17th September 2018

Warszawa, 17th September 2018

Dr. Izabela Kudelska

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Warszawa

SELF-PRESENTATION

Contents

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1. Personal data

Name and surname: Izabela Kudelska

[redacted]

2. Diplomas and degrees

Philosophy Doctor in Physics	2005; obtained at the Institute of Physics, Polish Academy of Sciences, Warsaw, upon the thesis entitled: *Magnetic and transport properties of ferromagnetic semiconductor multinary alloys PbMnEuSnTe and GaMnAs* Supervisor: Prof. Dr. hab. Witold Dobrowolski
Master of Science in Physics	1998; obtained at Faculty of Physics, Warsaw University, upon the thesis entitled: *Magnetooptical and magnetic properties of zinc blende $Mg_{1-x}Mn_xTe$ crystals* Supervisor: Prof. Dr. hab. Andrzej Twardowski

3. Information about employment in scientific institutions

February 2014 untill now	Research Assistant, Institute of Physics PAS, Warszawa, Semimagnetic Semiconductors Group
February 2006 – February 2014	Assistant Professor, Institute of Physics PAS, Warszawa, Semimagnetic Semiconductors Group
February 2004 – February 2006	maternity leave
December 2003 – February 2004	Research Assistant, Institute of Physics PAS, Warszawa, Semimagnetic Semiconductors Group
January 1998 – November 2003	PhD Studies, Institute of Physics PAS, Warszawa, Semimagnetic Semiconductors Group

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In the period from December 2003 to February 2004, I was employed in part-time (0.1 time). In the period from February 2004 to February 2006, I was on maternity leave.

4. Bibliometric data

Scientific achievements include 37 publications (under the name Kuryliszyn-Kudelska and maiden Kuryliszyn).

According to the Web of Science database as of August 24, 2014:

Hirsch index H-index = 12

total numer of citations 776 (728 without auto-citations)

average number of citations per publication: 21

The Total Impact Factor according to the Journal Citation Reports (JCR) list is **50.483**.

The papers were published in peer-reviewed physical journals, listed below:

Physical Review B, Applied Physics Letters, Journal of Alloys and Compounds (6 publications), Journal of Applied Physics, Journal of Magnetism and Magnetic Materials, Journal of Raman Spectroscopy, Materials Research Bulletin, Optical Materials (2 publications), Physica E: Low Dimensional Systems and Nanostructures, Semiconductor Science and Technology, Journal of Physics and Chemistry of Solids, Physica Status Solidi A – Applications and Materials Science, Physica Status Solidi B – Basisc Solid State Physics, Solid State Communications, Journal of Optoelectronics and Advanced Materials, Journal of Superconductivity (2 publications), Acta Physica Polonica A (6 publications), Hemijska Industrija, Journal of the Korean Physical Society, Science of Sintering (2 publications), Magnetochemistry, Journal of Physics: Conference Series, Materials Science – Poland, AIP Conference Proceedings.

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5. Scientific achievement forming the basis for habilitation procedure

5.1 Title of the scientific achievement and list of publications being the basis for habilitation procedure

The scientific achievement that is the basis for applying for the habilitation procedure is the series of eight monothematic papers published in international journals entitled:

"Magnetic semiconductor nanocrystals based on oxide compounds – magnetic and structural properties".

List of publications included in the cycle:

H1 "Magnetic properties of nanocrystalline ZnO doped with MnO and CoO", **I. Kuryliszyn-Kudelska**, W. D. Dobrowolski, Ł. Kilański, B. Hadžić, N. Romčević, D. Sibera, U. Narkiewicz, P. Dziawa, Journal of Physics: Conference Series 200, 072058-1 – 072058-4 (2010).

H2 "Nanocrystalline ZnO Doped with Fe_2O_3 – Magnetic and Structural Properties", **I. Kuryliszyn-Kudelska**, B. Hadžić, D. Sibera, Ł. Kilański, N. Romčević, M. Romčević, U. Narkiewicz, W. Dobrowolski, Acta Physica Polonica A 119, 689-691 (2011).

H3 "Dynamic magnetic properties of ZnO nanocrystals incorporating Fe", **I. Kuryliszyn-Kudelska**, B. Hadžić, D. Sibera, M. Romčević, N. Romčević, U. Narkiewicz, W. Dobrowolski, Journal of Alloys and Compounds 509, 3756-3759 (2011).

H4 "Magnetic properties of ZnO(Co) nanocrystals", **I. Kuryliszyn-Kudelska**, B. Hadžić, D. Sibera, M. Romčević, N. Romčević, U. Narkiewicz, W. Łojkowski, M. Arciszewska, W. Dobrowolski, Journal of Alloys and Compounds 561, 247-251 (2013).

H5[1] ”Transition metals in ZnO nanocrystals – magnetic and structural properties”, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Arciszewska, N. Romčević, M. Romčević, B. Hadžić, D. Sibera, U. Narkiewicz, W. Łojkowski, Science of Sintering 45, 31-48 (2013).

H6 ”Influence of Fe doping on magnetic properties of ZrO_2 nanocrystals”, **I. Kuryliszyn-Kudelska**, M. Arciszewska, A. Małolepszy, M. Mazurkiewicz, L. Stobiński, A. Grabias, M. Kopcewicz, W. Paszkowicz, R. Minikaev, V. Domukhovski, N. Nedelko, W. Dobrowolski, Journal of Alloys and Compounds 632, 609-616 (2015).

H7 ”Superparamagnetic and ferrimagnetic behavior of nanocrystalline ZnO(MnO)”, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Arciszewska, N. Romčević, M. Romčević, B. Hadžić, D. Sibera, U. Narkiewicz, Physica E: Low-dimensional Systems and Nanostrucures 98, 10-16 (2018).

H8[2] ”Adjusting the magnetic properties of ZrO_2:Mn nanocrystals by changing hydrothermal synthesis conditions”, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Arciszewska, A. Małolepszy, L. Stobiński, and R. Minikayev, Magnetochemistry[3] 4, 28-1 – 28-16 (2018).

5.2 Introduction to the results obtained within the habilitation topic

After obtaining the doctoral degree, I was employed as an assistant professor at the Institute of Physics of the Polish Academy of Sciences in the Semimagnetic Semiconductor Group.

At that time, oxide semiconductors doped with transition metals (TM) (including ZnO, ZrO_2, TiO_2: Mn, Co, Fe, V, Ni) became one of the most intensively studied materials due to theoretically predicted high temperature ferromagnetism.

High interest in this group of materials was caused by the results of theoretical work carried out on the basis of the Zener model [1, 2]. They showed that in semiconductors consisting of light elements (in particular ZnO) and Mn-doped, the values of the Curie temperature can exceed 300 K. This prompted many research groups to study new oxide semiconductors doped with Mn and other transition metals.

[1] Review article
[2] Invited article
[3] The journal indexed in databases: *Emerging Sources Citation Index* (*Web of Science*) and *Inspec* (*IET*); during the evaluation in the *SCIE-indexing database* (*Web of Science*)

Starting from the year 2000, the number of publications on this subject has increased dramatically, in particular the research on properties of materials based on ZnO. A simple search in *Web of Science* with just the single key word "ZnO" results in over 150 000 scientific works that were published in the years 2000-2018.

ZnO is a broad-band semiconductor (with an energy gap $E_g \approx 3.3$ eV at $T = 300$ K) and with high exciton binding energy (60 meV) [3]. It has good optoelectronic and piezoelectric properties, it is also a non-toxic and biocompatible material [4]. ZnO has been placed on the list of the US governmental Food and Drug Administration (FDA) as a safe material ("generally recognized as safe" – GRAS) [5].

A significant part of the published works on ZnO concern the magnetic properties of samples doped with transition metal ions. The reported results of experimental work have caused great interest in nanoscopic magnetic systems based on ZnO.

It should be emphasized that inorganic magnetic nanomaterials are important due to a number of potential practical applications in many fields, for example: biotechnology, medical diagnostics, addressed drug delivery, cancer treatment, high density data storage, magnetic sensors [6-9]. Among the nanoscopic magnetic materials, magnetic nanoparticles with superparamagnetic properties [6, 8] are very popular, mainly due to applications in biomedicine, e.g., magnetic hyperthermia. Also, paramagnetic nano- and microparticles have found application – they were used as biosensors to detect, e.g., cancer cells, viruses, bacteria [10-13]. Iron oxide nanoparticles, considered as biocompatible and non-toxic, are now in clinical use, e.g., as contrast agents in magnetic resonance imaging (MRI), in magnetic hyperthermia, for magnetic separation [14-17].

Other solutions are in request: studies on the application of other magnetic particles to biomedicine, e.g., those containing Co, Ni, Mn [18-21] are being carried out. Biocompatibility is achieved in them by coating magnetic nanoparticles with various synthetic or natural compounds.

Many reports show that the magnetic properties of nanoscopic oxide semiconductors strongly depend on the type of chemical synthesis and the conditions under which the synthesis was carried out [22]. There is no understanding of the origin of the observed magnetic properties. Various magnetic properties were reported: high temperature ferromagnetism [23-26], low temperature ferromagnetism [27], superparamagnetism [28, 29], Curie-Weiss paramagnetism [30, 31], spin glass

type behavior [32]. There is no clarity about the mechanism of interactions responsible for the observed ferromagnetic properties.

The authors suggest that the origin of observed high temperature ferromagnetism [22] may be, e.g., mixed valence of magnetic ions and double-exchange mechanism, indirect exchange mechanism by the dopant band (Coey's model). The role of defects, e.g., oxygen vacancies, is indicated [22, 33]. There are also literature data, which indicate that the presence of nanoscopic phases of magnetic oxides may be responsible for the observed ferromagnetic properties [26, 34].

In the case of magnetically doped nanoscopic samples of semiconductor oxides we deal with more complex systems than in the case of thin films or bulk samples. The magnetic properties of magnetic nanoparticles differ from the properties of their bulk counterparts and are mainly determined by size effects and surface effects. In particular, as a size effect, we expect to observe the superparamagnetic properties of the studied materials. From the application point of view, these are very desirable magnetic properties. It should be stressed that reducing the size of nanoparticles has its consequences in the form of more atoms located on the surface, thus increasing the surface to volume ratio. It causes that surface effects, e.g., noncollinear ordering of spins on surfaces or spin glass type behaviors, have a decisive influence on the magnetic behavior of the magnetic nanoparticles system.

In addition, there are interactions between magnetic nanoparticles in real systems. They modify the magnetic properties of the nanoparticle system. Long-range dipole interactions are present. These interactions can lead to frustration effects (similar to spin glasses) and the appearance of spin glass type behavior [35, 36]. Additionally, in the case of a high concentration of magnetic nanoparticles, when they are in direct contact with one another, short-range exchange couplings will be present. These interactions modify the properties of the nanoparticle system [37].

The magnetic properties of the magnetic nanoparticles system depend on many parameters, i.e., size and size distribution of nanoparticles, the shape of nanoparticles, degree of agglomeration, surface effects, the presence of an additional magnetic phase. It causes that the problem of studying the magnetic properties of such systems is a complex task.

Part of research on ZnO doped with transition metals includes work on samples prepared in the form of thin layers using various technologies, eg: atomic layer deposition (ALD), molecular beam

epitaxy (MBE), pulsed laser deposition (PLD), magnetron sputtering (MS), metal organic chemical vapor deposition (MOCVD).

For layers, as in the case of nanoscopic samples obtained by chemical synthesis methods, also various, often contradictory, results of magnetic measurements are reported. A number of different magnetic properties are observed for them: paramagnetic [38-41], ferromagnetic [38, 42-50], superparamagnetic [51, 52], spin-type behavior [51, 53].

There are large discrepancies in the interpretation of the observed ferromagnetism. There are a number of papers in which the authors argue that the following mechanisms correspond to the observed ferromagnetic properties: RKKY (Ruderman-Kittel-Kasuya-Yosida) [42], double exchange [42], mechanism (bound magnetic polaron) BMP [46]. The origin of ferromagnetism was correlated with the presence of point defects such as: oxygen vacancies [54] or zinc vacancies [55]. The role of grain boundaries of defects [56] and strain [57] was pointed out.

It is worth noting that in many works, especially those from the initial period, the structural characterization of samples is based mainly on classical measurements, e.g., X-ray diffraction (XRD). In recent years, it has become clear that the nanoprecipitation of magnetic metals [38, 51, 52] or magnetic oxides [59-61], nanoparticles containing high concentration of magnetic ions [62] are often responsible for observed ferromagnetic and superparamagnetic properties. To determine the origin of the observed magnetic properties, it is necessary to use characterization methods that are able to detect even traces of nanoinclusions. It should be emphasized that standard characterization, e.g., by X-ray diffraction measurements, is not sensitive enough to detect, for example, traces of such precipitations. Literature data has shown that the magnetic properties of samples strongly depend on the type and conditions of synthesis. It is necessary to correlate magnetic studies with accurate structural investigations to determine the origin of the observed magnetic properties and to get to know the nature of magnetic interactions in these compounds.

