Mössbauer Spectroscopy
in
Materials Science

MSMS `10

Book of Abstracts
with Programme

January 31 – February 5, 2010

Liptovský Ján, Slovakia

ORGANIZERS

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PROGRAMME

Sunday, 31 January, 2010

16:00 – 19:00 **Registration**
19:00 **Welcome party with dinner**

Monday, 1 February, 2010

7:00 – 8:45 **Breakfast**

8:45 – 8:55 **Opening: Marcel Miglierini**

PLENARY SESSION 1: ***Chair: I. Sergueev***

8:55 – 9:20 **M. Kopczewicz:** NOVEL AMORPHOUS Fe-Zr-Si(Cu) BORON-FREE ALLOYS
9:20 – 9:45 **J. Bednarčík**: THERMAL EXPANSION OF NANOPERM-TYPE ALLOYS FROM IN-SITU X-RAY DIFFRACTION
10:00 - 10:30 **M. Miglierini**: ^{57}Fe and ^{119}Sn MÖSSBAUER EFFECT STUDY OF Fe-Sn-B AMORPHOUS ALLOYS

10:10 – 10:40 **Coffee break**

PLENARY SESSION 2: ***Chair: E. Elsukov***

10:40 – 11:05 **I. Sergueev:** NUCLEAR FORWARD SCATTERING ON NICKEL AND GERMANIUM MÖSSBAUER ISOTOPES
11:05 – 11:30 **C. Aparicio:** HIGH TEMPERATURE DECOMPOSITION OF ALMADINE AND PYROPE IN REDUCING ATMOSPHERE
11:30 – 11:55 **V. Slugeň:** CORROSION AT NUCLEAR POWER PLANT FROM MÖSSBAUER SPECTROSCOPY POINT OF VIEW

12:00 – 14:30 **Lunch**

14:30 – 17:30 **Tutorial**

17:30 – 19:00 **Dinner**

Tuesday, 2 February, 2010

7:00 – 8:55	**Breakfast**

PLENARY SESSION 3:

Chair: V. Sedykh

8:55 – 9:20	**A Lančok:** STUDY OF Fe-Co NANOCOMPOSITE FILMS
9:20 – 9:45	**J. Frydrych:** DEPOSITION AND CHARACTERIZATION OF NANOCRYSTALLINE HEMATITE CATALYTIC FILMS
10:00 - 10:30	**B. David:** MÖSSBAUER SPECTROSCOPY OF Fe-BASED NANOPOWDERS BY MICROWAVE PLASMA TORCH

10:10 – 10:40	**Coffee break**

PLENARY SESSION 4:

Chair: K. Ruebenbauer

10:40 – 11:05	**E. Elsukov:** NANOSTRUCTURE AND PHASE FORMATION UNDER SEVERE MECHANICAL TREATMENT OF Fe-BASED SYSTEM
11:05 – 11:30	**V. Šepelák:** MECHANOSYNTHESIS AND LOCAL STRUCTURE OF NANOCRYSTALLINE STANNATES
11:30 – 11:55	**K. Zábranský:** PREPARATION AND PROPERTIES OF Fe-Co NANOPARTICLES

12:00 – 14:30	**Lunch**

PLENARY SESSION 5:

Chair: M. Kopczewicz

14:30 – 14:55	**M. Oshtrakh:** MÖSSBAUER SPECTROSCOPY OF MAGNETITE AND MAGHEMITE NANOPARTICLES FOR MAGNETIC FLUIDS: A PROBLEM OF SPECTRA FITTING AND INTERPRETATION
14:55 – 15:20	**J. Degmová:** INFLUENCE OF WEAK EXTERNAL MAGNETIC FIELD ON AMORPHOUS AND NANOCRYSTALLINE Fe-BASED ALLOYS
15:20 – 15:45	**P. Roupcová:** CHANGES OF PHASE COMPOSITION OF $NaAlH_4$ BASED COMPLEX HYDRIDE
15:45 – 16:10	**V. Sedykh:** COMPARISON OF FEATURES OF STRUCTURAL TRANSFORMATIONS IN $LaMnO_{3+\delta}$ and $La_{1-x}Sr_xMnO_{3+\delta}$

