

# OSSM

XIII Ogólnopolskie Seminarium  
Spektroskopii Mössbauerowskiej

16-19 czerwca 2024

## ABSTRAKTY & PROGRAM



PATRONAT HONOROWY



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UNIwersytetu Śląskiego  
W KATOWICACH

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XIII

Ogólnopolskie Seminarium  
Spektroskopii Mössbauerowskiej



PROGRAM KONFERENCJI

&

ABSTRAKTY

16-19 czerwca 2024



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XIII

*Ogólnopolskie Seminarium  
Spektroskopii Mössbauerowskiej*

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- *Dr hab. Tadeusz Szumiata*
- *Prof. Vinh Hung Tran*

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- **dr hab. Aneta Hanc-Kuczkowska** – Instytut Inżynierii Materiałowej, Wydział Nauk Ścisłych i Technicznych

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- **dr hab. Jacek Krawczyk** (sekretarz) – Instytut Inżynierii Materiałowej, Wydział Nauk Ścisłych i Technicznych
- **dr inż. Robert Paszkowski** (sekretarz) – Instytut Inżynierii Materiałowej, Wydział Nauk Ścisłych i Technicznych

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- **dr hab. Dariusz Malczewski** – Instytut Nauk o Ziemi, Wydział Nauk Przyrodniczych
- **dr Maria Dziurawicz** – Instytut Nauk o Ziemi, Wydział Nauk Przyrodniczych

## *Drodzy Uczestnicy*

### *XIII Ogólnopolskiego Seminarium Spektroskopii Mössbauerowskiej*

*Witamy na kolejnym spotkaniu Społeczności mössbauerowskiej, złożonej z Naukowców stosujących w badaniach o znaczeniu poznawczym jak i aplikacyjnym, wyjątkową technikę eksperymentalną opartą na zjawisku Mössbauera.*

*Stosowanie spektroskopii mössbauerowskiej w Polsce zapoczątkował Profesor Andrzej Hrynkiewicz, który trzy lata po odkryciu zjawiska Mössbauera zarejestrował sygnał mössbauerowski za pomocą własnoręcznie zbudowanej aparatury w Instytucie Fizyki Uniwersytetu Jagiellońskiego. Zainicjowane wówczas wykorzystanie tej spektroskopii w badaniach materii skondensowanej trwa do chwili obecnej, otwierając nowe możliwości naukowego poznania w wielu obszarach badawczych. Obecnie w Polsce spektroskopia mössbauerowska jest aktywnie rozwijana w ośrodkach naukowych w: Białymstoku, Częstochowie, Katowicach, Krakowie, Lublinie, Radomiu, Warszawie i Wrocławiu.*

*W 1996 roku, dzięki zaangażowaniu Prof. Mieczysława Budzyńskiego, ośrodek lubelski zainicjował organizację Ogólnopolskich Seminariów Spektroskopii Mössbauerowskiej (OSSM), umożliwiających prezentację aktualnych trendów w badaniach z wykorzystaniem spektroskopii efektu Mössbauera, najnowszych osiągnięć, wymianę doświadczeń oraz nawiązywanie współpracy. Seminarium odbywa się cyklicznie, co dwa lata, a organizacją spotkań zajmują się grupy badawcze z różnych ośrodków naukowych w Polsce, które stosują tę metodę badawczą.*

*Kolejna, XIII edycja Seminarium przygotowana została przez grupę pracowników Uniwersytetu Śląskiego w Katowicach z Wydziału Nauk Ścisłych i Technicznych oraz Wydziału Nauk Przyrodniczych. Do udziału w Seminarium zaprosiliśmy Badaczy, którzy w pracy naukowej stosują i rozwijają metodę spektroskopii mössbauerowskiej oraz wszystkich zainteresowanych, chcących poznać aktualną tematykę badań prowadzonych tą metodą. Do czynnego udziału w Seminarium zaprosiliśmy również Doktorantów i Studentów, stwarzając możliwość poszerzenia wiedzy, doskonalenia metodologii badań oraz konstruktywnej dyskusji uzyskanych rezultatów badań własnych.*

*Drodzy Uczestnicy XIII Ogólnopolskiego Seminarium Spektroskopii Mössbauerowskiej, jesteśmy przekonani, że pobyt w samym sercu Jury Krakowsko-Częstochowskiej zaowocuje nowymi pomysłami naukowymi, a urokliwa przyroda Perły jurajskich krajobrazów, przyczyni się do regeneracji sił witalnych.*

*Mamy nadzieję, że OSSM 2024 pozostanie w Waszej pamięci jako owocne i sympatyczne wydarzenie naukowe.*

*Organizatorzy*

*XIII OSSM*





**Poniedziałek**  
**17 czerwca 2024**

## Notatki

- 9:00**                    **Inauguracja**
- prof. dr hab. Mieczysław Budzyński:** Spektroskopia mössbauerowska w Polsce i konferencje OSSM
- prof. dr hab. S. Dubiel** - wykład inauguracyjny XIII OSSM: Effect of magnetism on lattice vibrations
- Sesja I**
- 11:00 – 11:30    **prof. dr hab. Kvetoslava Burda:** Effects of TiO<sub>2</sub> nanoparticles on erythrocytes functionality
- 11:30 – 12:00    **dr hab. Jolanta Gałązka-Friedman:** Role of Mössbauer spectroscopy in smashing myths related to mechanisms of neurodegeneration
- 12:00 – 12:20    **dr Joanna Fiedor:** Interplay between carotenoids, nanoparticles and red blood cells - what can we learn from Mössbauer study
- 12:20 – 12:40    **dr Mariola Kądziołka-Gaweł:** Identification of iron in oral iron pharmaceuticals: Mössbauer spectroscopy study
- 12:40 – 13:00    **dr Katarzyna Dzedzic-Kocurek:** Magnetic moment of iron in oxyhaemoglobin
- 13:00 – 14:30**    **Obiad**
- Sesja II**
- 14:30 – 15:00    **prof. dr hab. inż. Artur Blachowski:** <sup>57</sup>Fe Mössbauer studies of Fe<sup>2+</sup>-phyllsilicates dehydrogenation and Fe-bearing minerals in gas/oil source rocks
- 15:00 – 15:30    **dr hab. Tadeusz Szumiata:** Metallic iron evidence in dust from ventilation system - Mössbauer, synchrotron and magnetic analysis
- 15:30 – 15:50    **dr inż. Jan Michalik:** Mossbauer spectroscopy studies of the magnetic fraction of the particulate matter emitted from coal-fired power plants and related to urban transport
- Sesja III**
- 16:00 – 16:30    **dr Tomasz Piłkuła:** The structure and magnetic properties of selected multiferroics based on BiFeO<sub>3</sub>
- 16:30 – 16:50    **dr inż. Karolina Siedliska:** Structure and magnetic properties of Bi<sub>0.9</sub>La<sub>0.1</sub>FeO<sub>3</sub> prepared by the sol-gel method with subsequent thermal treatment
- 16:50 – 17:10    **mgr inż. Jakub Grotel:** Structural, magnetic and electric properties of Co-doped Bi<sub>5</sub>Ti<sub>3</sub>FeO<sub>15</sub> Aurivillius phases
- 17:10 – 17:30    **dr Vitaliy Bilovol:** Estimating recoilless f-factor in nickel ferrite
- 19:00**                    **Uroczysta kolacja**

## Notatki



# Impact of magnetism on lattice vibrations

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According to the standard theory (ST) of the electron-phonon interaction (EPI) the effect of magnetism on EPI is negligible, because the so-called small adiabatic parameter equal to the ratio between the Debye and the Fermi energy is of the order of  $10^{-2}$  [1 and references therein]. Kim, however, reported that the impact of the EPI on the spin susceptibility of metals can be enhanced by two orders of magnitude in the case of delocalized magnetism [1]. In other words, the effect of the EPI on magnetic properties of metallic systems, and vice versa, should be much stronger than the ST-prediction. There are results obtained recently by calculations and/or measurements that are at variance with these predictions. In particular, M. S. Lucas et al. wrote [2]: “The phonon densities of states of bcc Fe-V alloys across the full composition range were studied by inelastic neutron scattering, nuclear resonant inelastic x-ray scattering, and ab initio calculations. Changes in the PDOS were revealed at crossing the Curie temperature”, B. Alling et al. based on disordered local moments molecular dynamics calculations came to conclusion that [3]: “Lattice vibrations strongly affect the distribution of local magnetic moment in experimental evidence paramagnetic Fe”, and I. S. Tupitsyn et al. [4] communicated that: “This theory (ST) neglects the effect of magnetism on lattice dynamics and fails to explain enhancement of the critical temperature in phonon-mediated superconductors.”

In this lecture I will present results acquired by means of  $^{57}\text{Fe}$  Mössbauer spectroscopy (MS) on  $\sigma$ -phase Fe-Cr, Fe-V and Fe-Cr-Ni,  $\lambda$ -phase  $\text{NbFe}_2$  and Fe-As compounds as well as with  $^{119}\text{Sn}$  MS and Nuclear resonance inelastic X-ray scattering (NRIXS) on metallic Cr. The common feature of all these systems is delocalized (itinerant) magnetism. The results give clear evidence that the lattice dynamics in the magnetic state of the investigated samples is significantly different than the one in the paramagnetic state. The latter means that the effect of magnetism, at least in the studied samples, on lattice vibrations cannot be neglected. Details on the issue can be found elsewhere [5].

- [1] D. J. Kim, *Phys. Rev. B*, 25 (1982) 6919
- [2] M. S. Lucas et al., *Phys. Rev. B*, 82 (2010) 144306
- [3] J. B. Alling et al., *Phys. Rev. B*, 93 (2016) 224411
- [4] [4] I. S. Tupitsyn et al., *Phys. Rev. B*, 94 (2016) 155145
- [5] S. M. Dubiel, *J. Magn. Magn. Mater.*, 561 (2022) 169688

## Notatki

## Effects of TiO<sub>2</sub> nanoparticles on erythrocytes functionality

K. Burda<sup>1</sup>, M. Peter<sup>1</sup>, J. Fiedor<sup>1</sup>, A. Siniarski<sup>2,3</sup>, J. Kiecana<sup>2,3</sup>, G. Gajos<sup>2,3</sup>  
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Over the last two decades, nanoparticles (NPs) have been at the centre of interest in nano(bio)technologies. TiO<sub>2</sub> NPs are among the most widely used nanomaterials today because of their unique electronic, photonic, catalytic and therapeutic properties [1]. The wide range of TiO<sub>2</sub> NPs in use in our daily lives results in a particularly high exposure of humans to their effects. The aim of the study was to evaluate their potential toxicity to red blood cells. In particular, the use of Mössbauer spectroscopy allowed us to follow the changes in the haemoglobin types inside the erythrocytes under the influence of their treatment with TiO<sub>2</sub> NPs. We also observed the ability of haemoglobin to reversibly bind oxygen using a method we developed [2].

Results will be presented for selected concentrations of TiO<sub>2</sub> NPs, for which we also observed osmotic changes in erythrocyte membranes related to changes in their membrane skeleton.

Acknowledgements:

The work was supported by the National Science Centre, Poland grant 2019/33/B/NZ7/02724.