Slightly less interest than ZnO: TM, attract the properties of the magnetically doped ZrO_2 semiconductor. An important motivation for the studies was the theoretical work from 2007 [63]. Based on calculations of the *ab initio* electronic structure, the authors argued that the cubic ZrO_2 doped with Mn is ferromagnetic at high temperature, above 500 K. Zirconium dioxide has a number of practical applications including: fuel cells, gas sensors, a catalyst in chemical synthesis processes, optical filters, anti-reflective coatings, it is also an important ceramic material [64-67]. ZrO_2 is a wide band-gap semiconductor, it crystallizes in monoclinic, tetragonal, and cubic structures (the E_g

value for a cubic structure is 6.1 eV, in the case of tetragonal and monoclinic structures: E_g ~ 5.87 eV and 5.83 eV, respectively) [68].

It is an important material for optoelectronics applications. It should be emphasized that from the point of view of practical applications, it is important to obtain ZrO_2 in two crystalline phases: cubic or tetragonal – due to excellent mechanical, thermal, and dielectric properties [69]. ZrO_2 is also an important material from the point of view of biomedical applications, due to its nontoxicity and biocompatibility [70]. Zirconium dioxide has been used as a biomaterial in the technique of orthopedic or dental implants [71, 72]. The application of ZrO_2 as a biosensor in the diagnostics of cancer disease has been reported [73].

In the case of ZrO_2 doped with transition metals, there are definitely less works in the literature compared to ZnO. For the nanoscopic ZrO_2:TM, experimentally, as in the case of magnetically doped ZnO, a number of different magnetic properties are observed: ferromagnetic [74-76], paramagnetic [76-78]. They depend strongly, as in the case of ZnO, on the method and conditions of sample preparation.

Also in the case of ZrO_2:TM layers, various magnetic properties are reported: ferromagnetic [79, 80], paramagnetic [79, 81]. It was pointed out that higher magnetization values are obtained for samples doped with Mn, compared to those doping with Fe and Co. High temperature ferromagnetism was also reported for the undoped ZrO_2 layers and was associated with the presence of oxygen vacancies [82]. It has been suggested that structural defects are responsible for the high temperature ferromagnetism observed for the ZrO_2:Mn layers [83].

Further systematic studies are required and the correlation of magnetic investigations with accurate structural characterization is necessary. Due to the occurring polymorphism, the problem is more complex than in the case of ZnO. In many works samples in which there was more than one crystalline phase of ZrO_2 were obtained. It has been experimentally shown that doping of ZrO_2 with transition metals (Mn, Fe) can stabilize the cubic or tetragonal crystalline phase [84, 85]. It was reported that obtaining the cubic phase ZrO_2 is crucial for the practical applications of ZrO_2, because high temperature ferromagnetism was observed only for this phase of magnetically doped ZrO_2 [80].

5.3 Objectives of the research

Taking into consideration the huge interest in the topic and the importance of magnetic studies of nanoparticles, I chose iron, cobalt and manganese-doped nanocrystalline zinc oxide (ZnO:Fe, Co,

Mn) and iron and manganese-doped nanocrystalline zirconium dioxide (ZrO_2:Fe, Mn) for my research.

The nanocrystalline samples I studied were obtained by chemical synthesis methods. It should be noted that chemical synthesis techniques are important from the point of view of practical applications. Compared to the methods of producing thin films, these techniques are cheap to use and relatively simple to implement and to adopt on a larger scale. They give the possibility of relatively easy doping of samples, control of nanocrystallites size and nanocrystalline shapes [7, 86]. It should be emphasized that these methods have been successfully used for many years for the synthesis of: oxides (eg., ZnO, ZrO_2, HfO_2, TiO_2) and magnetic particles (e.g., magnetic oxides).

The nanocrystals I studied were obtained by two methods: coprecipitation followed by calcination and hydrothermal synthesis. Both synthesis methods are characterized by: low preparation temperatures, low synthesis costs, the ability to obtain relatively large amounts of product and a relatively low dispersion of nanocrystalline sizes. In addition, the hydrothermal method gives the possibility to control the shapes of nanocrystallites [7, 86].

In many works on magnetically doped nanoscale oxide semiconductors, structural studies are mainly based on classical structural analysis techniques, e.g., only X-ray diffraction measurements. In order to know the nature of magnetic interactions and the origin of observed magnetic properties, it is important to include studies that would allow more accurate physico-chemical characteristics.

One of my goals was to get know the real structure of nanomaterials in the broadest possible range of doping. In my research on complex magnetic systems, I strived to achieve the widest possible physico-chemical characteristics, going beyond classical methods of structural research. For this purpose, in addition to standard X-ray diffraction measurements[4], and micro-Raman spectroscopy studies[5] were carried out. In the case of iron-doped samples, measurements of Mössbauer spectroscopy[6] were helpful.

Another element of the research was the proper recognition which magnetic system we are dealing with, e.g., distinguishing the freezing process from blocking magnetic moments. It was important to include dynamic studies using AC magnetic measurements. Obtaining the widest

[4] Cooperation with the group of Prof. W. Paszkowicz (Institute of Physics Polish Academy of Sciences, Warsaw)

[5] Cooperation with the group of Prof. N. Romčević (Institute of Physics, Belgrad University)

[6] Cooperation with the group of Prof. M. Kopcewicz (Institute of Electronic Materials Technology, Warsaw)

magnetic characteristics was important to determine the nature of magnetic interactions in the investigated compounds.

In the case of magnetic investigations, it is important to obtain the entire characteristics of the magnetic properties. Often, the authors point to the phenomenon of superparamagnetism based only on static magnetic studies, e.g., based on the lack of hysteresis in a certain temperature range and the difference between ZFC (zero field-cooling) and FC (field-cooling) curves for magnetization curves [28, 87]. These characteristic features are also observed for other magnetic systems (e.g., spin glass type). In the case of complex magnetic systems, AC measurements are helpful in resolving the type of magnetic material.

It was important to determine the influence of the synthesis method on the magnetic properties of the obtained nanocrystals and their structure. The use of two methods of chemical synthesis allowed to compare the results obtained and to indicate a more advantageous method from the point of view of optimizing the magnetic properties of the systems studied (in relation to practical applications). I also investigated the impact of the conditions of the indicated synthesis (e.g., pH value, type of used precursor) on the magnetic and structural properties of the studied materials. The ability to regulate magnetic parameters such as the Curie-Weiss temperature θ, the value of the coercive field H_C, by controlling the synthesis conditions is important from the point of view of applications.

An important goal was to investigate the influence of size of magnetic nanoparticles on magnetic properties. Determining whether it is possible to obtain compounds with superparamagnetic properties .

In the publication cycle, I focused on understanding the nature of magnetic interactions in nanocrystalline ZnO doped with: Mn (**H1**, **H5**, **H7**), Co (**H1**, **H4, H5**), Fe (**H2**, **H3**, **H5**) and ZrO_2 doped with Fe (**H6**) and Mn (**H8**). I carried out research on both dynamic and static magnetic properties. Detailed and extensive structural studies were carried out (X-ray diffraction measurements, micro-Raman spectroscopy, Mössbauer spectroscopy), sample morphology (SEM scanning electron microscopy, TEM transmission electron microscopy, STEM scanning transmission electron microscopy).

5.4 Investigations of magnetically doped nanocrystalline zinc oxide

5.4.1 Nanocrystalline ZnO doped with Fe (publications H2, H3, H5)

At the time when I studied the properties of nanocrystalline ZnO doped with Fe, very different results of magnetic measurements of these oxide samples, produced in the form of layers or nanostructures, were reported. For example, there was observed: high temperature ferromagnetism [88-90], paramagnetism [90], superparamagnetism [91, 92]. It was reported [88] that the solubility of Fe ions in the ZnO crystal lattice is x = 0.03, for nanoparticles obtained by the sol-gel method. Higher solubility values (x = 0.1) were given for: layers obtained by pyrolysis [90], nanoparticles synthesized by precipitation [91] and nanofibers obtained by electrospinning [89]. In many works only classical methods of structural characterization of samples (XRD) were used [90, 91]. It was also reported that Fe doping does not play any essential role in introducing magnetism in ZnO films [93]. In addition, in the case of the superparamagnetic behavior of ZnO: Fe nanoparticles [91,92] indicated by the authors, only DC magnetic techniques were used.

The first series of samples of nanocrystalline ZnO doped with Fe_2O_3 was obtained by calcination (the nominal concentration of Fe used for the reaction was recalculated to Fe_2O_3 concentration; consequently, such notation was used in all works related to ZnO) in a wide range of nominal concentration ranging from 5 wt.% to 70 wt.%.

For these samples I performed dynamic magnetic measurements, the results of which were published in the publication **H2**.

XRD measurements revealed the presence of two crystalline phases: hexagonal ZnO and spinel $ZnFe_2O_4$. Intensity of diffraction peaks of $ZnFe_2O_4$ spinel increases with increasing nominal Fe_2O_3 concentration. The average size of the $ZnFe_2O_4$ phase nanocrystallites (determined from the Scherrer equation) is in the range between 8 nm and 12 nm). XRD spectra and results of morphology studies were presented in review paper **H5**.

Considering that the X-ray diffraction method may not be sensitive enough to detect, for example, traces of nanoscopic phases, complementary studies using micro-Raman spectroscopy have been carried out. They confirmed the results obtained from XRD measurements. In the **H2** publication, two phases were shown: ZnO and $ZnFe_2O_4$ for three studied samples with nominal Fe_2O_3 content: 5 wt.%, 30 wt.%, 70 wt.%.

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In the temperature dependence of the real part of the AC magnetic susceptibility, I observed the occurrence of a characteristic broad maximum (Fig. 1a). It is visible that the maximum shifts towards higher temperatures with the increase of driving frequency *f* (Fig. 1b).

This behavior may indicate the occurrence of superparamagnetism (SPM) above the blocking temperature T_b. However, in the case of complex magnetic systems of nanoparticles, in which the agglomeration of nanoparticles, interactions between them, and surface effects occur, the behavior of the spin glass type can not be excluded [36, 94, 95].

Analysis of the maximum shift with the driving frequency *f* allows to distinguish between these two different magnetic behaviors.

A useful criterion here is the empirical parameter Φ [96-98]:

$$\Phi = \Delta T_f / T_f \Delta \log_{10}(f) \qquad (1),$$

where T_f is temperature maximum, ΔT_f is the difference between the peak temperature measured in $\Delta \log_{10}(f)$, and *f* is the AC magnetic field frequency.

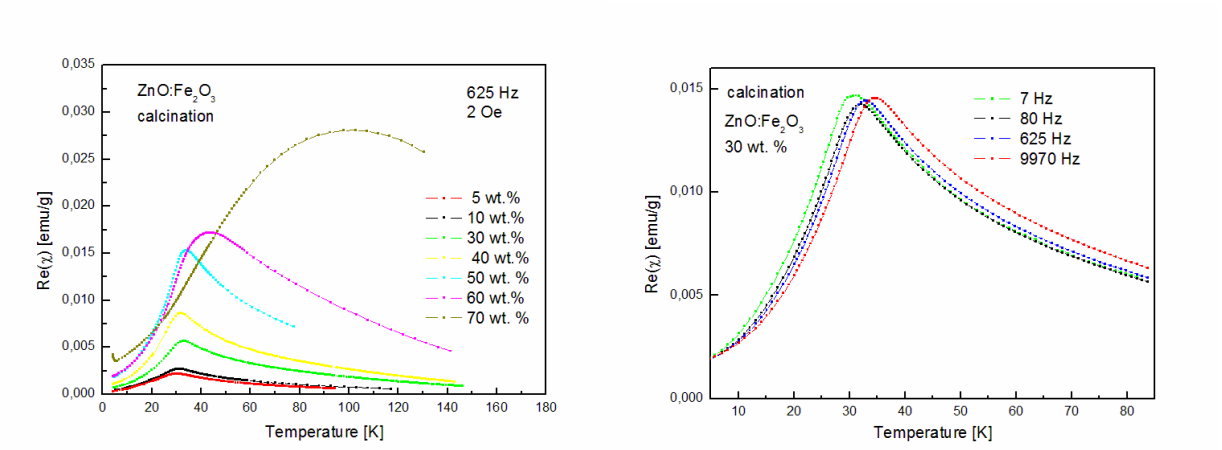

Fig1 a) Temperature dependence of the real part of magnetic susceptibility of nanocrystalline ZnO samples doped with Fe_2O_3 obtained by calcination process; b) Shifting the maximum towards higher temperatures with the increase of driving frequency f for a sample with a nominal concentration of Fe_2O_3 equal to 30 wt.%. ***H2*** *publication.*

In the case of superparamagnetic interacting magnetic nanoparticles, the parameter Φ adopts values in the range between 0.05 and 0.10, while for spin glasses lower values $\Phi < 0.05$ [97, 98].

The performed analysis showed that only weakly doped sample (5 wt.% of Fe_2O_3) shows superparamagnetic behavior. For this sample, the parameter Φ is equal to 0.06. For other samples, the determined values of parameter Φ are lower and indicate the behavior of the spin glass type.

Because the analysis of dynamic magnetic measurements proved that the most of the calcined samples revealed spin glass properties, I attempted to study nanocrystals synthesized by another chemical method. The purpose of the planned research was to obtain samples with superparamagnetic properties. The microwave-assisted hydrothermal synthesis was chosen due to the low synthesis temperature and short processing time. The results of studies were presented at publication **H3**. Samples were synthesized within the nominal concentration range between 5 wt.% and 70 wt.% of Fe_2O_3.