17:30 - 19:00	**Dinner**

Wednesday, 3 February, 2010

7:00 – 8:55	**Breakfast**

PLENARY SESSION 6: ***Chair: V. Šepelák***

8:55 – 9:20	**N. Chistyakova:** MÖSSBAUER INVESTIGATIONS OF FORMATION OF IRON MINERALS BY ALKALIPHILIC BACTERIUM
9:20 – 9:45	**M. Oshtrakh:** MÖSSBAUER STUDY OF VITAMINS AND DIETARY SUPPLEMENTS CONTAINING FERROUS FUMARATE AND FERROUS SULPHATE
9:45 - 10:10	**K. Ruebenbauer:** MÖSSBAUER SPECTROSCOPY OF IRON-BASED SUPERCONDUCTOR FeSe

10:10 – 10:40	**Coffee break**

PLENARY SESSION 7: ***Chair: N. Chistyakova***

10:40 – 11:05	**V. Nadutov:** MÖSSBAUER AND SANS STUDIES OF ANTI-INVAR Fe-Ni-C ALLOY UNDER MAGNETIC FIELD
11:05 – 11:30	**E. Elsukov:** SOLID-PHASE REACTIONS IN THE $Mo_{80}Fe_{20}$ SYSTEM DURING MECHANICAL ALLOYING
11:30 – 11:55	**V. Semenov:** FEATURES OF THE QUANTITATIVE ANALYSIS IN MÖSSBAUER SPECTROSCOPY

12:00 – 14:30	**Lunch**
14:30 – 17:30	**Tutorial**
17:30 – 19:00	**Dinner**

Thursday, 4 February, 2010

7:00 – 8:55	**Breakfast**

PLENARY SESSION 8: ***Chair: O. Schneeweiss***

8:55 – 9:20	**J. Čuda:** MÖSSBAUER STUDY OF POWDERED ILMENITE ($FeTiO_3$) - REVISITED
9:20 – 9:45	**Z. Marušák:** THERMAL DECOMPOSITION OF IRON(III) SULPHATE
9:45 - 10:10	**L. Machala:** THERMAL DECOMPOSITION OF BARIUM FERRATE(IV) IN AIR

10:10 – 10:40 **Coffee break**

PLENARY SESSION 9: ***Chair: M. Oshtrakh***

10:40 – 11:05	**O. Schneeweiss:** MÖSSBAUER STUDY OF IRON PHASES OF IRON THIN FILMS ON Si/SiO$_x$ SUBSTRATE AND AT DEPOSITED CARBON NANOTUBES
11:05 – 11:30	**M. Bystran:** HYPERFINE INTERACTIONS IN SrFe$_2$As$_2$
11:30 – 11:55	**M. Mašláň:** CLOSING