[1] R. Javed, N. Ain, A. Gul, M.A. Ahmad, W. Guo, Q. Ao, S. Tian, *IET Nanobiotechnol.* 16 (2022) 171–189

[2] M. Kaczmarska, M. Fornal, F.H. Messerli, J. Korecki, T. Grodzicki, K. Burda, *Cell Biochem Biophys.* 67 (2013) p.1089

## Notatki

## Role of Mössbauer spectroscopy in smashing myths related to mechanisms of neurodegeneration

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Neurodegeneration is a process of progressive death of nervous cells in human brain structures – substantia nigra (SN) in Parkinson's disease (PD), SN and globus pallidus (GP) in progressive supranuclear palsy (PSP), and hippocampal cortex (HIP) in Alzheimer disease (AD). All these structures contain high concentrations of iron, comparable to that in the liver. Several studies claimed a significant increase of iron in SN in PD compared to control with a change of the ratio  $Fe^{2+}/Fe^{3+}$  (3:1 vs. 1:1). Our Mössbauer spectroscopy studies performed in collaboration with Racah Institute of Physics in Jerusalem have shown that over 95% of human brain iron is  $Fe^{3+}$  iron bound to ferritin, and only in PSP there is an increase of the concentration of iron compared to control (see table) [1]. Additional studies with the use of the ELISA technique demonstrated various dysfunctions of ferritin in these diseases [2]. These results were further confirmed by NMR studies in living subjects [3].

Disease	Tissue	Status	No of samples	Iron concentration (ng/mg)	Disease/control ratio
PD	SN	Disease	17	177±18	1.00±0.13
		Control	29	177±14	
PSP	GP	Disease	10	257±19	1.40±0.20
		Control	12	183±22	
PSP	SN	Disease	10	301±26	1.60±0.23
		Control	9	188±22	
AD	HIP	Disease	10	66±13	1.47±0.44
		Control	10	45±10	

- [1] Galazka-Friedman J, Bauminger ER, Friedman A- Iron in neurodegeneration. In: Sharma VK, Klingelhöfer G, Nishida T (eds.) Mössbauer spectroscopy- Applications in chemistry, biology and nanotechnology. Wiley 2013, New York; 324-332
- [2] Friedman A, Arosio P, Finazzi D, Kozirowski D, Galazka-Friedman J- Ferritin as an important player in neurodegeneration, *Park Rel Disord.* 2011; 17: 423-430
- [3] Kulinski R, Bauminger ER, Friedman A, Duda P, Galazka-Friedman J- Iron in typical and atypical parkinsonism – Mössbauer spectroscopy and MRI studies. *Hyperfine Interactions* 2016; 237: 4

## Notatki

## **Interplay between carotenoids, nanoparticles and red blood cells – what can we learn from Mössbauer study**

J. Fiedor<sup>1</sup>, A. Siniarski<sup>2,3</sup>, G. Gajos<sup>2,3</sup>, N. Spiridis<sup>4</sup> and K. Burda<sup>1</sup>

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Carotenoids constitute a group of widespread natural pigments produced by all photosynthetic organisms, as well as some prokaryotes and fungi. By acting as efficient antioxidants, reducing the risk of several chronic diseases and cancer, and serving as precursors of vitamin A, they turned out to be important dietary compounds [1, 2]. Carotenoids can be dispersed in hydrophobic regions of the membranes of red blood cells. Incorporated into model bilayers, they were shown to affect their physicochemical properties. Some of them were suggested to act as inhibitors of hemoglobin oxidation in reactions induced by exogenous radical generators [3, 4].

The objective of our study was to explore the effect of model carotenoids on the stability and functioning of red blood cells in the presence and absence of nanoparticles. Emphasis was placed on interactions resulting in changes in hemoglobin-oxygen affinity. To monitor the response of red blood cells, microscopic and spectroscopic techniques were applied including UV-VIS absorption and Mössbauer spectroscopies. Our results indicate that carotenoids can modulate the physicochemical properties of red blood cells. Furthermore, they affect the molecular parameters of hem-iron and its ability of efficient oxygen binding and releasing.

### Acknowledgements:

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[1] J. Fiedor and K. Burda, *Nutrients* 6 (2014) p. 466

[2] T. Bohn, M.L. Bonet, P. Borel, J. Keijer, J.F. Landrier, I. Milisav, J. Ribot, P. Riso, B. Winklhofer-Roob, Y. Sharihi and J. Corte-Real, *Nutrition Research Reviews* 34 (2021) p. 276

[3] S.T. Omaye, N.I. Krinsky, V.E. Kagan, S.T. Mayne, D.C. Liebler and W.R. Bidlack *Fundamental and Applied Toxicology*, 40 (1997) p. 163

[4] R.C. Chiste, M. Freitas, A.Z. Mercadante and E. Fernandes *Life Sciences* 99 (2014) p. 52

## Notatki



## Identification of iron in oral iron pharmaceuticals: Mössbauer spectroscopy study

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Iron plays a significant role in the living systems. It is an integral part of many proteins and enzymes and a mineral that works with other substances to create hemoglobin. Iron deficiency is one of the best-known forms of nutritional disorders [1]. It occurs when there is a negative balance between iron absorption and iron requirements and losses. Iron deficiency is caused not only by iron-deficient diets but also by the low iron bioavailability of the diet. Pregnant women, infants, young children, and adolescents have higher iron requirements and thus have a greater risk of developing its deficiency [2]. Iron deficiency causes anemia and other pathological changes in the body. The usual treatment for these anemias is the administration of oral hematinics, the common ones being ferrous fumarate, gluconate, and sulfate [3]. These are effective and usually relatively well tolerated, except for some gastrointestinal side effects, including constipation, diarrhea, and epigastric pain [3-5].

More and more Fe pharmaceuticals are available on the market, and they can be easily purchased without a prescription from a doctor. Moreover, the choice of a product is often determined by its price. Knowledge about the content of individual elements, particularly iron, and its state in such products is important for our health. Therefore, the study presents research on selected oral iron pharmaceuticals using Mössbauer spectroscopy and the X-ray fluorescence methods.

[1] N. Abbaspour, R. Hurrell, R. Kelishadi. *Journal of Research in Medical Sciences* 2014, 19(2), 164.

[2] M. Umang, R. Mrinmoy, S. Manmath, C. Pintu. *Food Bioengineering* 2023, 2, 53.

[3] P. Arredondo, C. Barrero, K. Garcia, J. Greneche. *Polyhedron* 2016, 105, 27.

[4] A. Souza, M. Batista Filho, C. Bresani, L. Ferreira, J. Figueiroa. *Cadernos de Saúde Pública* 2009, 25, 1225.

[5] Z. Tolkien, L. Stecher, A. Mander, D. Pereira and J. Powell. *PloS ONE* 2015, 10(2): e0117383.

## Notatki

## Magnetic moment of iron in oxyhaemoglobin

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Because of its wide appearance and its important role in nature, the mechanism of Fe-O<sub>2</sub> bonding in hemoproteins has been debated for decades [1-3]. Mössbauer spectrum of oxidized erythrocytes recorded at 5 K in the external magnetic field of 8 T shows a hyperfine field at <sup>57</sup>Fe nuclei equal to 8.6 T, i.e. 7.5 % higher than the applied one. This points out to a residual magnetic moment of nominally low spin Fe<sup>2+</sup> state in oxyhaemoglobin, assigned to the average spin of 0.17. We interpret this result as a consequence of a fast relaxation between the ground low LS (S=0) and excited intermediate IS spin (S=1) states.

The sign of the electric field gradient (EFG) tensor at Fe nuclei, i.e. the sign of the second derivative of the potential is negative, i.e. opposed to this in the myoglobin, with high spin state HS Fe<sup>2+</sup>, where the EFG is of “valence origin” predominantly produced by one 3d electron in 3d<sup>6</sup> configuration of HS Fe<sup>2+</sup>. Thus, the positive sign of EFG in the studied case suggested its lattice origin, as expected for LS Fe<sup>2+</sup>.

At the present stage, we postulate the following mechanism of oxygen bonding in oxyhaemoglobin:

1. the ground state of iron is LS Fe<sup>2+</sup> with neutral O<sub>2</sub> molecule in excited singlet state;
2. with increasing temperature, Fe gradually transforms to intermediate state IS S=1 jointly with singlet to triplet state transition of O<sub>2</sub>, antiferromagnetically coupled to Fe ion [4];
3. the temperature gradually induces spin transition, as in many metalloorganic iron compounds [5].

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[5] M. Magott, K. Płonka, B. Sieklucka, K. Dziedzic-Kocurek, W. Kosaka, H. Miyasaka and D. Pinkowicz, Chem. Sci. 14 (2023), 9651.

## Notatki

## <sup>57</sup>Fe Mössbauer studies of Fe<sup>2+</sup>-phyllosilicates dehydrogenation and Fe-bearing minerals in gas/oil source rocks

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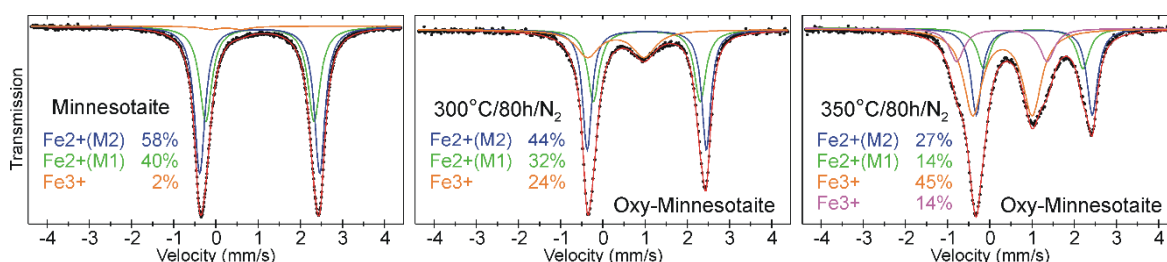
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The oxidation in Banded Iron Formation (BIF) is debated between biotic and abiotic, and/or primary and secondary oxidation. In contrast to the model of oxidation by incorporation of oxygen, Fe<sup>2+</sup> oxidation of phyllosilicates is driven by a thermally induced dehydrogenation reaction. Dehydrogenation does not require the presence of external oxygen and occurs via electron transfer between OH<sup>-</sup> and Fe<sup>2+</sup>, resulting in H<sub>2</sub> and H<sub>2</sub>O release during heating. <sup>57</sup>Fe Mössbauer spectroscopy was used to study dehydrogenation during thermal decomposition of minnesotaite and chlorite, as a potential abiotic secondary oxidation reaction of Fe<sup>2+</sup>-rich silicates in BIFs. The redox state of iron, structural changes, and the alteration products were studied. The results may be important in the context of current interest in the potential H<sub>2</sub> generation from BIFs and the oxidative dehydrogenation of Fe<sup>2+</sup>-bearing minerals on Mars.



**Fig. 1.** <sup>57</sup>Fe Mössbauer spectra of minnesotaite Fe<sub>3</sub>Si<sub>4</sub>O<sub>10</sub>(OH)<sub>2</sub> measured ex-situ at room temperature before and after isothermal heating at 300°C and 350°C for 80h under N<sub>2</sub> inert atmosphere conditions. Subspectra of octahedral Fe<sup>2+</sup> in site M1 (*cis*) / M2 (*trans*) and trivalent Fe<sup>3+</sup> are shown.

Additionally, the application of <sup>57</sup>Fe Mössbauer spectroscopy to the characterization of source rocks in geophysical well-logging studies of gas and oil shale will be presented.

### Acknowledgements

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## Notatki

## **Metallic iron evidence in dust from ventilation system - Mössbauer, synchrotron and magnetic analysis**

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In this work, the evidence of metallic iron in ventilation system dust has been performed. The presence of metallic iron can be regarded as an indicator of anthropogenic and technogenic "fresh activity" - contrary to the case of environmental samples from postindustrial regions [1,2].

Numerous experimental techniques have been applied for the characterization of the samples (raw ones and magnetic separates): transmission Mössbauer spectrometry, SEM-EDS, XRF-WDS, TEM, XRD, magnetometry and synchrotron XAS – NEXAFS.