XRD characterization of the samples (spectra were placed in review paper **H5**) indicated that, as with the calcination process, two phases were obtained: ZnO and spinel $ZnFe_2O_4$. The size of $ZnFe_2O_4$ nanoparticles was in the range of 8 nm to 12 nm, similar to the calcination method.

To compare the results of magnetic measurements of samples obtained with both methods, in the **H3** work, next to the results of samples synthesized by the hydrothermal method, the results obtained for the calcination method were again presented, complemented by measurements for a sample with a nominal concentration of Fe_2O_3 equal to 10 wt.%.

For the hydrothermal method, a similar temperature dependence of AC magnetic susceptibility was obtained as for calcined samples. A rather broad characteristic maxima are observed (Fig. 2a) and the maximum is shifted towards higher temperatures as the frequency *f* increases (Fig. 2b).

The $\chi_{AC}(T)$ results for the selected frequencies *f* obtained for the calcination method (including 30 wt.% – shown previously in the work **H3**), were compared with those obtained for the hydrothermal method. Parameter values Φ (0.06-0.07) determined for hydrothermal synthesis are higher than Φ values obtained for calcined samples.

In paper **H3** I also used the phenomenological Vogel-Fulcher (VF) method [97-102]. The Vogel-Fulcher rule is a modification of the Arrhenius law:

$$f = f_0 \, exp[-E_a/k_B(T_f - T_0)] \qquad (2),$$

where T_f is temperature of maximum, f_0 – the frequency factor for the relaxation proces, E_a – the height of energy barrier of a single particle, T_0 is a parameter that determines the strength of interactions between magnetic moments of particles. The phenomenological parameter T_0 is used to estimate the strength of interactions in various materials.

The VF rule is useful for comparing the $T_f(f)$ relationship for superparamagnetic systems and the spin glass type by the factor $(T_f - T_0)/T_f$. In the case of small interacting particles (superparamagnetic), coefficients $(T_f - T_0)/T_f$ in the range: 0.25-1.0 are reported.

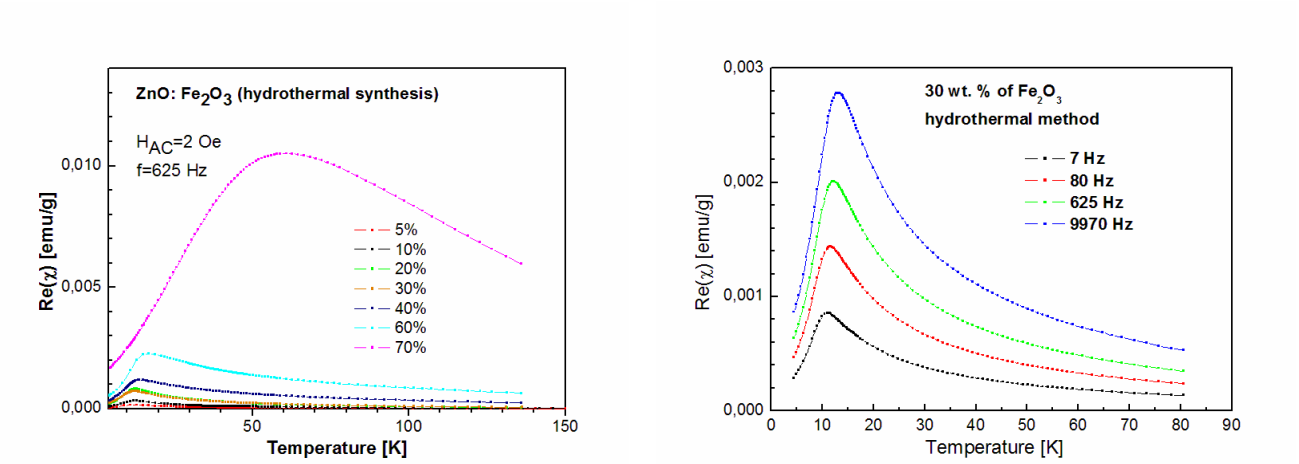

Fig. 2a) Temperature dependence of the real part of magnetic susceptibility for nanocrystalline ZnO samples doped with Fe_2O_3 obtained by hydrothermal method; b) Shifting the maximum towards higher temperatures with increasing driving frequency f for a sample with a nominal concentration of Fe_2O_3 equal to 30 wt.%. ***H3*** *publication..*

In the case of samples obtained by hydrothermal method, I obtained the values of the above coefficient in the range: 0.6-0.7. I also analyzed the samples obtained by calcination. The analysis showed lower values of $(T_f\text{-}T_0)/T_f$. For the sample doped with a nominal value equal to 5 wt.% of Fe_2O_3, I obtained the value of $(T_f\text{-}T_0)/T_f$ coefficient equal to 0.5. In the case of other calcinated samples, with a higher nominal concentration of Fe_2O_3, I received lower values of $(T_f\text{ -}T_0)/T_f$: in the range from 0.3 to 0.4. The results of the above analysis are therefore compatible with the results obtained in the analysis using the Φ criterion.

Both analyzes indicate clearly that the optimal method from the point of view of magnetic properties is the hydrothermal synthesis method. It leads to the superparamagnetic behavior of ZnO nanoparticles doped with Fe.

The structural results indicated that the nanocrystalline $ZnFe_2O_4$ phase is responsible for the obtained magnetic properties for the samples obtained using both methods of synthesis. The bulk spinel $ZnFe_2O_4$ is an antiferromagnet (T_N ~ 10 K). The ferromagnetic behavior was reported for $ZnFe_2O_4$ nanoparticles prepared by thermal decomposition [103]. It is worth noting that superparamagnetic behavior is observed not only for ferromagnetic particles. The sufficient

condition to observe superparamagnetism is having a non-zero resultant magnetic moment (e.g., when the magnetic moment of two sublattices is different).

A wider comparison of the structural properties and morphology of the samples for both methods of chemical synthesis is presented in the paper **H5**. This publication is a review work in which I collected the results of magnetic and structural research received by year 2013. In **H5** publication, the results of magnetic measurements from the **H3** work were completed by structural studies and morphological investigations. They allowed to answer the question: why the two different synthesis methods used lead to significantly different magnetic properties.

Comparison of XRD spectra for both methods in the **H5** paper leads to the conclusion that in the case of hydrothermal synthesis, the higher content of the ZnO phase is observed. For example, for samples doped with a nominal concentration of Fe_2O_3 equal to 50 wt.%, in the case of calcination, diffraction peaks coming from ZnO are much less recognizable than in the case of hydrothermal synthesis.

The micro-Raman spectrum for a calcined sample with nominal concentration of 5 wt.% repeated in the work has been completed by a more accurate analysis in the spectral range below 400 cm^{-1}. These results again confirmed the presence of the two phases described above.

In the **H5** paper the results of SEM measurements for samples obtained by two synthesis methods are shown (three samples with a nominal concentration of Fe_2O_3: 10 wt.%, 30 wt.% and 70 wt.%). I have observed two types of morphology: larger and smaller spheroidal agglomerates. In the case of synthesis by calcination, the samples are more agglomerated and more heterogeneous morphology of the samples is observed. For both methods, smaller agglomerates are observed as the nominal composition of Fe_2O_3 increases.

Transmission electron microscopy (TEM) investigations were also performed for samples synthesized with both methods. TEM images show smaller nanocrystallites than the agglomerates observed in SEM microscopy. Two morphologies are observed: hexagonal nanocrystallites and smaller – spheroidal nanocrystals. It was assumed that hexagonal nanocrystals correspond to the ZnO phase, while smaller spheroidal ones correspond to the $ZnFe_2O_4$ phase.

The performed research indicates that the observed type of spin glass behavior, instead of the expected superparamagnetic behavior, corresponds to a greater degree of agglomeration of magnetic nanoparticles and a smaller amount of the ZnO phase obtained in the calcination method. In the case of samples obtained by hydrothermal method, the smaller degree of agglomeration of nanocrystallites, and thus the reduction of the impact of short-range interactions between magnetic

nanoparticles, allowed to observe superparamagnetism. In the case of the calcination method, the superparamagnetic behavior was observed only for the sample with the lowest content of the foreign phase $ZnFe_2O_4$ and the highest content of the ZnO phase. The low concentration of magnetic nanoparticles and, hence, the limitation of direct contact of magnetic nanoparticles with each other, enabled the observation of superparamagnetism.

5.4.2. Nanocrystalline ZnO doped with Co (prace H1, H4, H5)

In the case of Co doped ZnO, various solubility values are reported in the literature. It was found that the solubility equals to x = 0.1 for layers obtained by PLD [104], as well as for nanoparticles synthesized by microwave method [105]. A slightly higher value (x = 0.12) was determined for layers prepared by the sol-gel method [58]. Depending on the sample preparation method, various magnetic properties are observed, both for the layers and for the nanoscopic samples. For example, the following were reported: high temperature ferromagnetism [22, 52, 106], paramagnetism [22, 107], antiferromagnetic interactions [22, 51, 104], spin glass [51], superparamagnetism [51, 52]. In the papers, the authors often use only standard characterization methods (XRD) [108]. It was reported that nanoscopic crystallites of metallic Co or cobalt oxides are often responsible for the observed magnetic properties [22, 51, 52, 58].

Initially, I investigated ZnO samples doped with cobalt prepared by calcination method. A series of nanocrystalline ZnO doped with CoO samples was synthesized (the nominal concentration of Co, used for the reaction, was recalculated into a CoO concentration) in a wide range of compositions: between 5 wt.% and 95 wt.%.

The results of the preliminary tests were collected in the **H1** work. XRD measurements showed the presence of two crystalline phases: ZnO (average size of nanocrystallites ranging from 43 mm to 156 nm) and Co_3O_4 (average size ranging from 14 mm to 55 nm). XRD spectra are presented in the **H4** paper.

Performed measurements of AC magnetic susceptibility as a function of temperature (in the range between 4.2 K and 150 K) for four samples doped with CoO nominal concentration: 10 wt.%, 30 wt.%, 40 wt.% and 80 wt.% revealed the presence of Curie-Weiss paramagnetism.

For all the samples I received a linear dependence of the inverse magnetic susceptibility as a function of the temperature to which I fitted the Curie-Weiss law:

$$\chi = C / (T - \theta), \qquad (3),$$

where C is Curie constant, θ is Curie-Weiss temperature.

The obtained negative values of θ: from -4.4 K (for nominal CoO concentration equal to 10 wt.%) to -27 K (for nominal CoO concentration equal to 80 wt.%) showed that antiferromagnetic interactions dominate in the studied samples.

Then, investigations of samples obtained by hydrothermal method were performed. The aim of the studies was to obtain samples in which ferromagnetic interactions would dominate. A series of nanocrystalline samples has been synthesized in the range of nominal CoO concentration from 5 wt.% to 60 wt.%.[7] The carried out studies allowed to compare the influence of synthesis on the results of magnetic, structural and morphological measurements of samples. Based on these results, papers **H4** and **H5** were prepared.

In the case of calcined samples, XRD studies showed (**H4**) that as the nominal concentration of CoO increases, the content of the nanocrystalline ZnO phase decreases, and the content of the magnetic phase of Co_3O_4 increases. The micro-Raman spectra observed for two samples with a nominal concentration of CoO: 5 wt.% and 50 wt.% confirmed the presence of two phases: hexagonal ZnO and magnetic Co_3O_4.

For the samples obtained by hydrothermal method, the presence of hexagonal ZnO phase (size of nanocrystallites in the range from 64 mm to 300 nm) and $ZnCo_2O_4$ phase (size of nanocrystallites in the range from 33 mm to 77 nm) were found. As in the case of the calcination method, the ZnO phase content decreases with the nominal CoO concentration, while the $ZnCo_2O_4$ content increases with the nominal CoO concentration. Measurements of micro-Raman[8] spectroscopy (for samples with nominal composition: 5 wt.% and 50 wt.%) confirmed that the above two crystalline phases are present in the samples.

In the **H4** paper I completed measurements of magnetic AC susceptibility for calcinated samples by the results for samples with the lowest nominal concentration of CoO: 5 wt. %, for which I received a negative Curie-Weiss temperature θ value equal to -0.3 K. For the other samples, in order to reduce the dispersion of experimental points, the measurements were repeated several times and then averaged. Figure 3a presents the determined strong dependence of the Curie-Weiss temperature θ on the nominal CoO concentration: absolute values of θ increase with the increase in the nominal concentration (in fact, the content of the magnetic nanocrystalline Co_3O_4 phase increases).

[7] The research was carried out as part of the project I led: "Magnetic semiconductor oxide nanocrystals – synthesis and investigations of physical properties" financed by the National Science Center.

[8] The Raman measurements carried out in parallel showed the presence of ZnO and $ZnCo_2O_4$ phases for all samples [P9].

