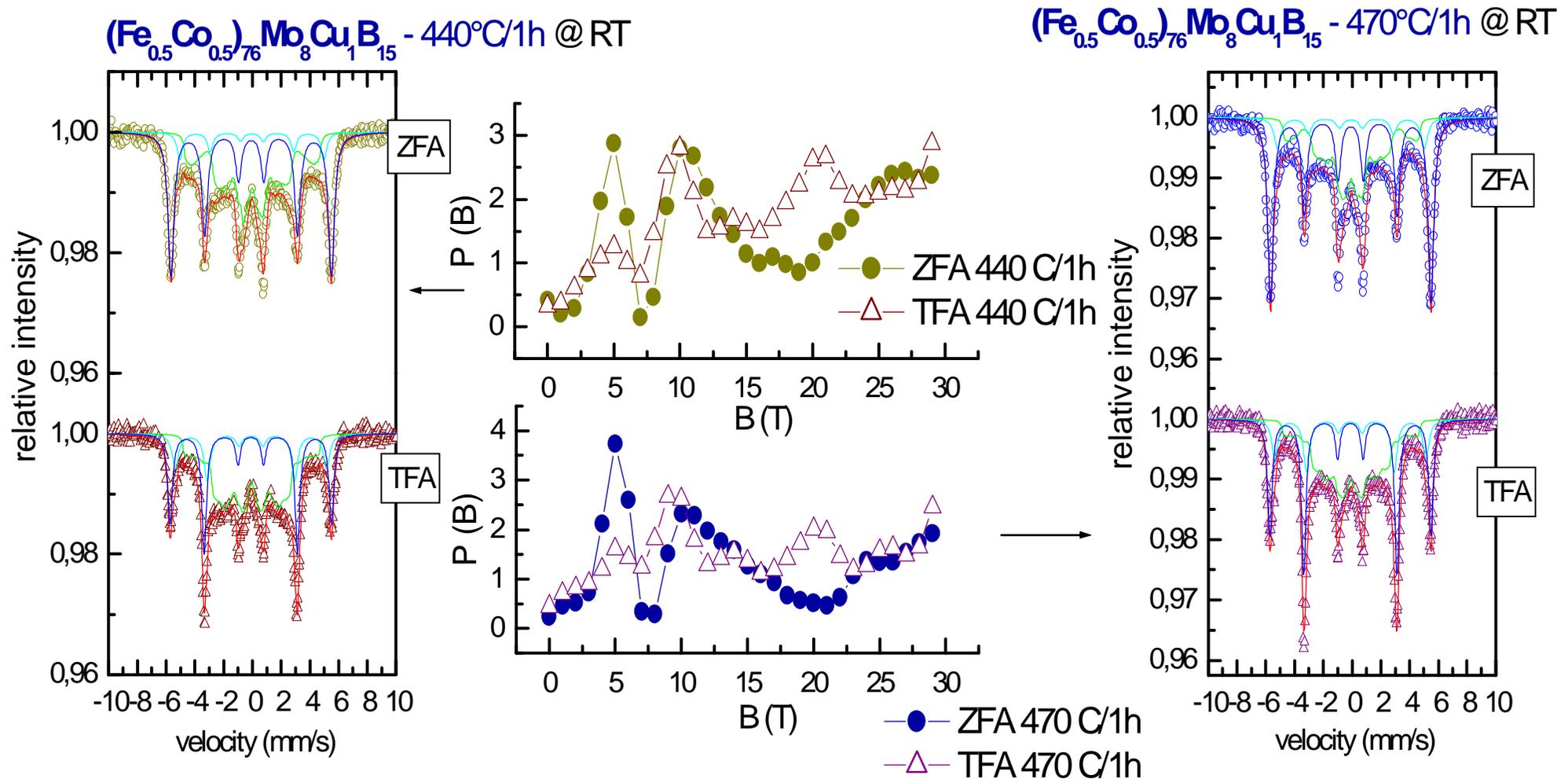
# Preliminary conclusion

- Results obtained showed that such a treatment caused not significant changes in magnetic texture for  $\text{Fe}_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  alloy, but slightly improved saturation magnetization and reduced coercive force.
- Some magnetic anisotropy effect is observed at the surface



### Mössbauer measurements

- We can expect more pronounced effect of in-external magnetic field annealing
- Higher value of hyperfine magnetic field of crystalline component due to Co substitution



# (Fe<sub>0.5</sub>Co<sub>0.5</sub>)<sub>76</sub>Mo<sub>8</sub>Cu<sub>1</sub>B<sub>15</sub>

## Mössbauer measurements – hyperfine parameters

	440°C/1h			470°C/1h		
RT	ZFA	LFA	TFA	ZFA	LFA	TFA
$\langle B_{am} \rangle$ (T) $\pm 0.1$	16.4		17.2	14.5		15.6
$\langle \sigma_{am} \rangle$ (T) $\pm 0.1$	8.4		7.8	8.5		8.2
$\langle \delta_{am} \rangle$ (mm/s) $\pm 0.01$	0.08		0.04	0.05		0,02
$B_{cr}$ (T) $\pm 0.1$	34.6		34.8	34.6		34.7
$B_{if}$ (T) $\pm 0.1$	31.1		32.6	31.7		32.6
$A_{am}$ (%) $\pm 2.5$	46		52	39		45
$A_{cr+if}$ (%) $\pm 2.5$	54		48	61		55
A23	2.13		4.0	1.56		4.0
$\Theta$ (deg)	<b>56</b>		<b>90</b>	<b>49</b>		<b>90</b>

## Conclusion

- Using magnetic as well as Mössbauer effect measurements performed in external magnetic field at room temperature and at 77 K enabled us to reveal magnetic texture of the  $\text{Fe}_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  nanocrystalline alloy.
- Obtained results were compared with those achieved on zero field annealed samples.
- We have found no direct correlation between the amount of nanocrystalline phase, the applied external magnetic field, and the magnetic texture.
- This might be due to:
  - (1) composition of the studied alloy,
  - (2) low external magnetic fields applied, and/or
  - (3) rather well stabilised spin texture of the annealed samples as the annealing temperatures used are close to those which show optimal magnetic properties.
- Further investigations employing MOKE experiments showed some induced anisotropy.

## Conclusions

- Pronounced effect of in-field annealing was observed for  $(\text{Fe}_{0.5}\text{Co}_{0.5})_{76}\text{Mo}_8\text{Cu}_1\text{B}_{15}$  where magnetisation turned into the ribbon plane

- Thank you for your attention