In the Mössbauer spectra, several phases – typical for environmental dust – were identified, like hematite, magnetite and aluminosilicates with Fe<sup>3+</sup> and Fe<sup>2+</sup> sites [1,2]. However, two other subspectra could be assigned to the metallic iron as well as to the metallic  $\alpha$ -Fe and iron carbides from steel. This could be a natural consequence of the fact that the ventilation system operated in the vehicle diagnostic laboratory hall (in the Faculty of Transport, Electrical Engineering and Computer Science at Casimir Pulaski Radom University, Poland). Unfortunately, clear and direct interpretation was not possible because the corresponding subspectra were very smeared – possibly due to the small size of the strongly defected grains. Such an explanation was confirmed by the results of thermomagnetic measurements (from liquid helium to room temperature), especially owing to the hysteresis loops decomposition procedure.

The synchrotron radiation absorption experiment (XAS – NEXAFS) indicated, first of all, the augmented intensity of the first L3 subline of hematite due to the presence of pure Fe<sup>3+</sup>. However, for lower energies an absorption tail was observed, which strongly supports the presence of metallic iron in the tested samples from ventilation installations.

T. Magiera, B. Górka-Kostrubiec, T. Szumiata and M. S. Bućko, *Science of The Total Environment* 865 (2023), p. 161186

B. Górka-Kostrubiec, R. Świetlik, T. Szumiata, S. Dytłow, M. Trojanowska, *Journal of Environmental Sciences* 124 (2023), p. 875

## Notatki



## **Mössbauer spectroscopy studies of the magnetic fraction of the particulate matter emitted from coal-fired power plants and sources related to urban transportation (tram tracks, streets)**

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In the current contribution, we are presenting the results of Mössbauer Spectroscopy supported by Scanning and Transmission Electron Microscopy with Energy Dispersive X-ray Spectroscopy and chemical analysis showing an important variety of compositions, structures and morphology of the micro- and nanoparticles. Contrary to the previous studies, we have focused on research material coming directly from emission sources (sampling was accomplished in industrial plants from flue gases behind air pollution control installations and from brake pads and disks testing installations). Also, the magnetic fraction of samples from tram tracks and street dust samples were investigated.

Due to a large variety of parameters of fuel combustion in a power plant (characteristics of coal, its initial processing, e.g. milling; technology of combustion (pulverized fuel boiler or fluidized bed boiler), distribution and gradient of process temperatures; air pollution control devices) a large variety of composition, crystal structure and morphology was expected. We have found an important correlation between the combustion parameters and the properties of the emitted dust. It is also worth noticing the complexity of the internal structure of the micro- and nanoparticles, especially when it comes to iron and iron oxide crystallites.

Peculiar morphological type of particles related to the shearing processes was observed in samples from tram tracks, often containing elongated Fe-rich forms (oxides or slightly oxidized metallic Fe) embedded in Si, Fe and O-rich fine-grained material with the orientation of some components parallel to the orientation of Fe-rich forms.

Dust from streets is dominated by quartz and various silicates and aluminosilicates as well as phosphates, sulphides and oxides. The presence of ferrihydrates, sulphates and green rust-related components is also suggested by Mössbauer spectroscopy.

## Notatki

## The structure and magnetic properties of selected multiferroics based on BiFeO<sub>3</sub>

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In spite of being studied for several decades, bismuth ferrite still continues to garner attention from the scientific community. It is a perovskite-like multiferroic that combines ferroelectricity with a canted antiferromagnetic order in a single phase at room temperature. This unique feature makes BiFeO<sub>3</sub> interesting not only from the basic research point of view but also due to numerous, possible applications like 4-state digital memory or rapid, voltage-based reading of magnetic memory. Bismuth ferrite also exhibits a long-range spin cycloid propagating in the [10-1] pseudocubic direction with a period equal to about 62 nm. This leads to a peculiar, asymmetrical shape of the Mössbauer spectrum being typically registered for BiFeO<sub>3</sub>.

It has been demonstrated numerous times that doping of BiFeO<sub>3</sub> or producing bismuth ferrite in the form of thin films or nanoparticles can destroy the cycloidal spin ordering [1]. However, the exact mechanism behind this destruction is still a topic of debate. We studied a few series of BiFeO<sub>3</sub> samples doped with rare earth (RE) ions like Nd, La, Pr, and Sm as well as nanocrystalline BiFeO<sub>3</sub> prepared by sol-gel synthesis. The influence of the RE ions concentration on the structure and magnetic properties was investigated using X-ray diffraction, Mössbauer spectroscopy, electron microscopy, and magnetometry. Mössbauer spectra were fitted with the model assuming both HMF and QS distributions as a consequence of cycloidal spin ordering. Interestingly, it turns out that the drop in crystal grain size below the cycloid period does not destroy the cycloidal ordering of magnetic moments. Despite that, the weak magnetic properties appear gradually with the lowering of  $d$  as well as with the increase of RE concentration.

The second set of samples briefly discussed within this presentation is the paramagnetic Aurivillius compound Bi<sub>5</sub>Ti<sub>3</sub>FeO<sub>15</sub>. It is shown that substituting at least 20% of Fe ions with Co ions leads to the emergence of weak ferromagnetism at room temperature.

This presentation will be a kind of summarization of the main author's research in the field of BiFeO<sub>3</sub>-based multiferroics carried out over recent years.

[1] T. Pikula *et al.*, *Acta Crystallogr. Sect. B Struct. Sci. Cryst. Eng. Mater.*, 79 (2023) p. 305–313.

## Notatki

## Structure and magnetic properties of $\text{Bi}_{0.9}\text{La}_{0.1}\text{FeO}_3$ prepared by the sol-gel method with subsequent thermal treatment

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$\text{BiFeO}_3$ , a perovskite bismuth ferrite, has garnered significant attention over the past few decades due to its exceptional physical properties. It is a multiferroic material that exhibits ferroelectricity and antiferromagnetism at room temperature with  $T_C = 1043$  K and  $T_N = 647$  K.  $\text{BiFeO}_3$  has an incommensurate cycloid spin structure with a periodicity of approximately 62 nm, which cancels out the macroscopic magnetization and hinders the observation of the linear magnetoelectric effect [1]. However, reducing the particle size has been found to be an effective way to suppress the cycloid structure and enhance the magnetic moment of  $\text{BiFeO}_3$ . Another efficient method to increase the magnetization in the  $\text{BiFeO}_3$  system is through ion substitution. For example, rare-earth ions, like La, Nd, Eu, or Sm, were used to partially replace Bi, which led to a boost in the room temperature ferromagnetism [2].

In this study, we investigated the impact of substituting  $\text{La}^{3+}$  cation on the structure and magnetic properties of  $\text{BiFeO}_3$ . We used the sol-gel method to prepare the material with the  $\text{Bi}_{0.9}\text{La}_{0.1}\text{FeO}_3$  formula and then annealed it at different temperatures ranging from 723 to 1073 K. X-ray diffraction analysis confirmed the formation of the rhombohedral  $\text{BiFeO}_3$ -like phase. However, the specimens annealed at 873 and 1073 K had some amount of parasite phases along with the desired solid solution. Mössbauer spectra were fitted with the model assuming the distribution of both HMF field and quadrupole shift as a consequence of cycloidal spin ordering. Finally, we measured the magnetization of the samples and found that the annealing temperature had a gradual effect on the appearance of a hysteresis loop. As the temperature decreased, we observed weak ferromagnetic behavior in fine-grained La-doped  $\text{BiFeO}_3$ .

[1] D. Lebeugle *et al.*, *Phys. Rev. B - Condens. Matter Mater. Phys.*, vol. 76, 2007, p. 024116.

[2] T. Pikula *et al.*, *Acta Crystallogr. Sect. B Struct. Sci. Cryst. Eng. Mater.*, vol. 79, 2023, pp. 305–313.

## Notatki

## Structural, magnetic and electric properties of Co-doped $\text{Bi}_5\text{Ti}_3\text{FeO}_{15}$ Aurivillius phases

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Aurivillius compounds, also known as Aurivillius phases, are ceramic materials with a multilayered crystal structure. They consist of perovskite- and fluorite-like layers stacked on top of one another in an alternating sequence. While Aurivillius phases are ferroelectric, it is also reported they may exhibit interesting magnetic properties. Of particular note is the room-temperature magnetoelectric effect. The aim of our study was to examine structural, magnetic and electric properties of  $\text{Bi}_5\text{Ti}_3\text{FeO}_{15}$  doped with Co ions (0-50% concentration). The idea behind the doping process was to modify magnetic properties and enhance the room-temperature magnetoelectric effect of the pure compound.

The magnetoelectric effect is a phenomenon which involves a coupling between magnetic and ferroelectric orders of a material. In other words, when a magnetoelectric material is placed in an external magnetic field, it becomes electrically polarized. On the other hand, if placed in an external electric field, the material becomes magnetized. The quantity that describes this relationship is known as the magnetoelectric coupling coefficient  $\alpha$ . Magnetoelectric materials have numerous potential applications as electronic or spintronic device components, e.g., transducers, logic gates, nonvolatile memory. There is a continuous search for materials with room-temperature magnetoelectric coupling that is sufficiently strong and easy to manipulate.

The structural analysis of our samples was carried out via X-ray diffractometry (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). The magnetic investigation involved vibrating sample magnetometry (VSM) measurements complemented with Mössbauer spectroscopy. Ferroelectric properties were studied with a Sawyer-Tower circuit. Finally, magnetoelectric properties were investigated via the lock-in technique.

To conclude, the samples were synthesized successfully using the standard ceramic method. The samples are ferroelectric and weakly ferromagnetic at room temperature. It has been confirmed that the doping process enhances the magnetic properties of the material and may produce a very weak room-temperature magnetoelectric coupling.

## Notatki



## Estimating recoilless $f$ -factor in nickel ferrite

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In the spinel  $\text{NiFe}_2\text{O}_4$ , the cations are arranged in two magnetic sublattices where the local oxygen environment is of a tetrahedral or octahedral type. The distribution of the cations in the two lattices defines the macroscopic magnetic response of the ferrimagnetic spinel, where  $\text{Fe}^{3+}$  and  $\text{Ni}^{2+}$  ions provide a magnetic moment of  $5 \mu_B$  and  $2 \mu_B$ , respectively. Proper estimation of the cation distribution becomes critical but can be accurately done using  $^{57}\text{Fe}$  Mössbauer spectroscopy, which is a considerably common technique for studying iron-containing solids, which requires knowledge of the recoilless  $f$ -factors ( $f_T$  and  $f_O$ ) of  $\text{Fe}^{3+}$  ions in both tetrahedral and octahedral sites.

We use a conventional method to calculate Mössbauer recoilless  $f$ -factors by measuring the temperature dependence of the isomer shifts. Applying the Debye approximation in the case of a lattice vibrational model, the Debye temperature  $\Theta$  of the individual sites was first estimated, and then the  $f$ -factors ( $f_O$  and  $f_T$ ) were extrapolated in the 5-300 K range. We show that the  $f_O/f_T$  ratio differs from unity in the whole temperature range. It can prevent a common error assumed by many researchers when calculating the inversion degree parameter (defined as a fraction of tetrahedral sites occupied by  $\text{Fe}^{3+}$  ions) of  $\text{NiFe}_2\text{O}_4$  and taking the  $f_O/f_T$  ratio to be unity.