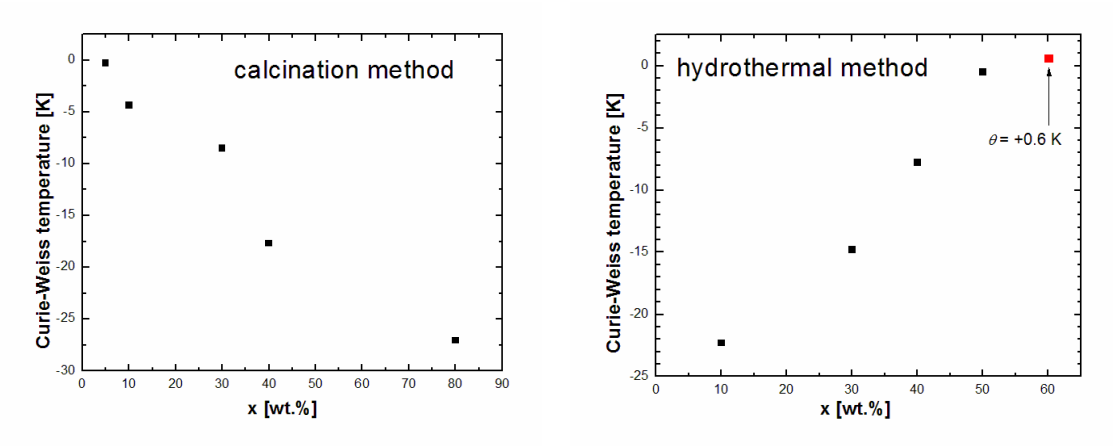

Fig. 3. Dependence of Curie-Weiss temperature θ on the nominal concentration of CoO (x) for samples obtained: a) by calcination, b) by hydrothermal method. ***H4*** *publication.*

The bulk Co_3O_4 is an antiferromagnet (T_N = 40 K). In the case of the nanocrystalline Co_3O_4 phase, antiferromagnetism (with a lower T_N = 26 K) [109] or superparamagnetism [110, 111] is observed.

The Curie-Weiss paramagnetism was also observed for samples obtained by hydrothermal method. I obtained the inverse dependence of the Curie-Weiss temperature on the nominal CoO concentration. The absolute values of the Curie-Weiss temperature decrease with the nominal CoO concentration (in fact, with the increase of the content of the magnetic $ZnCo_2O_4$ phase) (Fig. 3b).

For the sample with the highest nominal concentration of CoO (60 wt.%) I received a positive Curie-Weiss temperature. It means that ferromagnetic interactions dominate in the sample.

The literature reports both positive and negative Curie-Weiss temperatures for thin layers of $ZnCo_2O_4$, depending on the sample preparation method [112].

Comparison of results for both methods of synthesis leads to the conclusion that two different magnetic phases are responsible for the observed magnetic properties and two different trends observed for the obtained samples. It is thus possible to control the type of dominant interactions by changing the synthesis conditions.

For a sample synthesized by the hydrothermal method doped with the lowest nominal concentration CoO: 5 wt.% (with the lowest content of $ZnCo_2O_4$ and the highest content of ZnO), I observed superparamagnetic behavior. It is indicated by the analysis of the temperature dependence

of the AC magnetic susceptibility on several values of driving frequency f (value of Φ parameter equals to 0.06).

The review paper **H5** contains a study of the previously collected structural and magnetic results. This work was completed by the investigations of morphology of the samples. SEM measurements were carried out, which indicated high agglomeration of nanocrystallites.

In one of the samples obtained by hydrothermal method with a nominal concentration of CoO equal to 60 wt.%, the dominance of ferromagnetic interactions was observed. The occurrence of these interactions is related to the presence of the nanoscopic $ZnCo_2O_4$ phase.

5.4.3 Nanocrystalline ZnO doped with Mn (publications H1, H5, H7)

In my research I also studied the nanocrystalline ZnO doped with manganese.

In the case of ZnO doped with Mn, relatively high solubility values were initially reported, even up to x = 0.36, for layers obtained by PLD technique [113]. However, in this case, the samples were structurally characterized only by XRD technique. Lower solubility values were also indicated in the literature: x = 0.07 for layers obtained by RFS [39], x = 0.05 for nanoparticles obtained by sol-gel [33] and hydrothermal method [114], x = 0.01 for nanoparticles obtained by the sol-gel method [28]. A number of different magnetic properties were reported: paramagnetic [32, 39, 115], spin glass [32, 113, 115], high temperature ferromagnetism [33, 114, 115], superparamagnetism [28, 87]. It was pointed out that magnetic properties of nanoscopic magnetic phases are responsible for magnetic properties [26, 34]. In papers in which superparamagnetic behavior of nanoscopic samples was pointed out [28, 87], only DC magnetic measurements were performed.

The series of ZnO samples doped with MnO (nominal Mn concentration, used for the reaction was recalculated into the MnO concentration) in a wide range of composition between 5 wt.% and 95 wt.% was synthesized by calcination method.

The preliminary structural and magnetic measurements were performed in the **H1** paper. XRD measurements showed the presence of four crystalline phases in samples: ZnO (up to 50 wt.% of MnO), $ZnMnO_3$ (up to 50 wt.% of MnO) and for higher nominal concentrations of MnO: Zn_2MnO_4 (50 wt.% of MnO and 60 wt.% of MnO) and Mn_3O_4 (above 50 wt.% of MnO). The average size of nanocrystallites was determined: for the $ZnMnO_3$ phase it was between 9 nm and 13 nm, for the Mn_3O_4 phase – between 24 nm and 47 nm. XRD spectra are included in the review paper **H5**.

I carried out preliminary investigations of magnetic susceptibility as a function of temperature for most of the samples. For sample with a nominal concentration MnO equal to 30 wt.%, I additionally performed a magnetization measurements as a function of temperature using a SQUID magnetometer (FC and ZFC curves). Preliminary magnetic results showed the occurrence of a characteristic peak in the temperature dependence of magnetic AC susceptibility and the occurrence of bifurcation in the FC and ZFC magnetization for samples with nominal concentration of MnO below 50 wt.%. These results suggested the presence of superparamagnetic behavior. In turn, for the most doped samples, I observed the occurrence of a maximum at about 43 K in the $\chi_{AC}(T)$ dependence, typical for the Mn_3O_4 ferrimagnet.

In review publication **H5,** XRD spectra are shown. The peaks from the observed crystalline phases were denoted: ZnO, $ZnMnO_3$, $ZnMn_2O_4$, Mn_3O_4. Micro-Raman spectroscopy measurements were performed for four samples with a nominal concentration of MnO: 5 wt.%, 30 wt.%, 70 wt.%, 95 wt.%. They confirmed the presence of crystalline phases observed in XRD measurements. For the two samples with the highest composition: 70 wt.%, 95 wt.%, the micro-Raman investigation also showed the presence of the fifth crystalline phase (not detected by XRD measurements): MnO. Transmission electron microscopy (TEM) morphology studies revealed two morphologies: smaller spheroidal nanocrystallites and larger hexagonal ones.

In the **H5** paper the results of the AC magnetic susceptibility were completed by investigations for a sample with a nominal concentration of MnO equal to 30 wt.%.

I carried out further magnetic measurements in publication **H7**. At the same time, Raman spectroscopy studies[9] were continued. Below, I am going to list the main conclusions from the structural studies that I made in publication **H7**. These results were crucial in the interpretation of magnetic investigations. It was shown that in samples with a nominal composition below 50 wt.%, besides the ZnO and $ZnMnO_3$ phases, magnetic phases $ZnMn_2O_4$ and Mn_3O_4 were observed. However, the predominant magnetic phase in this doping range is the $ZnMnO_3$ phase.

XRD studies have shown that the content of the ZnO phase decreases with the increase in the nominal concentration of MnO. For a sample with a nominal concentration equal to 60 wt.% of MnO in XRD studies, the ZnO phase was no longer visible. XRD investigations showed that there are two phases: $ZnMn_2O_4$ and Mn_3O_4 for samples with nominal MnO concentration 50 wt.% and 60 wt.%. XRD measurements revealed the presence of the Mn_3O_4 phase for samples with the highest nominal

[9] Publication [P5] shows the results of micro-Raman spectroscopy studies for samples prepared in a wide range of concentration (between 5 wt.% and 95 wt.% MnO) obtained in frame of project I was a leader.

concentration of MnO. Measurements of micro-Raman spectroscopy confirmed the absence of $ZnMnO_3$ phase in a sample with a nominal concentration equal to 50 wt.% of MnO, showed the presence of MnO phase for samples with a nominal concentration from 60 wt.% to 95 wt.%, and confirmed the presence of Mn_3O_4 phase for samples with high nominal concentration of MnO. Micro-Raman spectroscopy studies also detected ZnO phase for all studied samples in the full range of nominal concentration of MnO (between 5 wt.% and 95 wt.%).

In the **H7** publication, the results of TEM microscopy were completed by TEM images for a sample with a high nominal concentration of MnO equal to 60 wt.%.

Previous magnetic studies (**H1**, **H5**) suggested the occurrence of superparamagnetism for samples with low nominal concentration of MnO (from 5 wt.% to 40 wt.%).

In the **H7** paper, I carried out measurements of AC magnetic susceptibility at various frequencies *f*. I observed a clear shift in temperature corresponding to the maximum (T_f) towards higher temperatures with increasing driving frequency *f* for four samples with a nominal concentration of MnO: 5 wt.%, 10 wt.%, 20 wt.%, 30 wt.%. Figure 4 presents the obtained results.

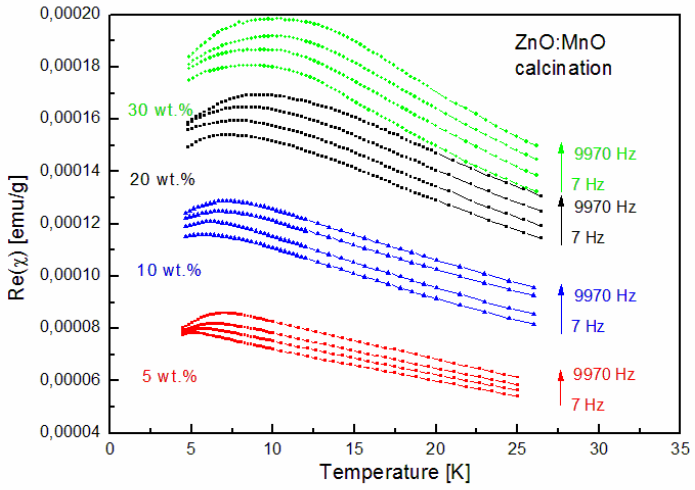

*Fig. 4. Temperature dependence of the real part of magnetic susceptibility measured for four frequencies f (7 Hz, 80 Hz, 625 Hz, 9970 Hz) for nanocrystalline ZnO samples doped with MnO obtained by calcination. Publication **H7**.*

I carried out the analysis of the results obtained. I determined the empirical parameter Φ, which for the lower compositions was 0.09, while for the sample with the nominal composition of 30 wt.% MnO has a lower value and equals 0.07. These results indicate superparamagnetic behavior.

This was confirmed by the VF analysis: the value of the coefficient $(T_f - T_0)/T_f$ for the sample with a nominal composition of 30 wt.% is 0.8, and for the other samples it equals 0.9.

The DC magnetic studies revealed the presence of the hysteresis loops below the blocking temperature. The coercive field value determined corresponds to those reported for $ZnMnO_3$ nanoparticles in the literature [116].

The conducted magnetic and structural investigations allowed to relate the observed superparamagnetism to the nanocrystalline phase of $ZnMnO_3$. This phenomenon occurs for samples with a relatively high content of the nanoscopic ZnO phase and low magnetic content of the $ZnMnO_3$ phase (for samples with a nominal concentration of MnO up to 30 wt.% and for a sample with a higher nominal composition: 40 wt.%, I did not observe superparamagnetic behavior).

For samples with the highest values of nominal concentration (from 70 wt.% to 95 wt.%) I observed a characteristic maximum in the temperature dependence of AC susceptibility at about 42 K. The magnetization measurements were completed by the results of magnetic susceptibility investigations. Observed coercive field values are consistent with those observed for Mn_3O_4 nanoparticles in the literature [117]. In this range of doping we observed the ferrimagnetic properties related to presence of Mn_3O_4.

A series of ZnO samples doped with MnO by hydrothermal method was also synthesized. Structural studies and morphology measurements were carried out for these samples. The obtained results revealed greater chemical homogeneity of the samples. There are two phases: ZnO and $ZnMn_2O_4$ with a smaller degree of the agglomeration compared to the synthesis by calcination [**P2**]. The results of magnetic studies have not been published yet.

5.5 Investigations of magnetically doped nanocrystalline zirconium dioxide

5.5.1 Nanocrystalline ZrO_2 doped with Fe (publication H6)

Various Fe solubility values for ZrO_2 samples are reported in the literature. It was reported that $x = 0.25$ for layers obtained by the ALD method [81, 118]. Lower values ($x = 0.05$) were reported for layers obtained by PLD [79]. Also in the case of nanoscopic samples, different solubility values were given: $x = 0.36$ for nanocrystals synthesized by the precipitation method [77], $x = 0.1$ for the nanocrystals obtained by combustion [75]. High temperature ferromagnetism was observed [74, 75, 79] as wel as paramagnetism [77, 81]. It was pointed out that the magnetic properties of ZrO_2 strongly depend on the method of sample preparation [119].

Conclusions about structural properties were often formulated only on the basis of XRD measurements [77].