12:00 – 14:30 **Lunch**

14:30 – 17.30 **Excursion**

19:00 – 23:00 **Conference dinner**

Friday, 5 February, 2010

7:00 – 10:00 **Breakfast**

Departure

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ABSTRACTS

HIGH TEMPERATURE DECOMPOSITION OF ALMANDINE AND PYROPE IN REDUCING ATMOSPHERE

Claudia Aparicio, Jan Filip, Miroslav Mašláň

Centre for Nanomaterial Research, Olomouc, Czech Republic

Thermal decomposition of two garnets of near end-member composition – almandine $(Fe_{2.85}Mg_{0.15})(Al_{1.99})Si_{2.99}O_{12}$ and pyrope $(Mg_{2.22}Fe_{0.47}Ca_{0.33})(Cr_{0.11}Fe_{0.06}Al_{1.81})Si_{2.98}O_{12}$ – has been carried out in reducing atmosphere (forming gas: 10% of H_2 in N_2). High-temperature behavior of both samples was monitored using of simultaneous thermogravimetry and differential scanning calorimetry (heating rate of 10 °C/min). The decomposition of almandine and pyrope turned out to proceed at slightly different temperatures above 1000 °C. Subsequently, two series of samples were prepared based on results of thermal analysis: almandine (ALM) heated up to 950 °C, 1070 °C and 1200 °C, and pyrope (PY) heated up to 1000 °C, 1100 °C, 1125 °C and 1200 °C. The identification of the decomposition products was performed by using of X-ray powder diffraction and Mössbauer spectroscopy (Figure 1). The common decomposition products are metallic iron and spinel phase, while the other products include fayalite, cristobalite and cordierite for almandine; and pyroxene and anorthite for pyrope. The formation of this last component was enabled due to Ca content in pyrope.

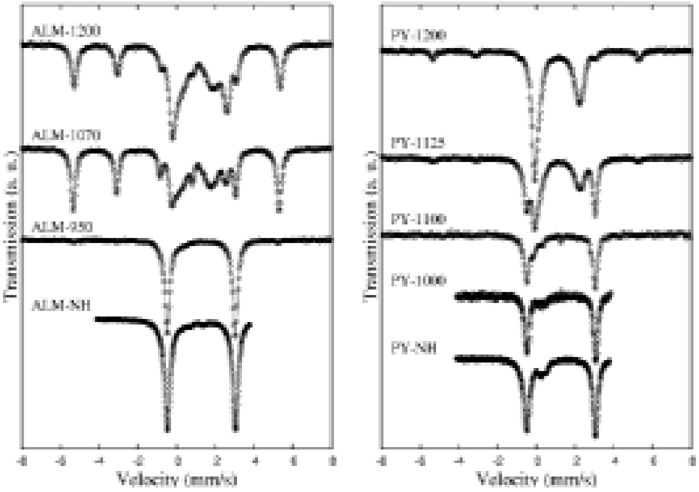

Figure 1: Mössbauer spectra demonstrating the thermal decomposition of almandine (left) and pyrope (right) under reducing atmosphere.

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NOTES

THERMAL EXPANSION OF NANOPERM-TYPE ALLOYS FROM IN-SITU X-RAY DIFFRACTION

J. Bednarcik[a], **M. Miglierini**[b,c], **C. Curfs**[d], **H. Franz**[a]
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NANOPERM-type alloys with nominal compositions of $(Fe_{1-x}Co_x)_{79}Mo_8Cu_1B_{12}$ and $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$ ($x = 0$ and 0.5) were prepared in a form of 6 mm wide and about 20 μm thick ribbons by single-roller melt-spinning technique. Room temperature Mössbauer (MSB) effect experiments on as-prepared amorphous ribbons were carried out in transmission geometry using a ^{57}Co/Rh source mounted on a constant acceleration driving system. Temperature evolution of the as-quenched ribbons during constant-rate heating (10 °C/min) was continuously followed using a high-energy (88 keV) X-ray diffraction (XRD), performed on the ID11 undulator beamline at the ESRF (Grenoble, France). MSB spectroscopy and XRD confirm amorphous nature of melt-spun ribbons. Furthermore, MSB reveals significant change of magnetic state of the as-quenched precursors when substituting of Fe by Co ($x=0.5$). Analysing series of XRD patterns in a reciprocal space yields thermal expansion of amorphous alloys providing insight about the thermally activated effects such as relaxation and crystallization.

This work was supported by the grants VEGA 1/0033/10 and MSM6198959218.