## Notatki



XIII OSSM

**Wtorek**  
**18 czerwca 2024**

## Notatki

## Sesja IV

- 9:00 – 9:30 dr hab. Rafał Idczak: Coexistence of magnetism and superconductivity in 112-type iron pnictides  $\text{EuFeAs}_2$  doped with Co
- 9:30 – 9:50 dr inż. Karolina Czarnacka: Mössbauer spectroscopy studies of mechano-synthesized  $\text{Fe}_2\text{CrSi}$  and  $\text{Co}_2\text{FeAl}$  Heusler alloys
- 9:50 – 10:10 mgr inż. Anna Sławek: Badanie stopów wysokiej entropii na bazie  $\text{FeNiCoPd}$
- 10:10 – 10:30 mgr Piotr Sobota: Antiferromagnetic ordering in  $\text{VfNbTaMo}$  high entropy alloy at low temperatures

## 10:30 – 11:00 Przerwa

## Sesja V

- 11:00 – 11:30 dr hab. Zbigniew Surowiec: Synteza i właściwości nanocząstek magnetytu modyfikowanych powierzchniowo chitosanem
- 11:30 – 12:00 dr Piotr Fornal: New Model of Nanoparticles Mobility in Liquids
- 12:00 – 12:20 dr Wojciech Olszewski: The impact of synthesis conditions on the properties of iron oxide nanoparticles – temperature studies
- 12:20 – 12:40 mgr Sławomir Strzelec: Study of the influence of multi-wall carbon nanotubes with nickel ions on the functioning of red blood cells
- 12:40 – 13:00 mgr Władysław Gumiennik: Mössbauer and magnetometry study of magnetite like nanoparticles

## 13:00 – 14:30 Obiad

## Sesja VI

- 14:30 – 15:00 prof. dr hab. inż. Jakub Cieślak: Badania granatów żelazowych w silnych polach magnetycznych
- 15:00 – 15:30 dr hab. Dariusz Malczewski:  $^{57}\text{Fe}$  Mössbauer spectroscopy of annealed highly metamict perrierite: Activation energy for thermal recrystallization
- 15:30 – 15:50 mgr Martyna Jakubowska: Porównanie metod klasyfikacji chondrytów OC: klasycznej-opartej na zawartości fajalitu i ferrosilitu oraz 4M-opartej na spektroskopii Mössbauerowskiej
- 16:00 **Sesja w jurajskich plenerach**
- 19:00 **Kolacja**

## Notatki

## Coexistence of magnetism and superconductivity in 112-type iron pnictides EuFeAs<sub>2</sub> doped with Co

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Kamihara *et al.* in 2008 discovered superconductivity (SC) in layered iron arsenide (LaFeAsO<sub>1-x</sub>F<sub>x</sub>) with a transition temperature ( $T_C$ ) of 26 K [1]. This discovery prompted intensive study with the goal of creating novel iron-based superconductors with diverse crystal structures, high transition temperatures, and multiband superconductivity [2-4].

This contribution examines polycrystalline samples of Eu(Fe<sub>1-x</sub>Co<sub>x</sub>)As<sub>2</sub> with  $x = 0.05, 0.10,$  and  $0.13$  utilizing <sup>57</sup>Fe Mössbauer spectroscopy, magnetization, and electrical resistivity studies. The acquired results provide intriguing information on the interaction of magnetism with superconductivity in the investigated compounds at low temperatures. As previously demonstrated, all three compounds exhibit Eu<sup>2+</sup>-related long-range antiferromagnetic order below 40 K, and superconductivity in FeAs layers that develop deep within the antiferromagnetic phase. In the instance of Eu(Fe<sub>0.95</sub>Co<sub>0.05</sub>)As<sub>2</sub>, the <sup>57</sup>Fe Mössbauer spectroscopy data clearly show that this system experiences the Spin-Density-Wave (SDW) transition at 80 K, hence, the superconductivity observed below 18 K coexists with SDW and *4f*-europium magnetic order inside the same volume. In samples with stronger Co-concentration doping, the SDW order is completely suppressed, and superconductivity occurs at a significantly higher critical temperature, around 28 K.

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## Notatki



## Mössbauer spectroscopy studies of mechanosynthesized Fe<sub>2</sub>CrSi and Co<sub>2</sub>FeAl Heusler alloys

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Heusler alloys are a rich family of materials which exhibit many interesting properties, e.g., half-metallic ferromagnetism, thermoelectricity, magnetoresistivity, exchange bias, shape memory effect, etc. The structure of the Heusler compounds is closely related to the preparation method. The classical arc-melting and non-equilibrium processes, like melt-spinning or mechanosynthesis, allow one to prepare a highly ordered crystalline structure of the Heusler alloys. Especially, nanostructured Heusler alloys prepared via mechanosynthesis can exhibit entirely new properties. In general, mechanical deformations and solid-state reactions taking place at the grain boundaries do not lead to the formation of the desired Heusler phase; therefore, additional heat treatment is often needed.

In the present work, two Heusler alloys, namely Fe<sub>2</sub>CrSi and Co<sub>2</sub>FeAl, were prepared by mechanosynthesis from a mixture of pure powders using a planetary ball-mill with stainless steel vial and balls. The samples for X-ray diffraction, vibrating sample magnetometry and Mössbauer spectroscopy studies were taken after 1, 2, 5, 10 and 20 h of milling. Then, the powders after 20 h were thermally treated via isothermal annealing at 300, 500 and 700 °C or continuous heating up to 700 °C with a heating rate of 20 °C min<sup>-1</sup>. In both cases, *bcc* solid solutions were formed after 20 h of milling with the average crystallite size of the order of 10-25 nm. After thermal treatment, the Fe<sub>2</sub>CrSi Heusler phase was crystallized together with small amounts of Cr<sub>3</sub>Si, FeCr and FeSi compounds, while in the case of Co<sub>2</sub>FeAl, the desired Heusler alloy was formed without secondary phases. Narrow hysteresis loops registered for both alloys confirmed their soft magnetic properties with magnetic moments 2.3 and 5.0 μ<sub>B</sub>/f.u. for Fe<sub>2</sub>CrSi and Co<sub>2</sub>FeAl, respectively. Mössbauer spectra of the obtained alloys exhibited hyperfine magnetic field distributions with fields between 9 and 28 T for Fe<sub>2</sub>CrSi and between 29 and 32 T for Co<sub>2</sub>FeAl. The observed distributions proved atomic disorder in the crystalline structure of the obtained Heusler alloys.

## Notatki

## Badanie stopów wysokiej entropii na bazie FeNiCoPd

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Stopy wysokiej entropii (High Entropy Alloys, HEAs) stanowią innowacyjną i zaawansowaną klasę materiałów wyróżniającą się wyjątkowo atrakcyjnymi właściwościami. HEAs wykazują wysoką wytrzymałość, twardość, odporność na korozję, pękanie oraz odporność na zmęczenie, a także stabilność w wysokich temperaturach. Co ważne, przewyższają one konwencjonalne stopy o kompleksowe właściwości, łączące w sobie zarówno wysoką wytrzymałość, jak i plastyczność. Właśnie te cechy sprawiają, że HEAs są przedmiotem intensywnych badań [1].

Warto zauważyć, że w praktyce większość analizowanych układów okazała się być układami wielofazowymi. Jedynym z wyjątków jest system FeNiCoPd, który okazał się być jednofazowy (krystalizujący w układzie *fcc*). Wstępne badania nad tym systemem wykazały ciekawe właściwości fizyczne związane z magnetyzmem: wzrost namagnesowania wraz ze wzrostem temperatury, wysoka temperatura uporządkowania magnetycznego oraz efekty związane z lokalnymi zniekształceniami sieci krystalicznej, powodowane przez różnorodność występujących atomów [2], [3].

Niniejszym, prezentujemy wyniki badań nad stopami FeNiCoPd-X (X = V, Cu, Ti, Mn oraz Al.). Wiodące techniki badawcze obejmowały metody dyfrakcyjne oraz spektroskopię mössbauerowską. Za pomocą spektroskopii mössbauerowskiej zmierzono widma w temperaturze pokojowej oraz w temperaturze ciekłego azotu. Na podstawie uzyskanych informacji stwierdzono, że większość z badanych stopów składa się z dwóch podwidm, co zgadza się z wynikami otrzymanymi za pomocą techniki SEM-EDX.

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## Notatki

## Antiferromagnetic ordering in VFeNbTaMo high entropy alloy at low temperatures

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High Entropy Alloys (HEAs) are solid solutions of five or more elements mixed in non-negligible proportions (more than 5 at.% of each element). They have recently attracted much attention because of their unique and promising physical properties. They form well-defined, simple, close packed structures (body centered cubic, face centered cubic or hexagonal). The name HEA comes from the large changes in configurational entropy during the formation of a solid solution from multiple elements, which was predicted by Yeh *et al.* [1]. Due to their structural features, HEAs are known for their exceptional mechanical properties, thermal stability, and corrosion resistance. These materials have great potential for various applications if their high durability is combined with more sophisticated phenomena, such as magnetic order and superconductivity.

We have synthesized VFeNbTaMo alloy by conventional arc melting and conducted characterization of it by several experimental methods: XRD diffractometry, SEM-EDXS microscopy, MPMS-SQUID magnetometry and <sup>57</sup>Fe TMS.

The analysis of XRD and SEM-EDXS results revealed the presence of two phases in the studied alloy. The magnetic and TMS measurements showed the unusual transition from paramagnetic to antiferromagnetic ordering at the low temperatures.

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## Notatki

## Synteza i właściwości nanocząstek magnetytu modyfikowanych powierzchniowo chitosanem

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Niemalające zainteresowanie nanokompozytami, złożonymi z części nieorganicznej i organicznej nadal przyciąga uwagę wielu zespołów badawczych. Poprzednie nasze badania wykazały, że nanocząstki magnetytu charakteryzują się obecnością fazy superparamagnetycznej już od temperatury 150 K w stopniu zależnym od związku modyfikującego powierzchnię [1,2]. Fakt ten oraz ciekawe doniesienia na temat jednoczesnego procesu syntezy i funkcjonalizacji powierzchni nanocząstek sprawiły, że podjęto próbę przygotowania nanocząstek magnetytu modyfikowanych chitosanem pod działaniem ultradźwięków. W tym celu przygotowano preparaty opłaszczane chitosanem, syntezowane w obecności ultradźwięków oraz stanowiące próbę kontrolną, bez udziału ultradźwięków.

Strukturę i morfologię otrzymanych nanocząstek scharakteryzowano za pomocą dyfrakcji rentgenowskiej i transmisyjnego mikroskopu elektronowego, wyznaczając ich średnie rozmiary i rozkłady wielkości. Informacji na temat aglomeratów i ich rozdywersyfikacji pod wpływem ultradźwięków dostarczyła technika DLS. Wpływ ilości surfaktantu oraz sposobu syntezy nanokrystalitów na właściwości magnetyczne przeanalizowano przy użyciu spektroskopii mössbauerowskiej, natomiast pomiary kalorymetryczne wykazały przydatność nanocząstek magnetytu ze zmodyfikowaną chitosanem powierzchnią do potencjalnych zastosowań hipertermii.

Wyniki badań mössbauerowskich pozwoliły stwierdzić, że większa ilość surfaktantu użytego do funkcjonalizacji powierzchni nanocząstek osłabia dipolowe oddziaływania między nimi, przyczyniając się tym samym do łatwiejszego przechodzenia w stan superparamagnetyczny. Dodatkowo w przypadku próbek syntezowanych z udziałem ultradźwięków, składowa superparamagnetyczna pojawia się w niższych temperaturach niż w przypadku próbek modyfikowanych tą samą ilością chitosanu, otrzymywanych metodą standardową.

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## Notatki



## New model of nanoparticles mobility in liquids

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For an object moving with a varying velocity  $v(t)$ , the probability of recording its particular velocity  $v$ ,  $p(v)$ , is given by Eq. 1.

$$p(v) = \left| \frac{dt(v)}{dv} \right| p(t) \quad (1)$$

Where  $t(v)$  is the reverse function of  $v(t)$ ,  $p(t) = \frac{1}{T} = f$ , where  $T$  is the time of movement duration or its period. For testing this formula we measured the single line Mössbauer spectrum of immobile  $\text{Na}_4\text{Fe}(\text{CN})_6$ , Fig. 1a, next the absorber moving sinusoidally,  $v(t) = v_0 \sin(2\pi ft)$  (here  $f = 100\text{Hz}$ ,  $t(v) = \frac{1}{2\pi f} \arcsin\left(\frac{v}{v_0}\right)$ ) in unsynchronized with the  $^{57}\text{Co}$  source with frequency 10Hz, Fig.1b. The spectrum was decomposed by 80 Lorentzian, which intensity is equal to  $p(v) = C \cdot \left(1 - \frac{v^2}{v_0^2}\right)^{-1/2}$ , see Fig. 2.