The nanocrystalline ZrO_2 samples doped with Fe studied in publication **H6** were synthesized only by the hydrothermal method. The influence of the conditions of the conducted synthesis on the obtained structural and magnetic properties was analyzed[10].

Two series of samples S and K were obtained in the range of nominal Fe concentrations: 2.8 wt.% and 26 wt.% [11]. The influence of the ratio of the reaction mixture volume used for the reaction to the air volume of the container in which the synthesis was carried out on selected properties of the obtained samples was investigated. In addition, two samples from the K series were bubbled with O_2 gas. TEM microscopy allowed to observe two morphologies: finer spherical and larger non-oval nanocrystals (observed only for samples with a nominal concentration of Fe equal to 26 wt.%). We assume that the larger non-oval nanocrystals correspond to the α-Fe_2O_3 phase, while the smaller ones spherical – the ZrO_2 phase.

Structural investigations were carried out using X-ray diffraction measurements and Mössbauer spectroscopy measurements. The use of high-resolution powder X-ray diffraction allowed crystalline phases to be determined. The analysis of XRD results for samples with nominal concentration of Fe: 2.8 wt.% revealed the presence of two crystalline ZrO_2 phases: tetragonal and monoclinic. For the nominal Fe concentration equal to 26 wt.% two phases were obtained: cubic ZrO_2 (averaged size of nanocrystallites: 5 – 7 nm) and α-Fe_2O_3 (average size ~ 50 nm).

The studies of Mössbauer spectroscopy revealed no additional magnetic phase for samples with low nominal Fe concentration (2.8 wt.%) and demonstrated that Fe^{3+} ions built into the ZrO_2 crystal lattice. Figure 5 shows the example of Mössbauer spectra obtained for samples from the S series with low and high nominal Fe content.

[10] The results of extensive research on magnetically doped ZnO showed that, from the point of view of the obtained magnetic properties, structural hydrothermal synthesis is more optimal in comparison with the calcination method.

[11] After recalculation into nominal Fe_3O_4 concentration, this corresponds to: 5 wt.%, 20 wt.%, respectively.

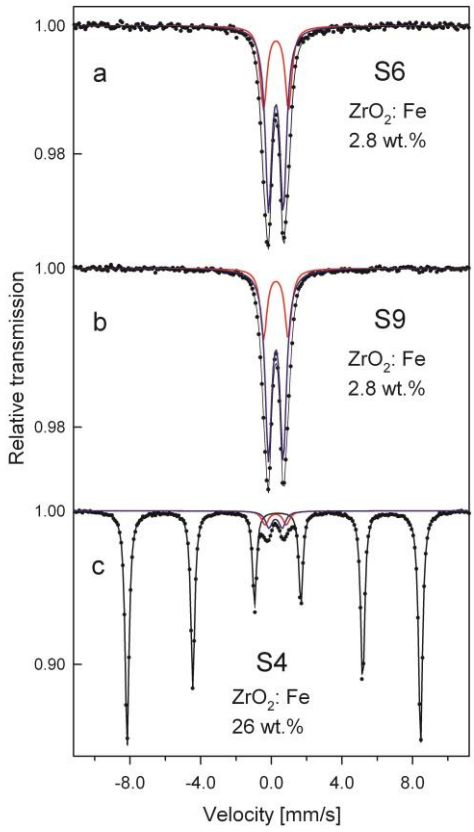

Fig. 5. Mössbauer spectra for samples from the S series (points show experimental data, continuous lines - numerical fits). Publication ***H6****.*

Due to the polymorphism, the interpretation of the obtained results of Mössbauer spectroscopy is relatively difficult. For samples with a nominal Fe content of 2.8 wt.%, analysis of Mössbauer spectra showed that Fe^{3+} ions occur in two different positions. The spectra were numerically fitted using two quadrupole doublets. It is worth mentioning that similar doublets (with similar values of determined parameters: isomeric shift, quadrupole splitting, as well as relative area) were reported for each of the three ZrO_2 structures (monoclinic, tetragonal, cubic) [74, 120-123]. Analysis of the obtained fit parameters showed that the occurrence of two doublets is related either to the presence of oxygen atoms / oxygen vacancies in the environment of Fe^{3+} ions or with non-equivalent surface positions / in the volume of nanoparticles.

The spectra obtained for samples with a nominal Fe concentration of 26 wt.% showed the presence of a magnetic foreign α-Fe_2O_3 phase. Therefore, doping with a higher nominal Fe concentration (26 wt.%), leads to the stabilization of the cubic phase ZrO_2 desirable from the

practical point of view, but then the additional magnetic phase of the nanoscopic α-Fe_2O_3 precipitates. In this case, it was also shown that Fe^{3+} ions built into the ZrO_2 structure.

Both magnetic measurements and Mössbauer spectroscopy measurements have shown that samples with a lower nominal Fe composition are paramagnetic. For all samples, I received negative Curie-Weiss θ values, which indicate that antiferromagnetic interactions are dominant in the samples.

I observed that the determined Curie-Weiss temperature θ strongly depends on the synthesis conditions. By changing the synthesis conditions, one can control the absolute values of Curie-Weiss temperature θ. For example, the value of θ for two samples S6 and S9 with the same nominal concentration of Fe (2.8 wt.%) increases from -5.6 K to -1.3 K, only after changing the ratio of the volume of the mixture used for the reaction to the volume of air in the reaction container. A similar tendency was observed for samples from the K series. The increase in the volume of the mixture causes the decrease of the absolute Curie-Weiss θ temperature. Figure 6 shows a set of results of AC magnetic susceptibility measurements for samples with a nominal Fe concentration of 2.8 wt%.

Measurements of magnetic susceptibility for samples with a nominal Fe concentration equal to 26 wt.% were carried out in the temperature range up to 350 K. The observed dependence of the real and imaginary components of magnetic susceptibility suggests the occurrence of a wide maximum in the range of high temperatures (going beyond the experimental possibilities of the measurement system).

I also carried out magnetization measurements in the range of magnetic fields up to 9 T for all samples. I used the effective Brillouin function to fit the magnetization curves [124]. This function has been used in the literature for paramagnetic nanocrystallites, e.g., nanocrystalline ZrO_2 doped with Fe and Mn [77], nanocrystalline GaN:Mn [125]. The obtained results indicate a low content of Fe ions in the ZrO_2 crystal lattice. An effective composition of x_{eff} = 0.82 wt.% was obtained for the sample with a nominal Fe concentration of 2.8 wt.% and x_{eff} = 0.59 wt.% for the sample with a nominal Fe concentration of 26 wt.%. The discrepancy between the determined values of effective composition and nominal concentration values was reported in [77].

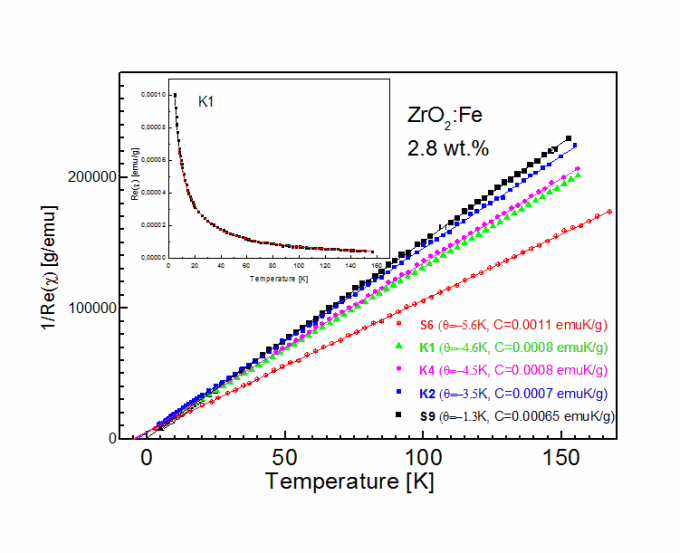

Fig. 6. Inverse of the real part of magnetic susceptibility as a function of temperature for samples with low nominal Fe concentration (2.8 wt.%). Parameters values determined: θ and C. ***H6*** *publication.*

Studies have shown that it is possible to regulate the absolute value of the Curie-Weiss temperature by changing the synthesis conditions.

For samples with low nominal Fe concentration (2.8 wt.%), semimagnetic semiconductor ZrO_2: Fe was obtained. Fe^{3+} ions have been shown to have been incorporated into the ZrO_2 crystal lattice.

5.5.2 Nanocrystalline ZrO_2 doped with Mn (publication H8)

In the case of ZrO_2 doped with Mn, as in the case of doping with Fe, different values of the solubility limit are reported. It was indicated that the solubility of Mn ions in the monoclinic ZrO_2 lattice is: x = 0.30 for the layers obtained by sol-gel method [69]. For cubic layers, the authors received a lower value: x = 0.05 [69]. For nanoscopic samples, the following values were reported: x = 0.20 [76], x = 0.27 [77]. The following magnetic phenomena were reported: ferromagnetism [69, 126], paramagnetism [77, 78, 127]. In many works, it was pointed out that the presence of nanoscopic magnetic oxides accounts for the observed magnetism of samples [77, 78, 127, 128].

In paper **H8**, I investigated the influence of hydrothermal synthesis conditions on the magnetic and structural properties of nanocrystalline ZrO_2 doped with Mn. From the application point of view, it was important to obtain magnetically doped ZrO_2 samples with a cubic or tetragonal structure. Samples with a nominal concentration of Mn: 4.9 wt.%, 9.8 wt.%, 19.5 wt.% were obtained. Three types of precursors were selected for the synthesis: $KMnO_4$, $MnCl_2$, $Mn(NO_3)_2$. In the case of the $KMnO_4$ precursor, the influence of the pH value of the mixture on the obtained

properties of the samples was investigated. Structural studies (XRD) have revealed that only the use of precursors: $MnCl_2$ and $Mn(NO_3)_2$ leads to cubic nanocrystalline ZrO_2. Measurements of STEM microscopy showed that more homogeneous and less agglomerated samples were obtained for the precursor $Mn(NO_3)_2$. X-ray diffraction measurements did not reveal the presence of an additional magnetic phase. In the case of the precursor $KMnO_4$, the cubic phase ZrO_2 is not obtained, but the mixture of the tetragonal phase (dominant phase) and monoclinic phase. The average size of nanocrystallites was determined which is between 7 nm and 8 nm for the tetragonal ZrO_2 phase and between 10 nm and 17 nm for the monoclinic ZrO_2 phase. The use of the precursors $MnCl_2$ and $Mn(NO_3)_2$ allowed to obtain a pure cubic phase for samples with a nominal concentration of Mn equal to 9.8 wt.%. The average size of nanocrystallites was determined: 6.6 nm for the precursor $MnCl_2$ and 5.8 nm for the precursor $Mn(NO_3)_2$. For the lower nominal concentration of Mn equal to 4.9 wt.%, two crystalline phases were obtained: cubic (predominant – the content of this phase for the precursor $MnCl_2$ was 95.4%, in the case of the precursor $Mn(NO_3)_2$: 94.2%) and a small amount of monoclinic phase. For all samples, a paramagnetic behavior with negative θ values is observed, which indicates the presence of dominant antiferromagnetic interactions in the samples.

In the case of the synthesis carried out using the $KMnO_4$ precursor, it was shown that the Curie-Weiss temperature θ strongly depends on the pH of the mixture used for the synthesis.

Figure 7 shows the obtained dependence of Curie-Weiss temperature θ on the pH value of the mixture used for the synthesis. The absolute values of Curie-Weiss temperature increase with increasing of pH of the mixture.

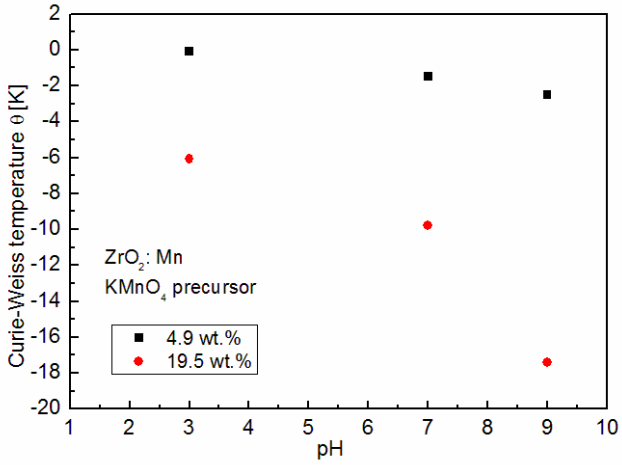

*Fig. 7. Dependence of the Curie-Weiss temperature values θ on the pH values used to synthesize the mixture. Publication **H8**.*

I also studied the effect of the type of precursor used on the Curie-Weiss temperature values. The performed analysis showed that higher absolute values of θ were obtained for the precursor $KMnO_4$. The performed analysis suggests that the increase in the pH value of the mixture used for the synthesis leads to the increase of the antiferromagnetic interactions in the samples.

I also studied static magnetic properties. I performed magnetization measurements up to 9 T for all tested samples. The measurement results were described using the effective Brillouin function. The analysis allowed to obtain an effective x_{eff} composition of Mn ions in the samples. The values determined are lower than the values of nominal Mn concentrations. It should be remembered that the x_{eff} values do not correspond to the actual values of the Mn ion content in the samples due to the presence of antiferromagnetic interactions. A similar discrepancy was observed in the literature [77].