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NOTES

HYPERFINE INTERACTIONS IN $SrFe_2As_2$

M. Bystran[1], **J. Kohout**[1], **K. Závěta**[1] **and A. Lančok**[2]

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In 2008, one of the most significant events in the field of superconductivity was the discovery of a new class of high-temperature superconductors (SC) based on iron arsenide layered structures, with maximum critical temperature $T_C \approx 56K$. Three related Fe-As SC types of compounds have been known by now. We report on hyperfine interactions in $SrFe_2As_2$, a member of the group consisting of 122 phases, i.e. three-component AFe_2As_2 arsenides where A = Ca, Sr, Ba or Eu. Similarly to the cuprates, the parent compounds tend to be antiferromagnetic and superconductivity emerges under chemical doping (K, Na, Co, Ni) or in some cases at high pressure once antiferromagnetism is suppressed. $SrFe_2As_2$ crystallizes in the tetragonal structure at room temperature and exhibits structural transitions at 203K to orthorhombic lattice symmetry in agreement with the group-subgroup relationship between *I*4/*mmm* and *Fmmm*. ^{57}Fe Mössbauer spectroscopy (MS) experiments in standard transmission geometry reveal hyperfine field splitting below the structural phase transition temperature. Temperature dependence of the MS spectra of the samples with thickness $\approx 80 \mu m$ was measured. At room temperature a singlet or narrow doublet with isomer shift *IS*=0,41mm/s was observed corresponding to the crystal structure. At low temperatures a sextet ($B_{hf} \approx 11.9$ T) was present confirming the magnetic order of the iron magnetic moments probably of the antiferromagnetic type. After grinding the samples and exposing them to air and moisture a doublet was found in all the spectra. We believe that this is due to defects or impurities produced by processing.

This work is supported by AVCR project KAN400100653 and MSMT project VZ 0021620834.

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NOTES

MÖSSBAUER STUDY OF POWDERED ILMENITE ($FeTiO_3$) – REVISITED

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Ilmenite ($FeTiO_3$) is an interesting material for its magnetic properties particularly in a ilmenite-hematite solid solution, in which it governs magnetic behaviour of hematite causing hematite to be canted with strong magnetic response. In solution with hematite ilmenite behaves as a magnetically hard phase and it induces exchange bias phenomenon in the solid solution. In addition, ilmenite-hematite solid solutions exhibit a strong and time stable remanent magnetization and that is why they are now used as a model system for explanation of crustal magnetism of soils and rocks on Mars.

For better understanding of role of ilmenite in the ilmenite-hematite system, we re-examined the Mössbauer spectra and macroscopic magnetic properties of ilmentite in the form of a commercial powdered sample of $FeTiO_3$. We measured the Mössbauer spectra of ilmenite in the paramagnetic regime from 57 K to 300 K and in a magnetically ordered state below 57 K. Macroscopic magnetic measurements were carried out by an MPMS XL–7 magnetometer to determine a temperature dependence of the magnetization and hysteresis loops of this sample. We show how to correctly fit the Mössbauer spectra below 57 K both spectra and magnetization measurements confirm the below ordering temperature of ilmentite it behaves as canted antiferromagnetic material with significant magnetic hardening at low temperatures.

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MÖSSBAUER SPECTROSCOPY OF Fe-BASED NANOPOWDERS SYNTHESISED BY MICROWAVE PLASMA TORCH

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[2]*Faculty of Science, Masaryk Univeristy, Brno, Czech Republic*

Microwave plasma torch at atmospheric pressure was used for the synthesis of Fe-based nanoparticles [1]**.** The discharge was ignited in argon which flowed through the central gas flow channel whereas the reactive mixture of H_2/O_2 gas and $Fe(CO)_5$ vapour was added by a concentric opening of the outer channel. The morphology and composition of the synthesised nanopowders were studied by TEM, XRD, Raman spectroscopy and Mössbauer spectroscopy.

Only γ-Fe_2O_3 (d_{XRD} = 20 nm) phase was identified by XRD in the **T89** sample. However the Fe_3O_4 characteristic peaks (669 cm^{-1}, 349 cm^{-1}) were also observed in the Raman spectrum.

γ-Fe_2O_3 (d_{XRD} = 18 nm, 41 wt.%), ε-Fe_2O_3 (d_{XRD} = 27 nm, 28 wt.%), and α-Fe_2O_3 (d_{XRD} = 39 nm, 31 wt.%) phases were identified in the XRD pattern of the **T104** sample. In its Raman spectrum the characteristic bands of α-Fe_2O_3 were observed besides the week bands of γ-Fe_2O_3 and Fe_3O_4. The bands of ε-Fe_2O_3 were overlapped with the bands of α-Fe_2O_3, Fe_3O_4, and γ-Fe_2O_3.