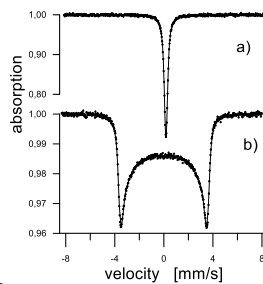


Fig.1

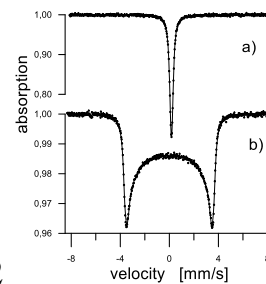


Fig.2

Next, we used this method to describe the character of hematite nanoparticles mobility in collagen gel. The obtained Mössbauer spectrum with corresponding  $p(v)$  distribution shows that the displacement of such particles may be described as the result of a superposition of fast, Brownian movement leading to the Gaussian broadening of the resonant line and rare, distant translation. During this “jump”, the particle velocity decreases exponentially,  $v(t) = v_0 e^{-t/\tau}$ , which leads to  $p(v) \sim 1/v$ . The estimates of  $v_0$ ,  $\tau$  and average distance of the individual translation are given, and compared with related values obtained for nanoparticles suspended in water solution of sucrose [1].

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## Notatki

## The impact of synthesis conditions on the properties of iron oxide nanoparticles – temperature studies

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Despite the similar chemical composition, nanoparticles can exhibit different optical, thermal or magnetic properties due to the synthesis method used [1]. This is because size, structural defects and surface termination largely determine nanoparticle characteristics. In addition, uncontrolled surface modification can occur, causing an increase in the average particle size and, for example, changes in the saturation magnetization value [2]. It has been shown that wide distribution of the nanoparticles impacts their reaction to the external magnetic field (both in dispersed and agglomerated forms) and to each other. In addition, various diameters, shapes and crystal phases of nanostructures significantly affect the reactivity as well as the efficiency of the reactions in which they may participate. These processes are directly related to the ratio of the number of surface atoms to the volume of atoms in nanoparticles, which drastically changes in the case of the nanometric size range. Surface to volume ratio determines not only the properties of the particles, but also allows for the development of the surface area available for chemical reactions. Therefore, the synthesis conditions should be appropriately selected properly to for the application.

In this work we will focus on iron oxides nanoparticles prepared in different synthetic procedures. The interdependence between features such as physicochemical properties of the core, specificity and modification of the surface, efficiency and selectivity of the occurring reactions lead to characteristic changes in the shape of the Mössbauer spectra of iron oxide nanoparticles. Analysis of such spectra will allow the authors to investigate the transformations of nanoparticles, including their degradation and the formation of other iron-containing species.

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## Notatki

## Study of the influence of multi-wall carbon nanotubes functionalised with nickel ions on the functioning of red blood cells

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Carbon nanotubes are attractive nanocarriers for a variety of biotechnological applications, in part due to their high mechanical strength and significant specific surface area, as well as their conductive properties [1]. Unfortunately, the potential applications of nanocarbon structures are severely limited by their toxicity. This depends on many factors, such as their structure, length-to-diameter ratio, possible functionalisation and the presence of impurities [2-3]. Systematic studies are therefore needed to understand how functionalised carbon nanotubes affect biological systems.

The research will be presented on the influence of multi-wall carbon nanotubes [4] functionalised with Ni<sup>2+</sup> ions on the stability of red blood cells, on changes in the state of the haemoglobin they contain and on their ability to carry oxygen.

Mössbauer spectroscopy is a sensitive tool for studying different forms of iron in haemoglobin [5-6].

Acknowledgements:

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## Notatki

## Mössbauer and magnetometry study of magnetite like nanoparticles

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The investigation of synthesis methods and the properties of superparamagnetic iron oxide nanoparticles (SPIONS) is still a relevant topic due to their applications in different sectors, from wastewater treatment [1] to biomedicine [2]. In our work, we investigated the structure and magnetic properties of magnetite-like nanoparticles obtained by the co-precipitation method [3] using X-ray powder diffraction,  $^{57}\text{Fe}$  Mössbauer spectroscopy, and DC magnetometry.

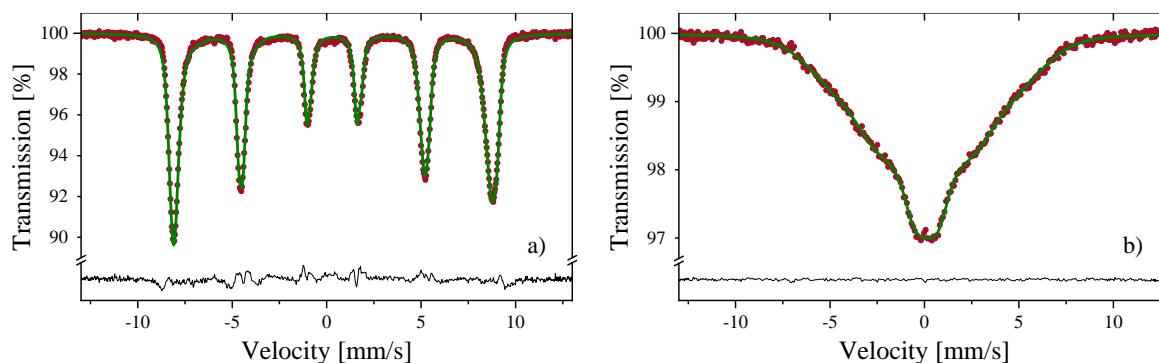


Fig.1  $^{57}\text{Fe}$  Mössbauer spectra of  $\text{Fe}_3\text{O}_4$  nanoparticles at temperatures:  
a) 4 K and b) 450 K

Room temperature X-ray diffraction measurements revealed a single phase magnetite-like structure with lattice parameter  $a=8.3706 \text{ \AA}$  and volume-averaged size of the crystallites of 7.6 nm. To characterize the magnetic properties of the compound, the DC (VSM) ZFC/FC susceptibility and magnetization measurements from 3 K up to 300 K and for the fields up to  $\pm 90\text{kOe}$  were performed.

The representative  $^{57}\text{Fe}$  Mössbauer spectra measured between 4 K and 450 K are shown in Fig.1. At 4K, a maghemite-type broadened asymmetric sextet is observed. Measurements in the high temperature range revealed a strongly relaxational nature of the spectra. Their analysis, assuming static distribution and dynamic relaxations models, allowed for determining temperature dependencies of significant parameters of hyperfine interactions, such as the average value of the hyperfine field, isomer shifts, or relaxation times.

The research project partly supported by the „Excellence Initiative – Research University” program for the AGH University of Krakow.

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## Notatki



## Badania granatów żelazowych w silnych polach magnetycznych

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Granaty żelazowe to układy o ogólnym wzorze  $\text{Fe}_2\text{Fe}_3\text{RE}_5\text{O}_{12}$ . Żelazo jest obecne na dwóch podsieciach, sprzężonych antyferromagnetycznie. Obecność trzeciej podsieci obsadzonej zwykle przez atom ziemi rzadkiej może skutkować ciekawymi własnościami magnetycznymi takiego układu. Namagnesowanie atomów RE jest zazwyczaj przeciwnie zorientowane do wypadkowego namagnesowania podsieci obsadzonych przez Fe. Ponieważ maleje ono z temperaturą znacznie szybciej, niż w podsieciach z żelazem, w pewnej charakterystycznej temperaturze zwanej temperaturą kompensacji,  $T_C$ , obserwuje się znikanie wypadkowego namagnesowania, co nie jest jednak spowodowane temperaturowym rozporządkowaniem momentów magnetycznych, ale znoszeniem się wpływów antyrównolegle zorientowanych podsieci RE i Fe. Ciekawe, choć rzadko obserwowane eksperymentalnie zjawiska zachodzą w okolicy  $T_C$ , w wysokich polach magnetycznych. Obserwowane jest wówczas *plateau* – obszar braku zależności namagnesowania od temperatury, zjawisko możliwe do wyjaśnienia na gruncie teorii antyferromagnetyzmu Neela.

W niniejszej pracy przedstawione zostaną wyniki pomiarów namagnesowania i pomiarów mössbaurowskich czterech próbek granatów, w zewnętrznym polu magnetycznym 9T. Omówione i wyjaśnione zostaną nieoczywiste cechy widma mössbauerowskiego oraz krzywych namagnesowania  $M(T)$ , przestawione zostaną także szczegółowo różne modele dotyczące wyjaśnienia tego zjawiska.

## Notatki

## **<sup>57</sup>Fe Mössbauer spectroscopy of annealed highly metamict perrierite: Activation energy for thermal recrystallization**

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This paper presents the results of <sup>57</sup>Fe Mössbauer spectroscopy and X-ray diffraction analysis of highly metamict perrierite (REE,Ca,Th)<sub>4</sub>(Fe<sup>2+</sup>,Mg)<sub>2</sub>(Ti,Fe<sup>3+</sup>)<sub>3</sub>Si<sub>4</sub>O<sub>22</sub> after annealing in argon from 673 to 1273 K for one hour. Metamict minerals contain radioactive elements that degrade their crystal structure over geologic time. Measurements were performed on fragments of a perrierite sample collected from granitoids near Amherst, Virginia (USA), dated at 1.1 Ga with a calculated absorbed  $\alpha$ -dose of  $7.8 \times 10^{15}$   $\alpha$ -decay/mg. Electron microprobe and gamma-ray spectrometry showed Fe concentration of 4.7 wt.% and Th and U concentrations of 0.64 and 0.06 wt.%, respectively. This study used Mössbauer spectroscopy to trace the recrystallization process of metamict perrierite following high-temperature annealing in an inert atmosphere.

The Mössbauer spectrum of the untreated sample can be fitted to two Fe<sup>2+</sup> and two Fe<sup>3+</sup> doublets in octahedral coordinations with a relative  $\Sigma\text{Fe}^{2+}/\Sigma\text{Fe}$  of 0.63. For samples annealed at 1173 and 1273 K, the spectra show a decrease in the total contribution of Fe<sup>2+</sup> to 0.58 due to dehydroxylation associated with the simultaneous oxidation of post-metamict Fe<sup>2+</sup> to Fe<sup>3+</sup>. The broad, predominant Fe<sup>3+</sup> doublet observed in the spectrum of the unannealed sample splits into two components at 973 K. The XRD pattern of the fragment annealed at 1273 K indicates that highly metamict perrierite recrystallized to the pre-metamict state that can be indexed to the C2/m space group. The Mössbauer spectra show a prominent decrease in the width of the high-energy absorption peak representing Fe<sup>2+</sup> components with increasing temperature. The variation in the width of this peak with annealing temperature seems to be an indicator of thermally induced recrystallization. Based on changes in this parameter and using an Arrhenius-type equation, the activation energy for the thermal recrystallization of perrierite was calculated to be 0.73 eV. The calculated activation energy was compared with the limited data on activation energies for other metamict minerals reported in the literature.