5.6 Summary and conclusions

At present, semiconductor oxides still attract the interest of many research groups. Futhermore, there is still a lot of interest in this group of semiconductors doped with magnetic ions. Despite so many publications published in the field of nanoscopic semimagnetic semiconductors, there are numerous discrepancies in the reported literature data.

The presented publications, which are the basis for applying for the habilitation procedure, have contributed to the improvement of knowledge about magnetically doped nanoscopic semiconductor oxides.

In the publications presented:

- **It has been shown that the solubility of transition metal ions in nanoscopic oxides: ZnO and ZrO_2, obtained by chemical synthesis methods, is limited.** The formation of additional phases of the nanoscopic magnetic oxides has been observed. Their presence is the direct cause of the variety of the observed magnetic properties. Additional magnetic phases were detected for all ZnO transition metal doped nanoscopic samples. Above nominal concentration: MnO, CoO, and Fe_2O_3 equal to 5 wt.%, foreign nanoscopic magnetic phases are obtained. In this case, the result of the used synthesis methods are therefore magnetic nanocomposites containing nanocrystallites: ZnO and magnetic oxides. Such composites can be useful from the point of view of future applications.

In the case of the nanoscopic ZrO_2, there is an doping area in which foreign magnetic phases do not precipitate. We did not observe the presence of foreign crystalline phases for Mn-doped samples with a nominal concentration up to 19.5 wt.% and for Fe doped samples with a nominal concentration of 2.8 wt.%. We have shown that Fe^{3+} ions build into the ZrO_2 crystal lattice. In the case of a low nominal concentration of Fe (2.8 wt.%), we obtained a pure semimagnetic semiconductor ZrO_2: Fe.

- **It was shown that the magnetic properties of nanoscopic semiconductor oxide doped with transition metals are associated with the presence of foreign phases.** We have shown that the observed superparamagnetic properties are associated with the presence of nanoscopic phases: $ZnFe_2O_4$, $ZnCo_2O_4$, $ZnMnO_3$. The observed spin glass type behavior is related to the presence of the $ZnFe_2O_4$ phase. Ferrimagnetic properties are related to the presence of the nanoscopic Mn_3O_4 phase. We observed the Curie-Weiss-type paramagnetic behavior associated with the presence of additional nanoscopic phases: Co_3O_4 and $ZnCo_2O_4$.

- **It has been shown that very extensive characterization is required to understand the magnetic properties of nanoscopic semiconductive oxides.** Going outside the classical characterization methods: the use of the micro-Raman method and the Mössbauer spectroscopy method as well as the performance of dynamic magnetic measurements allowed to determine the origin of magnetic properties. In the case of research on nanoscopic ZnO, micro-Raman measurements allowed for detailed physico-chemical characteristics and showed the presence of "hidden" nanoscopic phases. These investigations were crucial, for example, in the case of multiphase ZnO samples doped with Mn obtained by calcination. In turn, the obtained results of Mössbauer spectroscopy confirmed the incorporation of Fe^{3+} ions into the ZrO_2 structure. An important element of the research was the application of the technique of dynamic magnetic measurements, which allowed to distinguish superparamagnetic behavior from the behavior of the spin glass type.

- **It was shown that the magnetic properties can be controlled by modifying the technological processes accordingly.** It has been shown that by changing the method of synthesis (calcination, hydrothermal method) one can influence the magnetic interactions and magnetic properties. The hydrothermal method leads to a much smaller, compared to the

calcination method, degree of agglomeration of nanocrystallites, and thus reduces the impact of short-range interactions between magnetic nanoparticles. The selection of an appropriate synthesis method allowed the observation of superparamagnetism for the nanoscopic ZnO samples doped with Fe. It has been shown that sign of Curie-Weiss temperature can be changed, i.e., the type of dominant interactions in the samples by selecting the synthesis method (studies of Co doped nanoscopic ZnO).

- **It was shown that objects with superparamagnetic properties are formed under certain conditions.** Superparamagnetism was observed for ZnO samples containing nanoscopic magnetic phases: $ZnFe_2O_4$, $ZnMnO_3$, $ZnCo_2O_4$, under specified conditions: reduction of agglomeration degree of nanoparticles, relatively high content of nanopowder ZnO phase and low magnetic phase content: $ZnMnO_3$, $ZnCo_2O_4$, $ZnFe_2O_4$.

In order to better understand the phenomena occurring in the studied nanomaterials, the degree of agglomeration of nanoparticles should be reduced. In addition, it is an indispensable element in the practical applications of magnetic nanomaterials.

In the case of magnetically doped ZrO_2, it would be worth completing structural studies with more detailed measurements of micro-Raman spectroscopy, as well as conducting EPR spectroscopy studies. The results of such measurements would complete the information on structural properties and the local environment of transition metal ions introduced into the crystal ZrO_2 lattice.

It would be helpful to introduce, parallel to the nanocrystalline ZrO_2 crystal lattice doped with transition metals, a small amount of rare earth metal dopant. Such a treatment would aim to stabilize one of the high temperature crystalline ZrO_2 phases (cubic or tetragonal), while not modifying the magnetic properties of the studied samples.

6. Discussion of other scientific and research achievements

6.1 List of scientific publications unrelated to the topic of habilitation

P1 „Influence of laser-induced heating on MnO nanoparticles”, B. Hadžić, B. Vasić, B. Matović, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Romčević, N. Romčević, Journal of Raman Spectroscopy 49, 817-821 (2018).

P2 „Raman study of surface optical phonons in hydrothermally obtained ZnO(Mn) nanoparticles", B. Hadžić, N. Romčević, M. Romčević, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, Optical Materials 58, 317-322 (2016).

P3 „Laser power influence on Raman spectra of ZnO(Co) nanoparticles", B. Hadžić, N. Romčević, D. Sibera, U. Narkiewicz, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Romčević, Journal of Physics and Chemistry of Solids 91, 80-85 (2016).

P4 „Influence of SOP modes on Raman spectra of ZnO(Fe) nanoparticles", B. Hadžić, N. Romčević, M. Romčević, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, Optical Materials 42, 118-123 (2015).

P5 „Raman study of surface optical phonons in ZnO(Mn) nanoparticles", B. Hadžić, N. Romčević, M. Romčević, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, R. Wrobel, U. Narkiewicz, D. Sibera, Journal of Alloys and Compounds 585, 214-219 (2014).

P6 „Raman study of surface optical phonons in ZnO(Co) nanoparticles prepared by calcinations method", B. Hadžić, N. Romčević, M. Romčević**, I. Kuryliszyn-Kudelska,** W. Dobrowolski, M. Gilić, M. P. Damjanović, J. Trajić, U. Narkiewicz, D. Sibera, Journal of Optoelectronics and Advanced Materials 16, 508-512 (2014).

P7 „Raman study of surface optical phonons in ZnO(Co) nanoparticles prepared by hydrothermal method", B. Hadžić, N. Romčević, M. Romčević, I. **Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, Hemijska Industrija 67, 695-701 (2013).

P8 „The cation inversion and magnetization in nanopowder zinc ferrite obtained by soft mechanochemical processing", A. Miltunović, Z. Lazarević, C. Jovalekić, **I. Kuryliszyn-Kudelska**, M. Romčević, S. Kostić, N. Romčević, Materials Research Bulletin 48, 4759-4768 (2013).

P9 „Surface optical phonons in ZnO(Co) nanoparticles: Raman study", B. Hadžić, N. Romčević, M. Romčević, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, J. Trajić, D. Timotijević, U. Narkiewicz, D. Sibera, Journal of Alloys and Coumpounds 540, 49-56 (2012).

P10 „Enhanced coercivity of as-prepared and chemically modified multiwalled carbon nanotubes", **I. Kuryliszyn-Kudelska**, A. Małolepszy, M. Mazurkiewicz, L. Stobiński, K. Kurzydłowski, W. Dobrowolski, Physica Status Solidi A – Applications and Materials Science 208, 1787-1790 (2011).

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P11 „Magnetic Properties of "as-prepared" and chemically modified multiwalled carbon nanotubes", **I. Kuryliszyn-Kudelska**, A. Małolepszy, M. Mazurkiewicz, L. Stobiński, W. Dobrowolski, Acta Physica Polonica A 119, 597-599 (2011).

P12 „Raman scattering from ZnO incorporating Fe nanoparticles: vibrational modes and low-frequency acoustic modes", N. Romčević, R. Kostić, B. Hadžić, M. Romčević, **I. Kuryliszyn-Kudelska**, W. D. Dobrowolski, U. Narkiewicz, D. Sibera, Journal of Alloys and Compounds 507, 386-390 (2010).

P13 „Low-frequency Raman scattering from ZnO(Fe) nanoparticles", R. Kostić, N. Romčević, M. Romčević, B. Hadžić, R. Rudolf, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, Acta Physica Polonica A 116, 65-67 (2009).

P14 „Synthesis by wet chemical method and characterization of nanocrystalline ZnO doped with Fe_2O_3", U. Narkiewicz, D. Sibera, **I. Kuryliszyn-Kudelska**, Ł. Kilański, W. Dobrowolski, N. Romčević, Acta Physica Polonica A 113, 1695-1700 (2008).

P15 „Magnetoresistance near the ferromagnetic-paramagnetic phase transition in magnetic semiconductors", B. Brodowska, **I. Kuryliszyn-Kudelska**, T. Wojtowicz, M. Arciszewska, W. Dobrowolski, E. I. Slynko, V. E. Slynko, X. Liu, J. K. Furdyna, Applied Physics Letters 93, 042113 – 042113-3(2008).

P16 „Raman Scattering from ZnO(Fe) Nanoparticles", N. Romčević, R. Kostić, M. Romčević, B. Hadžić, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, Acta Physica Polonica A 114, 1323-1328 (2008).

P17 „Transport and magnetic properties of $Ge_{1-x-y}Mn_x(Eu,Yb)_yTe$ semimagnetic semiconductors", B. Brodowska, **I. Kuryliszyn-Kudelska**, M. Arciszewska, K. Dybko, V. Domukhovski, W. Dobrowolski, V. E. Slynko, E. I. Slynko, V. K. Dugaev, Materials Science-Poland 26, 927-932 (2008).

P18 „Magnetic properties of Fe doped SiC crystals", **I. Kuryliszyn-Kudelska**, R. Diduszko, E. Tymicki, W. Dobrowolski, and K. Grasza, Physica Status Solidi (b) 244, 1743-1746 (2007).

P19 „Magnetic properties of $Ge_{1-x-y}Mn_xEu_yTe$ mixed crystals", W. Dobrowolski, B. Brodowska, M. Arciszewska, **I. Kuryliszyn-Kudelska**, V. Domukhovski, M. Wójcik, V. E. Slynko, E. I. Slynko, and V. K. Dugaev, AIP Conference Proceedings 893, 1231-1232 (2007).

P20 „Curie temperature control by band parameters tuning in PbMnSnEuTe" **I. Kuryliszyn-Kudelska**, W. Dobrowolski, M. Arciszewska, V. Domukhovski, V. K. Dugaev, V. E. Slynko, E. I. Slynko, I. M. Fita, Semiconductor Science and Technology 21, 1083-1086 (2006).

P21 „IV-VI ferromagnetic semiconductors recent studies", W. Dobrowolski, M. Arciszewska, B. Brodowska, V. Domukhovski, V. K. Dugaev, A. Grzęda, **I. Kuryliszyn-Kudelska**, M. Wójcik, E. I. Slynko, Science of Sintering 38, 109-116 (2006).

P22 „The effect of Mn interstitials on the lattice parameter of $Ga_{1-x}Mn_xAs$", **I. Kuryliszyn-Kudelska**, J. Z. Domagala, T. Wojtowicz, X. Liu, E. Łusakowska, W. Dobrowolski, J. K. Furdyna, Journal of Applied Physics 95, 603-608 (2004).

P23 „Effect of annealing on magnetic and magnetotransport properties of $Ga_{1-x}Mn_xAs$ epilayers", **I. Kuryliszyn-Kudelska**, T. Wojtowicz, X. Liu, J. K. Furdyna, W. Dobrowolski, J. Z. Domagala, E. Łusakowska, M. Goiran, E. Haanappel, O. Portugall, Journal of Magnetism and Magnetic Materials 272, e1575-e1577 (2004).

P24 „Ferromagnetic III-Mn-V semiconductors: manipulation of magnetic properties by annealing, extrinsic doping, and multilayer design", J. K. Furdyna, X. Liu, W. L. Lim, Y. Sasaki, T. Wojtowicz, **I. Kuryliszyn**, S. Lee, K. M. Yu, and W. Walukiewicz, Journal of the Korean Physical Society 42, S579-S590, (2003).

P25 „Low temperature annealing studies of $Ga_{1-x}Mn_xAs$", **I. Kuryliszyn**, T. Wojtowicz, X. Liu, J. K. Furdyna, W. Dobrowolski. J. M. Broto, M. Goiran, O. Portugall, H. Rakoto, H. Raquet, Journal of Superconductivity and Novel Magnetism 16, 63-66 (2003).