In the **T107** sample α-Fe (d_{XRD} = 48 nm, 51 wt.%) and Fe_3O_4 (d_{XRD} = 10 nm, 49 wt.%) phases were found by XRD. The presence of Fe_3O_4 was clearly confirmed by Raman spectroscopy.

[1] L. Zajíčková, P. Eliáš, M., Jašek, O. et al. Plasma Phys. Control. Fusion 47 (2005) B655-B666

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NOTES

INFLUENCE OF WEAK EXTERNAL MAGNETIC FIELD ON AMORPHOUS AND NANOCRYSTALLINE Fe-BASED ALLOYS

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NANOPERM, HITPERM and FINEMET amorphous and nanocrystalline alloys were measured by Mössbauer spectrometry in the weak magnetic field of 0.5 T. It was shown that the most sensitive parameters of Mössbauer spectra are the intensities of 2^{nd} and 5^{th} lines. Rather small changes were observed also in the case of internal magnetic field values.

The spectrum of nanocrystalline NANOPERM showed the increase of A_{23} parameter (ratio of line intensities) from 2.4 to 3.7 and decrease of internal magnetic field from 20 to 19 T for amorphous subspectrum under the influence of magnetic field. Spectrum of nanocrystaline FINEMET shown decrease of A_{23} from 3.5 to 2.6 almost without change of internal magnetic field value. In the case of amorphous NANOPERM and FINEMET samples the changes are almost negligible.

HITPERM alloy shown the highest sensitivity to the weak magnetic field, when A_{23} increased from 0.4 to 2.5 in the external magnetic fields. The A_{23} of crystalline subspectrum increased from 2.7 to 3.8 and the value of internal magnetic field corresponding to amorphous subspectrum increased from 22 to 24 T.

The behavior of nanocrystalline alloys under weak external magnetic field was analyzed within three–level relaxation model of magnetic dynamics in an assembly of single-domain particles [1].

[1] M. A. Chuev: J. Phys. Cond. Mat. 20 (2008) 505201

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NANOSTRUCTURE AND PHASE FORMATION UNDER SEVERE MECHANICAL TREATMENT OF FE-BASED SYSTEM

E.P. Yelsukov

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So far, the mechanism of phase formation under mechanical alloying and grinding is a subject of extensive studies.The results on nanostructure formation, mechanical alloying of Fe-based binary systems with interstitial (B, C) and substitutional (Al, Si, Ge, Sn) elements, deformation-induced dissolution of interstitial (Fe2B, Fe3C) and substitutional (FeSn) phases in α-Fe are presented in this report. The following scheme of nanostructural formation is considered: increasing of dislocation density → polygonization → dislocation pile-up → dynamic recrystallization (grain size $L\approx100$ nm) → deformation twinning and polymorphous transformation ($L\leq10$ nm).A term of interface in nanostructure is defined as the area including boundary and close-to-boundary distorted zones. The width of interface has been estimated to be equal of about 1 nm.It has been established that solid-state reactions under severe mechanical treatment begin on condition that α-Fe reaches nanostructural state. The next stage includes penetration of the second component (B, C, Al, Si, Fe, Sn) atoms along grain boundaries of α-Fe and its segregating at boundaries. This segregation is the source to form the first phase in interfaces. Depending on the type of the second component, different phases can be formed: intermetallic compounds, supersaturated solid solutions and amorphous phases.

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NOTES

SOLID-PHASE REACTIONS IN THE $MO_{80}FE_{20}$ SYSTEM DURING MECHANICAL ALLOYING

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At present vast amount of information concerning evolution of metallic systems during mechanical alloying is available. Fe-based systems are most thoroughly studied due to the great practical importance. As it follows from published sources Fe-Mo systems were investigated by several workgroups, with Mo concentration ranging from 0 to 50%. However there were not found any papers where solid-state reactions in Fe-Mo alloys with Mo concentration exceeding 50% were described in details.