### Acknowledgements

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## Notatki

## Porównanie metod klasyfikacji chondrytów OC: klasycznej-opartej na zawartości fajalitu i ferrosilitu oraz 4M-opartej na spektroskopii mössbauerowskiej

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Chondryty zwyczajne (OC) to najliczniejsza grupa meteorytów. Dzielimy je na trzy grupy H, L, i LL. Klasyczna metoda klasyfikacji do poszczególnych grup opiera się na zawartości fajalitu (Fa) i ferrosilitu (Fs) wyznaczanych za pomocą mikrosondy elektronowej (EMP). W roku 2019 opublikowano pracę przedstawiającą metodę 4M (Meteorites, Mössbauer spectroscopy, Multidimensional discriminant analysis, Mahalanobis distance) [1]. Pozwala ona, na podstawie wyników pomiarów mössbauerowskich oraz w oparciu o zgromadzoną bazę danych, określić w jakim stopniu badana próbka jest „podobna” do próbek sklasyfikowanych w poszczególnych grupach. Metoda ta była testowana i udowodniono jej skuteczność [2].

Poniżej przedstawiono wynik klasyfikacji metodą 4M trzech próbek meteorytowych. Dla każdej z tych próbek określono też procentową zawartość molową fajalitu i ferrosilitu: Bassikounou - grupa H (Fa - 18,6%, Fs - 16,3%), Hyattville - grupa L (Fa - 25%, Fs - 21%) oraz Kilabo - grupa LL (Fa - 31,1 %, Fs - 27,1%). Skonstruowano bazę danych przedstawiającą zawartość tych faz w 4709 próbkach OC. Dla każdej z testowanych próbek policzono odległość do środka klastrów odpowiadających grupom H, L i LL. Dla metody 4M, w oparciu o wyniki badań spektroskopii mössbauerowskiej, obliczono procentowy poziom podobieństwa do każdej z grup.

	Odległość od środka klastra			Procentowy poziom podobieństwa do			Wynik klasyfikacji metodą	
	H	L	LL	H	L	LL	4M	klasyczną
Bassikounou	<b>0,3</b>	7,5	13,0	<b>89,5</b>	14,3	0,0	H	H
Hyattville	7,7	<b>0,5</b>	5,0	52,1	<b>61,0</b>	12,0	L	L
Kilabo	16,3	9,0	<b>3,8</b>	4,7	19,0	<b>68,6</b>	LL	LL

Można zauważyć, że obie metody dają takie same wyniki. W pracy przeprowadzimy podobną analizę dla większej liczby próbek meteorytowych.

[1] M. Woźniak, J. Gałązka-Friedman, P. Duda, M. Jakubowska, P. Rzepecka, Ł. Karwowski, *Meteoritics & Planetary Science* 54(8), (2019), p.1828–1839

[2] J. Gałązka-Friedman, M. Jakubowska, M. Woźniak, P. Bogusz, Ł. Karwowski, and P. Duda, *Hyperfine Interactions*, 242, (2021), p. 1-9.

## Notatki



**Środa**  
**19 czerwca 2024**

## Notatki



## Sesja VII

- 9:00 – 9:30 dr hab. Dariusz Satuła: Mössbauer study of MnNiGe and MnCoGe alloys doped by Fe atoms
- 9:30 – 10:00 dr inż. Robert Konieczny: A study of thermodynamic properties of dilute Fe-Rh alloys by the  $^{57}\text{Fe}$  Mössbauer spectroscopy
- 10:00 – 10:20 dr Kamila Komędera: Complex magnetic properties of  $\text{EuZn}_2\text{P}_2$
- 10:20 – 10:40 mgr Paweł Butkiewicz: Atomic Disorder and Properties of  $\text{GaFeO}_3$ : Insights from X-ray Diffraction and Mössbauer Spectroscopy

**10:40 – 11:00 Przerwa**

## Sesja VIII

- 11:00 – 11:30 prof. dr hab. Marta Marszałek-Wolny: Tracking of multimodal ordering process in FePd thin alloy films by CEMS
- 11:30 – 12:00 dr hab. Rafał Idczak: Local atomic and magnetic ordering of  $\text{Na}_{0.67}\text{Fe}_{1-y}\text{Mn}_y\text{O}_2$ -cathode materials for Na-Ion batteries
- 12:00 – 12:20 mgr Magdalena Sobota: Wysokotemperaturowa korozja stopów żelaza
- 12:20 – 12:40 mgr Wojciech Nowak: Właściwości antykorozyjne stopu  $\text{Fe}_{0.95}\text{Nb}_{0.05}$  otrzymanego poprzez mechaniczne stapianie

## Podsumowanie i zakończenie OSSM

**13:00 Obiad**

## Notatki

## Mössbauer study of MnNiGe and MnCoGe alloys doped by Fe atoms

D. Satuła<sup>1</sup>, W. Olszewski<sup>1</sup>, K. Rećko<sup>1</sup>, B. Kalska-Szostko<sup>2</sup> and K. Szymański<sup>1</sup>

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Ternary alloys MnNiGe [1] and MnCoGe [2] belong to MM'X systems (M, M'-transition metals, X- germanium, silicon or gallium elements). Both of them exhibit a

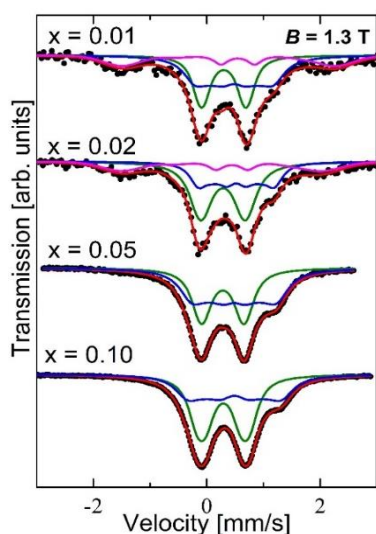


Fig.1. Mössbauer spectra of MnCo<sub>1-x</sub>Fe<sub>x</sub>Ge alloys measured in the external magnetic field.

magnetostructural transition from a high-temperature Ni<sub>2</sub>In-type hexagonal structure (space group *P6<sub>3</sub>/mmc*, 194) to a low-temperature TiNiSi-type orthorhombic structure (space group *Pmna*, 62). The observed phase transformation has garnered significant attention due to its substantial magnetocaloric effect, which holds relevance across various applications. This study presents the crystal properties, site occupation, and hyperfine fields of both alloys. X-ray diffraction and Mössbauer spectroscopy were employed, the latter both in the absence and presence of an external magnetic field ( $B = 1.3$  T) aligned parallel to the beam direction. Results demonstrate a strong correlation between phase content in the samples and Fe concentration, with a notable stabilization of the hexagonal phase observed with increasing Fe concentration. Mössbauer spectra reveal a superposition of two doublets originating from Fe atoms situated in the hexagonal structure, alongside an additional two sextets

stemming from Fe in the orthorhombic structure. Analysis of the Mössbauer spectra indicates that doped Fe atoms occupy both Mn and Co/Ni crystallographic positions in both hexagonal and orthorhombic phases. Additionally, the impact of the external magnetic field on the hyperfine parameters of MnNiFeGe and MnCoFeGe is elucidated.

- [1] Liu E, Wang W, Feng L, Zhu W, Li G, Chen J, et al. Stable magnetostructural coupling with tunable magnetoresponsive effects in hexagonal ferromagnets, *Nature Commun* 2012;3:873. <https://doi.org/10.1038/ncomms1868>.
- [2] G.J. Li, E.K. Liu, H.G. Zhang, Y.J. Zhang, J.L. Chen, W.H. Wang, H.W. Zhang, G.H. Wu, S.Y. Yu, Phase diagram, ferromagnetic martensitic transformation and magnetoresponsive properties of Fe-doped MnCoGe alloys, *J. Magn. Mater.* 332 (2013) 146–150. <https://doi.org/10.1016/j.jmmm.2012.12.001>.

## Notatki

## A study of thermodynamic properties of dilute Fe-Rh alloys by the $^{57}\text{Fe}$ Mössbauer spectroscopy

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The room temperature  $^{57}\text{Fe}$  Mössbauer spectra for binary iron-based  $\text{Fe}_{1-x}\text{Rh}_x$  solid solutions, with  $x$  in the range  $0.01 \leq x \leq 0.06$ , were measured using a constant-acceleration Polon spectrometer of standard design. The spectra are presented in Fig. 1. They were analyzed in terms of parameters of their components related to unlike surroundings of the iron probes, determined by different numbers of Rh atoms existing in the neighbourhood of iron atoms [1]. Based on the data we determined the binding energy  $E_b$  [1-3] between two Rh atoms in the studied materials. It was found that the energy is positive, which supports the suggestion that rhodium atoms interact repulsively in the  $\alpha$ -iron (*bcc*) matrix.

The obtained  $E_b$  values were used to calculate the extrapolated value of  $E_b$  for  $x=0$  and then the enthalpy  $H_{\text{Fe-Rh}}$  of solution of Rh atoms in  $\alpha$ -iron. According to findings of the  $E_b(0)=0,041(10)$  eV value is simply related to  $H_{\text{Fe-Rh}}=-z \cdot E_b(0)/2$ , where  $z$  is the coordination number of the  $\alpha$ -Fe crystalline lattice, amounted to 8 [4, 5]. The obtained value for  $H_{\text{Fe-Rh}}$ , equal to  $-0.164(40)$  eV/atom, was compared with the corresponding value  $-0.232$  eV/atom, resulting from the semiempirical Miedema's model of alloys [6], which is based on hundreds of calorimetric data concerning the heat of formation for different binary systems.

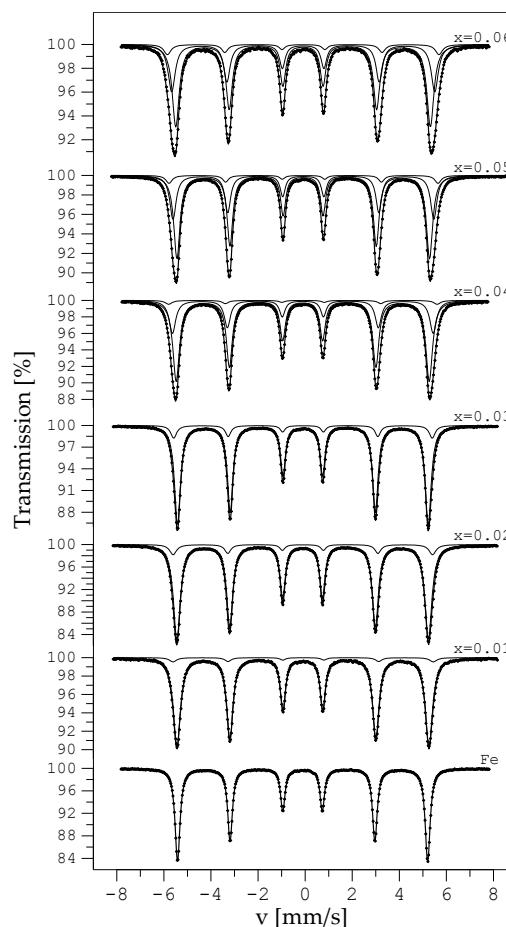


Fig. 1. The  $^{57}\text{Fe}$  Mössbauer spectra for the  $\text{Fe}_{1-x}\text{Rh}_x$  alloys measured at room temperature after the annealing process at 1270 K.

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- [2] A. Z. Hryniewicz and K. Królas, Phys. Rev. B 28 (1983), p. 1864.
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## Notatki

## Complex magnetic properties of $\text{EuZn}_2\text{P}_2$

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It was reported in the literature that the magnetic ordering of  $\text{EuZn}_2\text{P}_2$  is antiferromagnetic, with a simple magnetic structure [1-3]. However, the question of whether magnetic Eu moments are aligned in the basal  $ab$  plane of the trigonal structure was opened. Contradictory suggestions were the motivation to perform a thorough study of  $\text{EuZn}_2\text{P}_2$  single crystals. We obtained high-quality, homogeneous single crystals grown from the Sn flux. The quality of the crystals was confirmed by microprobe analysis (EDS) and X-ray diffraction studies. In this contribution, we report the results of bulk (heat capacity, AC susceptibility, DC magnetization) and microscopic (Mössbauer spectroscopy) techniques.