P26 „Anomalous Hall effect in $Sn_{1-x-y}Mn_xEu_yTe$ and $Sn_{1-x-y}Mn_xEr_yTe$ mixed crystals", K. Racka, **I. Kuryliszyn**, M. Arciszewska, W. Dobrowolski, J. M. Broto, O. Portugall, H. Rakoto, H. Raquet, V. K. Dugaev, E. I. Slynko, V. E. Slynko, Journal of Superconductivity and Novel Magnetism 16, 289-291 (2003).

P27 „Effect of the location of Mn sites in ferromagnetic $Ga_{1-x}Mn_xAs$ on its Curie temperature", K. M. Yu, W. Walukiewicz, T. Wojtowicz, **I. Kuryliszyn**, X. Liu, Y. Sasaki, J. K. Furdyna, Physical Review B 65, 201303 – 201303-4 (2002).

P28 „Magnetooptical study of s,p-d exchange interaction in zinc blende $Mg_{1-x}Mn_xTe$", **I. Kuryliszyn**, A. Stachow-Wójcik, A. Twardowski, E. Janik, E. Dynowska, J. Bąk-Misiuk, Solid State Communications 122, 213-216 (2002).

P29 „Transport and magnetic properties of LT annealed $Ga_{1-x}Mn_xAs$”, **I. Kuryliszyn**, T. Wojtowicz, X. Liu, J. Furdyna, W. Dobrowolski, J.-M. Broto, M. Goiran, O. Portugall, H. Rakoto, B. Raquet, Acta Physica Polonica A 102, 659-665 (2002).

6.2 Monographs

M1 „The role of interstitial Mn in GaAs-based dilute magnetic semiconductors”, P. Kacman**, I. Kuryliszyn-Kudelska**, “Local-Moment Ferromagnets”, Book Series Lecture Notes in Physics p. 147-161 (Springer, Berlin Heidelberg), M. Donath and W. Nolting (Eds.), 2005.

6.3 Description of research achievements unrelated to the topic of habilitation

During the master's studies at the Faculty of Physics of the University of Warsaw, I was involved in the study of magneto-optical and magnetic properties of the $Mg_{1-x}Mn_xTe$ semimagnetic semiconductor. The samples were obtained in the form of thin layers using the MBE method. The aim of the research work was to determine the parameter of s, p-d exchange interaction in a new II-VI type semimagnetic semiconductor (during this period). The results of the research were the basis for the master's thesis made under the supervision of prof. dr. hab. Andrzej Twardowski and were published in [P28].

In 1998, I started doctoral studies at the Institute of Physics of the Polish Academy of Sciences. During my PhD I performed research on thin layers of semimagnetic semiconductor $Ga_{1-x}Mn_xAs$ and bulk samples of semimagnetic semiconductors from group IV-VI: $Pb_{1-x-y-z}Mn_xEu_ySn_zTe$, $Sn_{1-x-y}Mn_xEu_yTe$, $Sn_{1-x-y}Mn_xEr_yTe$. The purpose of my research was to determine the physical mechanisms responsible for the magnetic and transport phenomena of the above-mentioned ferromagnetic semiconductors. A common feature of both studied systems was the mediation of free holes in the ferromagnetic interaction of Mn ions.

I performed studies on $Ga_{1-x}Mn_xAs$ partly as part of a one-year scholarship at the Department of Physics, University of Notre Dame, in the group of prof. Jack Furdyna. During this time, the analysis of the influence of the position of Mn atoms in the GaMnAs crystal lattice on the observed, induced by low temperature annealing, changes in the properties of ferromagnet (publications [P21, P27, M1]) was innovative. The performed research has shown that non-equilibrium growth of $Ga_{1-x}Mn_xAs$ by low-temperature MBE leads to high density of interstitial

Mn_I defects [P21, M1]. Such defects act as double donors and are coupled antiferromagnetically to the neighboring substitutional Mn [M1] ions. My research has shown that Curie temperature values, saturation magnetization, hole concentration can be significantly increased by low-temperature annealing [P21-P24, P27, P29, M1]. The mechanism of the annealing process was explained.

As part of the studies on the properties of mixed crystals $Pb_{1-x-y-z}Mn_xEu_ySn_zTe$, $Sn_{1-x-y}Mn_xEu_yTe$ i $Sn_{1-x-y}Mn_xEr_yTe$, the effect of the presence of two different magnetic ions introduced into the semiconductor matrix on the magnetic and magnetotransport properties of the obtained crystals was investigated [P25]. One of the reasons why chalcogenide compounds have been selected for this type of research was primarily the variety of magnetic properties observed in mixed crystals from IV-VI group, especially those induced by indirect RKKY exchange interaction: paramagnetic-ferromagnetic and ferromagnetic-spin glass .

After obtaining the doctoral degree and starting work at the Institute of Physics of the Polish Academy of Sciences, I was initially performed research on the bulk semi-magnetic semiconductor $Pb_{1-x-y-z}Mn_xEu_ySn_zTe$. I observed a strong correlation: a decrease in Curie T_C temperature and Curie-Weiss temperature θ with an increase in Eu content for samples showing ferromagnetic order at low temperatures. The analysis carried out showed that one of the reasons could be the change in the location of the heavy hole band Σ. The results of the research were published in the publication [P19]. I also performed research on other bulk ferromagnetic semiconductors from group IV-VI: GeMn (Eu, Yb) Te and SnMnTe [P20, P15], as well as thin layers of the ferromagnetic semiconductor InMnSb [P15].

In my research I was also involved in the study of bulk samples of ferromagnetic SiC crystals doped with Fe [P18]. During this time, silicon carbide was one of the intensively studied materials due to potential applications in electronics.

I studied various magnetic nanoscopic systems. I performed studies on multi-wall carbon nanotubes (MWCNTs) produced by chemical vapor deposition. I studied influence of the chemical functionalization of nanotubes on their magnetic properties [P10, P11]. I also investigated nanoscopic $ZnFe_2O_4$ spinel samples obtained by mechanochemical synthesis [P8]. The aim of the measurements was to examine the magnetic properties and determine the inversion degree of the obtained samples.

6.4 List of conference publications

1. „Thermodynamic limits to the maximum Curie temperature in GaMnAs", K. M. Yu , W. Walukiewicz, T. Wojtowicz, **I. Kuryliszyn**, X. Liu, Y. Sasaki, J. K. Furdyna, Institute of Physics Conference Series 171, F231 (2003).
2. „In quest of Mn-Eu interaction in IV-VI mixed crystals", **I. Kuryliszyn**, M. Arciszewska, M. M. Abdel Aziz, E. I. Slynko, V. I. Slynko, V. K. Dugaev, Proc. 9th Int. Conf. on Narrow Gap Semiconductors, N. Puhlman, H.-U. Müller, M. Von Ortenberg (Eds.), Humboldt University Berlin, 96-98 (2000).

6.5 Leading of scientific projects and participating in such projects

1. **Leader of the scientific project** „Magnetic semiconductor oxide nanocrystals – synthesis and studies of physical properties" funded by Narodowe Centrum Nauki, UMO-2011/01/B/ST5/06602; (2011-2014).
2. **Principal investigator of scientific project** „Magnetic and transport properties of ferromagnetic mixed crystals $Pb_{1-x-y-z}Mn_xEu_ySn_zTe$ and $Ga_{1-x}Mn_xAs$" funded by Komitet Badań Naukowych, 0296/P03/2003/24; (2003- 2004).
3. **Principal investigator of scientific project** „Ultra-precise measurements of magnetic properties of complex ferromagnetic semiconductors" funded by Narodowe Centrum Nauki, UMO-2012/05/D/ST3/03161; (2013-2017).
4. **Investigator of scientific project** „Transport properties of magnetic semiconductors and nanostructures related to the presence of domain walls" funded by Ministerstwo Nauki i Szkolnictwa Wyższego, 2 P03B 053 25; (2003-2006).

6.6 The list of invited lectures presented at international conferences

1. Plenary invited lecture: „Transition metals in ZnO nanocrystals: magnetic and structural properties", Advanced Ceramics and Application I (ACAI), Belgrade, Serbia (2012).
2. Invited lecture: „Transition metals in oxide diluted magnetic semiconductor nanocrystals", The 4th International Advances in Applied Physics & Material Science Congress Exhibition (APMAS), Fethiye-Mugla, Turkey (2014).
3. Key lecture: „Magnetic and structural properties of ZrO_2(Fe, Mn, Co) nanoparticles", Advanced Ceramics and Application III (ACAI), Belgrade, Serbia (2014).

6.7 Teaching activity

1. Preparing and conducting a semester lecture together with dr. Michał Szot and dr hab. Łukasz Kilański in the International PhD Studies, Institute of Physics PAS: "Selected methods of experimental investigations of semiconductor materials". I was conducting a part devoted to magnetic research of semimagnetic semiconductors, spring semester 2016.
2. Preparation and conducting of exercises for the semester lecture "Introduction to Solid State Physics" for students of the III year of the School of Sciences in Warsaw (in 2001, after the merger with the Faculty of Mathematics of the UKSW, the name was changed to the Faculty of Mathematics and Natural Sciences, School of Sciences UKSW) - spring semester 1999, spring semester 2000.
3. **Scientific supervision of a graduate student** (UKSW) Teresa Piasecka (2002).
4. **Scientific supervision of a graduate student** (UKSW) Agnieszka Grzęda (2003).
5. **Scientific supervision of a graduate student** (PW) Kornelia Ciok (2016-2017).

6.8 Achievements in the field of popularization of science

1. Lecture „The machine has started! So what was the age of electricity and steam? ” conducted at the Children's University in Warsaw, addressed to youth (2012).
2. Conducting a series of experimental workshops "Will the current flow thanks to a lemon?" for listeners of the Children's University in Warsaw (2012).
3. Organizing and conducting in the Institute of Physics of the Polish Academy of Sciences in 2013-2017 a series of experimental workshops (over 100 workshops) for school groups as part of the cycle "Wednesday meetings with physics". I conducted part of the workshops together with dr Beata Brodowska.
4. Conducting numerous experimental workshops (at around 20) promoting physics in primary schools in Warsaw, Pruszków in 2013-2015.
5. Science Festival in 2013-2017 - conducting experimental workshops addressed to school-age children – "Young researchers for the start!". I conducted part of the workshops with dr Beata Brodowska.
6. Conducting experimental workshops for children as part of the Open Days of the Institute of Physics of the Polish Academy of Sciences in 2013-2017. I conducted part of the classes together with dr Beata Brodowska.

7. Participation in exhibitions presented by the Institute of Physics PAS at the Science Picnic in Warsaw in 2013-2014.

6.9 Seminars

1. Laboratoire National des Champs Magnétiques Intenses, Toulouse: „Magnetic and transport properties of GaMnAs" (November 2001).
2. Institute of Physics, Humboldt University „Low temperature annealing studies of GaMnAs" (November 2002).
3. Seminar on Condensed Matter Physics, Institute of Physics PAS, Warsaw: „Low temperature annealing studies of GaMnAs: electronic, magnetic, structural properties" (November 2001).
4. Seminar on Condensed Matter Physics, Institute of Physics PAS, Warsaw „Magnetic semiconductor oxide nanocrystals – synthesis and magnetic properties" (May 2014).
5. Seminars of Division of Semiconductor Physics delivered at the Institute of Physics of the Polish Academy of Sciences in Warsaw:
   a) „ZnO nanocrystals doped with Fe_2O_3 and MnO - structural, optical and magnetic properties" (August 2009).
   b) „Magnetic properties of raw and functionalized multi-wall carbon nanotubes" (April 2010).
   c) „Comparison of structural and magnetic properties of ZnO: CoO nanocrystals synthesized by two methods: calcination and hydrothermal" (April 2011).
   d) „Nanoscopic spinel ferrites – $NiFe_2O_4$ - synthesis, magnetic and structural properties" (April 2012).
   e) „Synthesis and investigation of magnetic and structural properties of nanoscopic ZrO_2 doped with Fe, Mn" (April 2013).
   f) „Magnetic and structural properties of ZrO_2 nanoparticles (Fe, Mn, Co)" (May 2015).
   g) „Magnetic properties of metalloporphyrins" (April 2016).
   h) „ Magnetic properties of ZnO nanocrystals doped with Mn" (March 2017).

6.10 Organizational activity

1. Secretary of the international conferences of physics of semiconductor materials co-organized by the Institute of Physics of the Polish Academy of Sciences: "XXXVI International School

on the Physics of Semiconducting Compounds", Jaszowiec 2007 and "XXXVII International School on the Physics of Semiconducting Compounds", Jaszowiec 2008.

2. Scientific secretary of the workshops co-organized by the Institute of Physics PAS "VIII Semiconductor Semiconductor Physics Workshop", Obory 2003.
3. Participation in the preparation of post-conference materials of the E-MRS Fall Meeting 2002 international conference, Symposium G, "Solid Solutions of the II-VI Compounds - Growth, Characterization and Applications", Zakopane 2002.
4. Participation in the scientific committee of the conference "The 4th International Advances in Applied Physics" Materials Science Congress Exhibition "APMAS Turkey 2014.

6.11 Scientific visits abroad

1. Annual research stay at the Department of Physics, University of Notre Dame, USA (2000-2001).
2. In the years 2001-2012 short research stays (2 weeks) in the following scientific centers: Laboratoire National des Champs Magnétiques Pulses Toulouse (3 stays, Toulouse, France), Humboldt University (1 stay, Berlin, Germany), Belgrade University (2 stays, Belgrade, Serbia).