$Mo_{80}Fe_{20}$ (in atomic ratio) sample was chosen for the study. Mechanical alloying is carried out by laboratory ball mill Pulverizette 7 in Ar atmosphere. The vials and the balls were made of SHKh-15 bearing steel. Sample load weight was 10g. Powder contamination was controlled before and after mechanical alloying. The milling time ranged from 0.5 up to 24h.

The Mössbauer spectroscopy was performed on SM2201DR spectrometer. For the X-ray analysis DRON-3 diffractometer with Cu Ka filtered rays was used.

At the early stage of mechanical alloying the metastable $Mo_{63}Fe_{37}$ HCP-phase was formed. The formation of hcp-phase probable is due to minor quantity (1.5-2 at.%) MoO_3 phase in the initial Mo powder. Solid-state reactions took place only when the system had become nanocrystalline. The supersaturated solid solution Mo(Fe) with 20 at.% Fe (milled for 24h) was obtained. Two stages of mechanical alloying were observed: Mo+Fe→ bcc Mo-Fe + hcp Mo-Fe and Mo-Fe + hcp Mo-Fe → bcc Mo-Fe + Am Mo-Fe.

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NOTES

DEPOSITION AND CHARACTERIZATION OF NANOCRYSTALLINE HEMATITE CATALYTIC FILMS

Jiri Frydrych, Libor Machala, Miroslav Mashlan
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A new technique for deposition of nanocrystalline hematite film with photocatalytic applicability has been developed. Solid iron(III) chloride hexahydrate, used as a precursor, is thermally decomposed in dynamic air atmosphere, while iron(III) chloride oxide (FeOCl) film is formed on a substrate surface (FTO-coated glass). Afterwards, this FeOCl layer is converted to hematite utilizing a stream of hot air.

Phase compositions of the as-prepared samples were primarily characterized using ^{57}Fe Conversion Electron Mössbauer Spectroscopy and Grazing Angle X-Ray Diffractometry. Atomic Force Microscopy was used to evaluate the film thickness. The field emission Scanning Electron Microscopy was used to observe film morphology and Energy Dispersive X-ray analysis was carried out to evaluate element occurrence.

Main results of our measurements as well as early findings of photocatalytic activity evaluation will be presented. The photocatalytic activity is under influence of many factors, e.g. a chemical purity, a presence of doping atoms, film thickness and its morphology, what will be also discussed.

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NOTES

INVESTIGATIONS OF IRON MINERALS FORMED BY DISSIMILATORY ALKALIPHILIC BACTERIUM WITH ^{57}FE MÖSSBAUER SPECTROSCOPY

N.I. Chistyakova[a], **V.S. Rusakov**[a], **A.A. Shapkin**[a],
T N Zhilina[b], **D.G. Zavarzina**[b], **A. Lančok**[c], **J. Kohout**[d]
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Anaerobic alkaliphilic bacteria *Geoalkalibacter ferrihydriticus* (strain Z-0531) isolated from a bottom sediment sample from the weakly mineralized soda Lake Khadyn. The strain uses the amorphous Fe(III)-hydroxide (AFH) as an electron acceptor and acetate CH_3COO^- as an electron donor. Mössbauer investigations of solid phase samples obtained during the process of the bacterium growth were carried out at room temperature, 77 K, 4.2 K without and with the presence of an external magnetic field (6 T) applied perpendicular to the γ-beam. The magnetic structure of obtained minerals was refined. The AFH and quinone concentratione influence on iron reducing process were investigated. It has been found that the increase in AFH concentration leads to a decrease in siderite content and to an increase in the content of the magnetically ordered phases. The quinone concentration has a significant influence on the size of the particles of magnetically ordered phases. The concentration range of AFH 30 mM-70 mM and quinine 0.7 g/l – 1.0 g/l are the regions of unstable phase formations by strain Z-0531 for which the process of biomineralization is sensitive to fluctuations in bacterium cell number.