The results indicate that  $\text{EuZn}_2\text{P}_2$  is an antiferromagnet with strong Eu-Eu exchange coupling of ferromagnetic type within the basal plane and weaker antiferromagnetic interaction along the  $c$  axis. Magnetic susceptibility studies revealed that magnetic properties are anisotropic, and Eu magnetic moments likely form a more complex structure than the simple antiferromagnetic one. According to an analysis of low-temperature  $^{151}\text{Eu}$  Mössbauer spectra, namely the angle between the  $V_{zz}$  component of the electric field gradient tensor (EFG) and the hyperfine magnetic field at the Eu site, we found that Eu magnetic moments are tilted from the basal plane by nearly  $40^\circ$ . The derived value is close to the values reported for isostructural  $\text{EuZn}_2\text{As}_2$  and  $\text{EuNi}_2\text{As}_2$  compounds.

Considering the above results, it was possible to present the magnetic phase diagram for external magnetic fields parallel and perpendicular to the  $c$  crystallographic axis.

### Acknowledgements

Financial support by National Science Centre, Poland, Grants No. 2018/30/E/ST3/00377 and 2021/41/B/ST3/03454 is acknowledged. The research project was partly supported by the „Excellence Initiative – Research University” program for the AGH University of Krakow.

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[3] S. Krebber, et al., Phys. Rev. B 108, 045116 (2023)

## Notatki



## Atomic Disorder and Properties of GaFeO<sub>3</sub>: Insights from X-ray Diffraction and Mössbauer Spectroscopy

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The GaFeO<sub>3</sub> compound shows the existence of electrical and magnetic properties such as ferrimagnetism and piezoelectricity, magnetoelectric effect and large magnetic anisotropy [1]. The space group for orthorhombic GaFeO<sub>3</sub> is Pna2<sub>1</sub>, indicating the presence of spontaneous polarization, a phenomenon further explored by recent papers attempting to investigate its occurrence within the system [2, 3].

Regardless of the synthesis method, GaFeO<sub>3</sub> exhibits pronounced atomic disorder, characterized by iron partially occupying one of the gallium cation sublattice site, while part of the gallium is located in the iron subsites. The extent to which iron occupies selected sublattices affects the magnetic properties and plays a crucial role in the mechanism of electric polarization switching.

The presentation will focus on discussing atomic disorder in GaFeO<sub>3</sub> based on single X-ray diffraction and Mössbauer spectroscopy analyses. It will involve a comparative analysis of the results obtained through these two techniques, alongside demonstrating the consequential physical properties arising from this disorder.

[1] T. Arima et al., Physical Review B 70, 064426 (2004).

[2] S. Mukherjee et al., Phys. Rev. Lett. 111, 087601 (2013).

[3] M. Biernacka et al., Physical Review B 108, 195101 (2023).

## Notatki

## Tracking of multimodal ordering process in FePd thin alloy films by CEMS

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Fe/Pd multilayers and FePd alloys are of great interest due to their possible applications in magnetic recording, permanent magnets, surface acoustic wave filters, and terahertz magnonics [1]. In particular, the Fe/Pd bilayer shows the presence of spin-orbit torque and the Dzyaloshinskii–Moriya interaction at the Fe/Pd interface [2], which opens the possibility of skyrmion engineering in this system [3]. These properties of Fe-Pd systems are strongly related to the crystallographic structure and stoichiometry. For an FePd alloy with an atomic ratio close to 1:1, only the chemically disordered A1 phase and chemically ordered L10 phase are thermodynamically stable at room temperature. However, the above-mentioned magnetic properties require the presence of solely L10 phase.

This paper presents the process of phase transformation from spontaneously created A1 phase to chemically ordered L10 phase in FePd alloy obtained from the multilayered Fe/Pd system annealed after deposition. The annealing process induces the morphological and structural changes in the samples observed with scanning electron microscopy, X-ray reflectivity and conversion electron Mössbauer spectroscopy. The information about microscopic structural properties at different stages of annealing allows for tracking phase transformation progress. We found that the transformation to the L10 phase is a multimodal process with two distinct paths that, correlated with the solid-state dewetting, show a change in the kinetic phase growth process from low-dimensional growth into the 3-dimensional growth, which almost exclusively concerns the L10 phase.

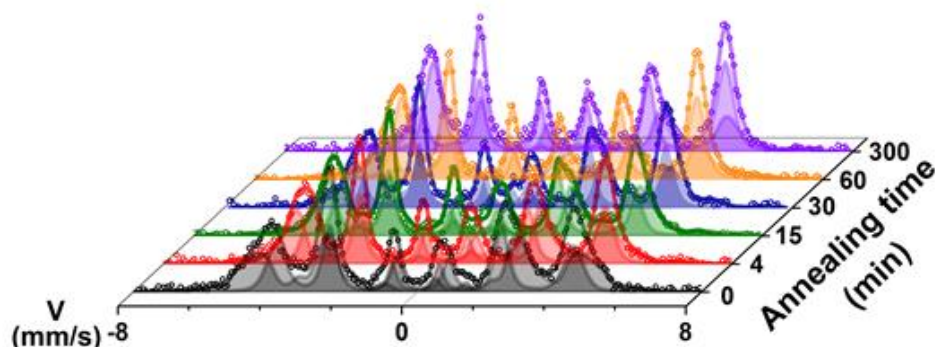


Fig. 1. CEMS spectra of annealed FePd samples.

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- [2] Mayr, S. et al, W. Phys. Rev. B, 101, 24404 (2020).
- [3] I.P Miranda et al., Phys. Rev. B, 105, 224413 (2022).

## Notatki

## Local atomic and magnetic ordering of $\text{Na}_{0.67}\text{Fe}_{1-y}\text{Mn}_y\text{O}_2$ -cathode materials for Na-Ion batteries

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Na-ion batteries have drawn a lot of attention for the past ten years as they represent the best candidate for stationary and large-scale applications where selection criteria are the cost and abundance of raw materials. Among the various materials studied, sodium layered oxides ( $\text{Na}_x\text{MO}_2$ , M = transition metal) represent a promising family. The most promising candidates are based on the Mn and/or Fe elements, which are the less expensive ones among the 3d elements [1].

As it was shown in our previous work, the  $\text{Na}_{0.67}\text{Fe}_{1-y}\text{Mn}_y\text{O}_2$  ( $y = 0.5$  and  $0.8$ ) samples crystallize in a layered P2-type crystal structure with hexagonal  $\text{P6}_3/\text{mmc}$  space group [2]. However, the room temperature  $^{57}\text{Fe}$  Mössbauer spectra of  $\text{Na}_{0.67}\text{Fe}_{1-y}\text{Mn}_y\text{O}_2$  samples revealed that they cannot be reliably fitted by one symmetric quadrupole-split component (paramagnetic doublet). Since XRD data gives only one iron site in the P2-type hexagonal  $\text{Na}_{0.67}\text{Fe}_{1-y}\text{Mn}_y\text{O}_2$  compounds, this result is rather unexpected. All recorded spectra have a slightly asymmetric shape, and due to that, the possible mechanisms of the observed asymmetry should be considered. Three alternatives are: 1) the texture effect (preferred orientation of the electric field gradient at  $^{57}\text{Fe}$  nuclei), 2) the Goldansky–Karyagin effect (an anisotropy of the recoilless fraction), and 3) the presence of more than one doublet in the recorded spectrum [3,4].

In order to select the correct interpretation of Mössbauer data, we performed measurements of Mössbauer spectra in the temperature range of 4.2–700 K as well as magnetic measurements down to 2 K. The obtained results indicate that the Mössbauer spectra can be reliably described by two components, which are ascribed to different local distortions of the  $\text{FeO}_6$  octahedra. Moreover, it was found that both studied compounds undergo antiferromagnetic transition below 10 K.

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[3] N. N. Greenwood, T. C. Gibb, *Mössbauer Spectroscopy*, Chapman and Hall Ltd., London 1971.

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## Notatki

## Wysokotemperaturowa korozja stopów żelaza

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Stopy żelaza są bardzo popularnymi materiałami stosowanymi w przemyśle. Materiały te często wystawione są na warunki sprzyjające korozji takie jak: powietrze, wysoka temperatura czy opady atmosferyczne. Według doniesień World Corrosion Organization straty ekonomiczne związane z korozją przekraczają 3% PKB wszystkich krajów uprzemysłowionych. Oznacza to, że wciąż istnieje potrzeba opracowywania nowych materiałów, które wykazują się dobrymi właściwościami antyutleniającymi oraz będą tanie w produkcji na skalę przemysłową.

Nasze badania obejmują poszukiwanie nowych stopów żelaza, które będą cechowały się dobrymi właściwościami antykorozyjnymi. W pracach opublikowanych w latach 2018 – 2020 [1, 2] zauważono, że dodatek atomów Cr i/lub Si do Fe drastycznie spowalnia proces utleniania atomów żelaza podczas utleniania w 870 K w gazach atmosferycznych. Zauważono, że w przypadku stopu  $\text{Fe}_{0.85}\text{Cr}_{0.10}\text{Si}_{0.05}$  na widmach uzyskanych z użyciem Transmisyjnej Spektroskopii Mössbauerowskiej (TMS) zawartość składowych, które można przypisać do atomów  $^{57}\text{Fe}$  w tlenkach żelaza jest niemal zerowa.

Badania nad tymi stopami były kontynuowane [3, 4] oraz poszerzone o materiały proszkowe. Zaobserwowano, iż można ograniczyć sumaryczną ilość atomów domieszek Cr i Si do 10% at. uzyskując materiał o podobnych właściwościach antyutleniających co stop  $\text{Fe}_{0.85}\text{Cr}_{0.10}\text{Si}_{0.05}$ . W tym celu wykorzystano zjawisko indukowanej tlenem segregacji powierzchniowej atomów domieszek. Skład chemiczny powierzchni próbek badany był z użyciem Spektroskopii Fotoelektronów w zakresie promieniowania rentgenowskiego (XPS), a po utlenianiu zawartość tlenków żelaza w próbce szacowana była na podstawie analizy widm TMS.

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## Notatki



## Właściwości antykorozyjne stopu $\text{Fe}_{0,95}\text{Nb}_{0,05}$ otrzymanego poprzez mechaniczne stapianie

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Stopy żelaza są materiałami powszechnie stosowanymi w przemyśle, przez co codziennie poddawane są narażeniu na różnorodne czynniki środowiskowe takie jak: wysoka temperatura, opady czy powietrze. W efekcie dochodzi do korozji czyli sukcesywnego pogarszania się właściwości fizykochemicznych stopu. Mimo że korozja jest problemem znanym od lat, według szacunków World Corrosion Organization pochłania ona rocznie 2,2 biliona USD.

Wykazano, że stopy Fe-Cr-Si charakteryzują się dobrymi właściwościami antykorozyjnymi zarówno w formie litej, jak i proszkowej [1-3]. Następnie zaczęto zastanawiać się czy inne domieszki atomów metali przejściowych mogą także pozytywnie wpłynąć na właściwości antykorozyjne stopów żelaza. Do dalszych badań wybrano stop  $\text{Fe}_{0,95}\text{Nb}_{0,05}$ , z uwagi na fakt, iż  $\text{Nb}_2\text{O}_5$  może wytworzyć na powierzchni stopu powłokę pasywną. Niestety, zgodnie z diagramem fazowym Fe-Nb [4], po wytopie w piecu łukowym układu  $\text{Fe}_{0,95}\text{Nb}_{0,05}$ , nie zostanie uzyskany czysty stop o strukturze bcc. Wytrąci się międzymetaliczna faza  $\text{NbFe}_2$ . Z tego powodu, synteza stopu  $\text{Fe}_{0,95}\text{Nb}_{0,05}$  została przeprowadzona w młynie planetarnym wykorzystując proces mechanicznego stapiania. Uzyskany w ten sposób mikrokryształiczny proszek jest jednofazowy. Materiał otrzymany bezpośrednio po stopowaniu mechanicznym poddano utlenianiu, a wyniki porównano z próbką otrzymaną w ten sam sposób, która przed utlenianiem została dodatkowo wygrzana w dynamicznej próżni w 900 K przez 2h pod ciśnieniem niższym niż  $10^{-5}$  mbar. Po utlenianiu materiałów w 870 K w gazach atmosferycznych rejestrowano widma mössbauerowskie. Zawartość tlenków żelaza w objętości badanych stopów oszacowano na podstawie składowych przypisanych do atomów  $^{57}\text{Fe}$  odpowiadających tlenkom żelaza.