6.12 Building of research equipment

Construction of the system for magnetotransport measurements in the Department of Physics, University of Notre Dame, USA (2000-2001). The measurement system is based on an existing magneto-optical measurement system (optical cryostat and a superconducting magnet from Oxford Instruments).

6.13 Refereeing

1. Reviewing research projects for the Austrian Science Fund (twice) and the Ministry of Science of Serbia.
2. Reviews of scientific articles for: Journal of Alloys and Compounds, Acta Physica Polonica A, Materials Research Bulletin, Science of Sintering, Nanoscale Research Letters, Journal of Engineering and Manufacturing, Journal of Physics and Chemistry of Solids, Journal of Magnetism and Magnetic Materials, Crystals.

6.14 Research works presented personally at international conferences

1. „Magnetooptical study of s,p-d exchange interaction in zinc blende $Mg_{1-x}Mn_xTe$ epilayers", **I. Kuryliszyn**, A. Stachow-Wójcik, A. Twardowski, E. Janik, E. Dynowska, J. Kossut, J. Bąk-Misiuk, T. Wojtowicz, G. Karczewski, XXVII International School on Physics of Semiconducting Compounds, Jaszowiec, 1998.
2. „Mn-Eu interaction in IV-VI mixed crystals", **I. Kuryliszyn**, M. Arciszewska, W. Dobrowolski, V. Domuchowski, E. I. Slynko, V. E. Slynko, V. K. Dugaev, XXIX International School on Physics of Semiconducting Compounds, Jaszowiec, 2000.
3. „Transport and magnetic properties of low temperarure annealed $Ga_{1-x}Mn_xAs$", **I. Kuryliszyn**, T. Wojtowicz, X. Liu, J. K. Furdyna, W. Dobrowolski, J. M. Broto, M. Goiran, O. Portugall, H. Rakoto, H. Raquet, XXXI International School on Physics of Semiconducting Compounds, Jaszowiec, 2002.
4. „Low temperature annealing studies of $Ga_{1-x}Mn_xAs$", **I. Kuryliszyn**, T. Wojtowicz, X. Liu, J. K. Furdyna, W. Dobrowolski, J. M. Broto, O. Portugall, H. Rakoto, H. Raquet, 2nd International Conference on the Physics and Applications of Spin-Related Phenomena in Semiconductors, Würzburg, 2002.
5. „Magnetic studies of Ga1-xMnxAs epilayers", **I. Kuryliszyn**, M. Goiran, J. K. Furdyna X. Liu, T. Wojtowicz, M. Baran, W. Dobrowolski, J. Z. Domagała, E. Łusakowska, R. Szymczak J. M. Broto, H. Rakoto, H. Raquet, XXXII International School on Physics of Semiconducting Compounds, Jaszowiec, 2003.
6. „Effect of annealing on magnetic and magnetotransport properies in $Ga_{1-x}Mn_xAs$", **I. Kuryliszyn-Kudelska**, T. Wojtowicz, X. Liu, J. K. Furdyna, W. Dobrowolski, J. Z. Domagała, E. Łusakowska, J. M. Broto, M. Goiran, E. Haanappel, O. Portugall, H. Rakoto, B. Raquet, Interenational Conference on Magnetism ICM, Roma, 2003.
7. „Magnetic properties of Fe doped SiC crystals", **I. Kuryliszyn-Kudelska**, R. Diduszko, E. Tymicki, W. Dobrowolski, K. Grasza, European Materials Research Society (E-MRS) Fall Meeting, Warsaw, 2006.
8. „Effect of iron addition on the properties of ZnO obtained by precipitation", U. Narkiewicz, D. Sibera, W. Dobrowolski, **I. Kuryliszyn-Kudelska**, European Materials Research Society (E-MRS) Fall Meeting, Warsaw, 2007.

9. „Synthesis by wet chemical method and characterization of nanocrystalline ZnO doped with Fe_2O_3", U. Narkiewicz, D. Sibera, **I. Kuryliszyn-Kudelska**, Ł. Kilański, W. Dobrowolski, N. Romčević, XXXVII International School on the Physics of Semiconducting Compounds, Jaszowiec, 2008.
10. „Magnetic properties of nanocrystalline ZnO doped with MnO and CoO", **I. Kuryliszyn-Kudelska**, W. Dobrowolski, L. Kilanski, B. Hadžić, N. Romčević, D. Sibera, U. Narkiewicz and P. Dziawa, International Conference on Magnetism ICM, Karlshrue, 2009.
11. **„**Nanocrystalline ZnO doped with Fe_2O_3 – magnetic and structural properties", **I. Kuryliszyn-Kudelska**, B. Hadžić, D. Sibera, L. Kilanski, N. Romčević, M. Romčević, U. Narkiewicz, W. Dobrowolski, XXXIX International School & Conference on the Physics of Semiconductors, Krynica-Zdrój, 2010.
12. **„**Magnetic properties of as-grown and chemically modified multiwalled carbon nanotubes", **I. Kuryliszyn-Kudelska**, A. Małolepszy, M. Mazurkiewicz, L. Stobiński, W. Dobrowolski, XXXIX International School & Conference on the Physics of Semiconductors, Krynica-Zdrój, 2010.
13. „Enhanced coercivity of as-prepared and chemically modified multiwalled carbon nanotubes", **I. Kuryliszyn-Kudelska**, A. Małolepszy, M. Mazurkiewicz, L. Stobinski , K. J. Kurzydłowski, W. Dobrowolski, , plakat prezentowany na European Materials Research Society (E-MRS) Fall Meeting, Warsaw, 2010.
14. „Magnetic and structural properties of ZnO doped with Fe_2O_3", **I. Kuryliszyn-Kudelska**, L. Kilański, D. Sibera, B. Hadžić, N. Romčević, M. Romčević, P. Dziawa, U. Narkiewicz, W. D. Dobrowolski, European Materials Research Society (E-MRS) Fall Meeting, Warsaw, 2010.
15. „Magnetic properties of ZnO nanocrystals incorporating Co", **I. Kuryliszyn-Kudelska**, D. Sibera, B. Hadžić, N. Romčević, M. Romčević, M. Arciszewska, U. Narkiewicz, W. Dobrowolski", The European Conference Physics of Magnetism (PM'11), Poznań, 2011.
16. „Magnetic and structural properties of nanosized $NiFe_2O_4$ obtained by the soft mechanochemically assisted synthesis from mixture powders", **I. Kuryliszyn-Kudelska**, M. Arciszewska, N. Nedelko, Z. Lazarević, N. Romčević, Č. Jovalekić, A.

Rečnik, V. N. Ivanovski, A. Milutinović, B. Cekić, 9th International Conference on Nanosciences & Nanotechnologies (NN12) Thessaloniki, 2012.

17. „Structural and magnetic properties of ZrO_2(Fe, Mn) nanoparticles", **I. Kuryliszyn-Kudelska**, M. Arciszewska, A. Małolepszy, M. Mazurkiewicz, L. Stobinski, V. Domukhovski, N. Nedelko, A. Grabias, M. Kopcewicz, W. Dobrowolski, 42nd International School & Conference on the Physics of Semiconductors "Jaszowiec 2013", Wisła, 2013.
18. „Structural and magnetic properties of ZrO_2(Fe, Mn) nanoparticles", **I. Kuryliszyn-Kudelska**, M. Arciszewska, A. Małolepszy, M. Mazurkiewicz, L. Stobiński, V. Domukhovski, N. Nedelko, A. Grabias, M. Kopcewicz, W. Dobrowolski, Euromat 2013, Sewilla, 2013.
19. „Transition metals in ZrO_2(Fe, Mn) nanoparticles – structural and magnetic studies", **I. Kuryliszyn-Kudelska**, M. Arciszewska, A. Małolepszy, M. Mazurkiewicz, L. Stobinski, R. Minikayev, W. Paszkowicz, N. Nedelko, A. Grabias, M. Kopcewicz, W. Dobrowolski, 6th Szeged International Workshop on Advances in Nanoscience, Siwan 6, Szeged, 2014.

6.15 Research works presented by co-workers at international conferences

1. „Room temperature magnetic phase in III-VI layered semiconductors InFeSe and InCoSe", W. Dobrowolski, M. Arciszewska, **I. Kuryliszyn**, W. Paszkowicz, A. V. Zaslonkin, Z. Kovalyuk, XXIX International School on Physics of Semiconducting Compounds, Jaszowiec, 2000.

2. „Anomalous Hall Effect in $Sn_{1-x-y}Mn_xEu_yTe$ Mixed Crystals", K. Racka, **I. Kuryliszyn**, M. Arciszewska, W. Dobrowolski, E. I. Slynko, V. E. Slynko, XXX International School on Physics of Semiconducting Compounds, Jaszowiec, 2001.

3. „Anomalous Hall efect in Sn(1-x-y)Mn(x)Eu(y)Te and Sn(1-x-y)Mn(x)Er(y)Te mixed crystals", K. Racka, **I. Kuryliszyn**, M. Arciszewska, W. Dobrowolski, J. M. Broto, O. Portugall, H. Rakoto, H. Raquet, V. K. Dugaev, E. I. Slynko, V. E. Slynko, 2nd International Conference on the Physics and Applications of Spin-Related Phenomena in Semiconductors, Würzburg, 2002.

4. „IV-VI ferromagnetic semiconductors recent studies", W. Dobrowolski, B. Brodowska, M. Arciszewska, **I. Kuryliszyn-Kudelska**, V. Domukhovski, M. Wójcik, V. E. Slynko, E. I. Slynko, and V. K. Dugaev, XXIX International Conference on the Physics of Semiconductors, Vienna, 2006.

5. „Raman Scattering from ZnO(Fe) Nanoparticles", N. Romčević, R. Kostić, M. Romčević, B. Hadžić, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera, XXXVII International School on the Physics of Semiconducting Compounds, Jaszowiec, 2008.

6. „Raman scattering from ZnO doped with Fe, Mn and Co nanoparticles", B. Hadžić, N. Romčević, M. Romčević, R. Kostić, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, and D. Sibera, European Materials Research Society (E-MRS) Fall Meeting, Warsaw, 2010.

7. „Raman scattering from ZnO doped with Fe, Mn and Co nanoparticles", B. Hadžić, N. Romčević, M. Romčević, R. Kostić, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, and D. Sibera, 14th International Conference on II-VI Compounds, St. Petersburg, 2011.

8. „Structural and magnetic properties of ZnO nanocrystals doped with transition metal ions", W. Dobrowolski, **I. Kuryliszyn-Kudelska**, B. Hadžić, N. Romčević, U. Narkiewicz, and D. Sibera, International Conference on Advanced Materials Isfahan, 2012.

9. „Raman Scattrering from ZnO(Mn) nanoparticles", B. Hadžić, M. Gilić, M. Petrović-Damjanović, N. Romčević, J. Trajić, D. Timotijević, M. Romčević, **I. Kuryliszyn-Kudelska**, W. Dobrowolski, U. Narkiewicz, D. Sibera; ICOM 2012, The 3rd International Conference on the Physics of Optical Materials and Devices, Belgrade, 2012.

10. „Influence of Preparation Conditions on the Iron Oxide/Graphene Composite Properties", A. Malolepszy, M. Mazurkiewicz-Pawlicka, K. Ciok, **I. Kuryliszyn-Kudelska**, L. Stobinski, The 18th International Conference of the Union of Materials Research Societies in Asia, Taipei, 2017.

11. „Far–Infrared spectroscopy of laser power modified MnO nanoparticles", N. Romčević, B. Babić, B. Hadžić, **I. Kuryliszyn-Kudelska**, M. Romčević, W. Dobrowolski, 2nd International Conference on Environment, Chemical Engineering & Materials, ECEM '18, Malta, June 22-24, 2018.

6.16 Research works presented personally at :

a) national workshops (oral presentations)

1. I. Kuryliszyn „Magnetooptyczne właściwości kryształów MgMnTe o strukturze blendy cynkowej", III Warsztaty Fizyki Półprzewodników Półmagnetycznych, Obory, 1998.
2. I. Kuryliszyn „Magnetyczne Właściwości Kryształów $Pb_{1-x-y-z}Mn_xEu_ySn_zTe$ i $Pb_{1-x-y}Mn_xEu_yTe$", V Warsztaty Fizyki Półprzewodników Półmagnetycznych, Obory, 2000.
3. I. Kuryliszyn „Termiczna obróbka warstw GaMnAs", VII Warsztaty Fizyki Półprzewodników Półmagnetycznych, Obory, 2002.

b) national conferences

1. I. Kuryliszyn-Kudelska, W. D. Dobrowolski, Ł. Kilański, B. Romčević, N. Romčević, D. Sibera, U. Narkiewicz, „Magnetyczne własności nanokrystalicznego ZnO domieszkowanego MnO, CoO oraz Fe_2O_3", Krajowa Konferencja Nanotechnologii, Warszawa, 2009.

6.17 Editorial experience

Co-editor of two issues of Acta Physica Polonica A (APPA Vol. 112 No. 2, 2007 and Vol. 114 No. 25, 2008). The post-conference materials were published in these numbers: texts of invited papers and original works.

Warszawa
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place, date

Izabela Kudelska

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