[1] R. Idczak, Corrosion 75 (10) (2018), 1083.

[2] M. Sobota, K. Idczak, R. Konieczny, R. Idczak, Metallurgical and Materials Transaction A 53 (2022), 3083.

[3] M. Sobota, K. Idczak, R. Konieczny, R. Idczak, Coatings 13 (2023), 1679.

[4] S. K. Balam, A. Paul, Metallurgical and Materials Transaction A 41 (2010), 2175.

## Notatki

# Mössbauer and magnetic study of Mn(Fe)NiGe

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Current interest to the half-Heusler alloys based on MnNiGe connected with with the presence of a large number of magnetic phases and phase transitions in this type of materials. These transitions are accompanied by significant magnetocaloric and magnetostrictive effects [1-3].

In the present work, we have investigated the magnetic and Mössbauer properties of Mn<sub>1-x</sub>Fe<sub>x</sub>NiGe solid solutions ( 0.05≤x≤1.00 ) with the purpose to find out the mechanisms of magnetostructural phases formation.

The mixed phase reaction method was used for the polycrystalline Mn<sub>1-x</sub>Fe<sub>x</sub>NiGe synthesis. The mixture of the initial component powders, taken in appropriate weight ratios, was slowly heated to 1323K, annealed for 3 days at 1223K and quenched in ice water. The Mössbauer experiment was performed in usual transmission geometry and a constant acceleration regime. <sup>57</sup>mFe/Rh was used as a resonance source, the sample temperatures were ~5K, 77K (liquid nitrogen temperature) and 290K (room temperature).

The Mössbauer data obtained on iron atoms at T=77K do not reveal magnetic interactions on Fe up to the iron content x=0.20. There is no magnetic splitting of the Mössbauer spectra. We suppose that at such iron concentrations Fe atoms enter the sublattice of nickel atoms (trigonal bipyramidal positions MeII), while the deficiency of manganese atoms in the octahedral MeI positions is compensated by vacancies. So the magnetic hyperfine interaction of iron atoms in the Me II sublattice is blocked by the local environment of nickel atoms.

All studied Mn<sub>1-x</sub>Fe<sub>x</sub>NiGe 0.05≤x≤1.00 alloys at T=290 K are single-phase solid solutions with a hexagonal structure of the Ni<sub>2</sub>In type. The analysis of magnetic and Mössbauer data obtained shows that in quaternary solid solutions Mn<sub>1-x</sub>Fe<sub>x</sub>NiGe the iron atoms at concentrations up to x~0.20 preferably replace nickel atoms in Me II structural sites. As iron concentration increases (at x>0.20), the iron atoms replace both the nickel atoms at MeII sites and the manganese atoms at MeI sites..

[1] E. K. Liu, H. G. Zhang, G. Z. Xu et al. Appl. Phys. Lett. 102 (2013) 122405.  
<https://doi.org/10.1063/1.4798318>

[2] E.H. Brück, O. Tegusi, F.R. De Boer. Material for magnetic refrigeration preparation and application. US Patent 7069729 B2 (2004).

[3] V.I. Valkov, V.I. Kamenev, A.V. Golovchan et al. Phys. Solid State 63 (2021) 1889.  
<https://doi.org/10.1134/S1063783421050188>

# Metallic iron presence confirmed by Mössbauer spectroscopy studies of the dust collected in Zawiercie

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Samples provided by the STOP Foundry Association were analysed. Samples consist of material from atmospheric fallout deposited on various surfaces. Mössbauer Spectroscopy supported by Scanning and Transmission Electron Microscopy with Energy Dispersive X-ray Spectroscopy and chemical analysis methods have been used during the study.

The results of analyses using Mössbauer spectrometry (see figure 1) indicate a chemically diverse group of Fe compounds with virtually identical shares in all tested samples. There are metallic Fe ( $\alpha$ -Fe), Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> and silicate compounds of the pyroxene type. On the one hand, this composition is very similar to that found in aerosol samples collected in Krakow. On the other hand, what is noteworthy is the very large, up to 20%, share of metallic iron. The obtained parameters indicate that iron occurs in the alpha phase in the form of large (at least hundreds of nanometers) crystallites. The presence of the pure  $\alpha$ -Fe phase was also not observed in soil samples from polluted areas or those located far from anthropogenic emission sources.

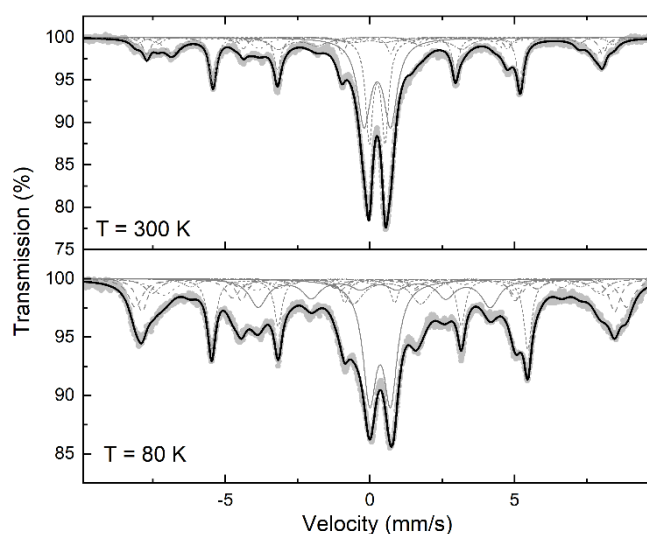


Figure 1. Mossbauer spectroscopy data measured at 300 K (top) and 77 K (bottom).



## Uczestnicy

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9:00		<b>Inauguracja</b> <b>prof. dr hab. Mieczysław Budzyński:</b> Spektroskopia mössbauerowska w Polsce i konferencje OSSM <b>prof. dr hab. Stanisław Dubiel - wykład inauguracyjny XIII OSSM:</b> Effect of magnetism on lattice vibrations		9:00 <b>dr hab. Rafał Idczak:</b> Coexistence of magnetism and superconductivity in 112-type iron pnictides $\text{EuFeAs}_2$ doped with Co <b>dr inż. Karolina Czarnacka:</b> Mössbauer spectroscopy studies of mechanosynthesized $\text{Fe}_2\text{C/Si}$ and $\text{Co}_2\text{FeAl}$ Heusler alloys <b>mgr inż. Anna Sławek:</b> Badanie stopów wysokiej entropii na bazie FeNiCoPd		9:00 <b>dr hab. Dariusz Satula:</b> Mössbauer study of MnNiGe and MnCoGe alloys doped by Fe atoms <b>dr inż. Robert Konieczny:</b> A study of thermodynamic properties of dilute Fe-Rh alloys by the $^{57}\text{Fe}$ Mössbauer spectroscopy <b>dr Kamila Komędera:</b> Complex magnetic properties of $\text{EuZn}_2\text{P}_2$	
11:00		<b>prof. dr hab. Kvetoslava Burda:</b> Effects of $\text{TiO}_2$ nanoparticles on erythrocytes functionality		10:10 <b>mgr Piotr Sobota:</b> Antiferromagnetic ordering in VFeNbTaMo high entropy alloy at low temperatures		10:20 <b>mgr Paweł Butkiewicz:</b> Atomic Disorder and Properties of $\text{GaFe}_3$ : Insights from X-ray Diffraction and Mössbauer Spectroscopy	
11:30		<b>dr hab. Jolanta Gałązka-Friedman:</b> Role of Mössbauer spectroscopy in smashing myths related to mechanisms of neurodegeneration <b>dr Joanna Fiedor:</b> Interplay between carotenoids, nanoparticles and red blood cells - what can we learn from Mössbauer study		10:30 Przerwa		10:40 Przerwa	
12:00		<b>dr Mariola Kądziołka-Gawel:</b> Identification of iron in oral iron pharmaceuticals: Mössbauer spectroscopy study		11:00 Przerwa		11:00 Przerwa	
12:20		<b>dr Katarzyna Dziezdic-Kocurek:</b> Magnetic moment of iron in oxyhaemoglobin					
12:40		<b>dr hab. Zbigniew Surowiec:</b> Synteza i właściwości nanocząstek magnezytu modyfikowanych powierzchniowo chitosanem		11:30 <b>dr Piotr Fornal:</b> New model of nanoparticles mobility in liquids		11:30 <b>prof. dr hab. Marta Marszałek-Wolny:</b> Tracking of multimodal ordering process in FePd thin alloy films by CEMS	
13:00		Obiad		11:30 <b>dr Wojciech Olszewski:</b> The impact of synthesis conditions on the properties of iron oxide nanoparticles – temperature studies		11:30 <b>dr hab. Rafał Idczak:</b> Local atomic and magnetic ordering of $\text{Na}_{0.67}\text{Fe}_{1-x}\text{Mn}_x\text{O}_2$ -cathode materials for Na-ion batteries	
14:30		<b>prof. dr hab. inż. Artur Blachowski:</b> $^{57}\text{Fe}$ Mössbauer studies of $\text{Fe}^{2+}$ -phylosilicates dehydrogenation and Fe-bearing minerals in gas/oil source rocks		12:00 <b>mgr Sławomir Strzelec:</b> Study of the influence of multi-wall carbon nanotubes functionalised with nickel ions on the functioning of red blood cells		12:00 <b>mgr Magdalena Sobota:</b> Wysokotemperaturowa korozja stopów żelaza	
15:00		<b>dr hab. Tadeusz Szumiatra:</b> Metallic iron evidence in dust from ventilation system – Mössbauer, synchrotron and magnetic analysis <b>dr inż. Jan Michalik:</b> Mössbauer spectroscopy studies of the magnetic fraction of the particulate matter emitted from coal-fired power plants and related to urban transport		12:20 <b>mgr Władysław Gumienik:</b> Mössbauer and magnetometry study of magnetite like nanoparticles		12:20 <b>mgr Wojciech Nowak:</b> Właściwości antykorozyjnie stopu $\text{Fe}_{0.95}\text{Nb}_{0.05}$ otrzymanego poprzez mechaniczne stapianie	
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15:50		Przerwa		13:00 Obiad		13:00 Obiad	
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16:00		<b>dr Tomasz Pikula:</b> The structure and magnetic properties of selected multiferroics based on $\text{BiFeO}_3$		14:30 <b>prof. dr hab. inż. Jakub Cieślak:</b> Badania granatów żelazowych w silnych polach magnetycznych		14:30 <b>dr hab. Dariusz Malczewski:</b> $^{57}\text{Fe}$ Mössbauer spectroscopy of annealed highly metamict perrierite: Activation energy for thermal recrystallization	
16:30		$\text{Bi}_{0.9}\text{La}_{0.1}\text{FeO}_3$ prepared by the sol-gel method with subsequent thermal treatment		15:00 <b>mgr Martyna Jakubowska:</b> Porównanie metod klasyfikacji chondrytów OC: Kasycznej-opartej na zawartości fajalitu i ferrosylitu oraz 4M-opartej na spektroskopii mössbauerowskiej		15:30 Sesja w jurajskich plenerach	
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16:00		Rejestracja Uczestników		17:10		17:10	
19:00		Kolacja		19:00		19:00	