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NOTES

NOVEL AMORPHOUS Fe-Zr-Si(Cu) BORON-FREE ALLOYS

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Amorphous and nanocrystalline Fe-based alloys have been studied extensively for recent years due to their excellent soft magnetic properties. Such alloys are very attractive for technical applications. Until now all known technically important alloys contain boron addition that increases glass forming ability and improves thermal stability of the amorphous alloys. However, boron is a relatively expensive element, therefore it would be of significant economical benefit to replace boron by a cheaper glass forming element, e.g., Si.

It is expected that amorphous Fe-Zr-Si(Cu) alloys revealing excellent soft magnetic properties can be prepared in a fairly wide composition range by the melt quenching technique.

The aim of the present study is the preparation and structural characterization of the novel amorphous $Fe_{80}(Zr_xSi_{20-x-y})Cu_y$ (x = 6 –10 at. %, y = 0, 1 at. %) boron-free alloys that will show soft magnetic properties comparable with the conventional iron-based amorphous rapidly solidified materials. The amorphous Fe-Zr-Si(Cu) alloys have been prepared in Ar atmosphere by the melt quenching technique in the form of ribbons about 1 mm wide and 20 μm thick. The Mössbauer spectroscopy, x-ray diffraction (XRD) and differential scanning calorimetry (DSC) were used in the characterization of the melt quenched alloys. The fully amorphous alloys, as determined by XRD and Mössbauer measurements, were obtained for x = 6, 7, 9 or 10, and y = 0, 1 with the highest cooling rate used, equivalent to the linear velocity at the wheel of 55 m/s in the single roller melt spinning machine.

The Mössbauer spectra of the as-quenched ribbons with $6 \le x \le 10$ at. % are composed of the broadened sextets characteristic for the ferromagnetic amorphous alloys. The hyperfine field distributions were calculated from the transmission spectra. No traces of the crystalline fractions were detected. The XRD patterns recorded for the amorphous alloys consist of only one broad peak characteristic for the amorphous phase.

The DSC measurements at 20K/min revealed that the onset temperatures of crystallization of the amorphous Fe-Zr-Si(Cu) alloys are rather high. In the available temperature range (50 – 720°C) the DSC curves of the amorphous alloys revealed one or two crystallization peaks, depending on the alloy composition. For several amorphous alloys studied the second crystallization step was expected at temperatures exceeding the available temperature range for DSC studies.

The soft magnetic properties of amorphous Fe-Zr-Si(Cu) alloys have been studied by the specialized "rf-Mössbauer" technique in which the spectra were recorded during exposure of the samples to the rf field of 0 to 20 Oe at about 61 MHz. The complete rf-collapse effect was observed. This study was accompanied by the conventional measurements of quasistatic hysteresis loops. Typical coercive fields determined for amorphous Fe-Zr-Si(Cu) alloys varied from 24 A/m to about 100 A/m, depending on alloy composition. Such coercivities correspond well to those of the conventional boron-containing soft magnetic amorphous Fe-based alloys. The coercivity was significantly larger for the partially nanocrystallized alloys.

It is concluded that the boron-free amorphous Fe-Zr-Si(Cu) alloys studied here (with $6 \le x \le 10$ at. %, y=0, 1 at. %) are good candidates for practical applications as the economical soft magnetic materials.

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NOTES

STUDY OF Fe-Co NANOCOMPOSITE FILMS

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Mössbauer study of nanogranular ferromagnetic FeCo films is presented. Two ways of production of nanocomposite systems were employed: (i) a hollow cathode plasma jet deposition process, and (ii) laser ablation from Fe-Co metallic target by means of a KrF excimer laser and r.f. magnetron sputtering. Conversion electron Mössbauer spectroscopy (CEMS) at ambient temperature using ^{57}Co/Rh source provided spectra which were decomposed into 3 sextets with hyperfine fields (B_{hf}) of 32, 33.8, and 35 T and one doublet. The relative areas of the components depend upon the composition of the sample and conditions of its preparation. Complementary information was obtainedby nuclear magnetic resonance (NMR) of ^{57}Fe and ^{59}Co nuclei performed in zero external magnetic fields at 4.2 and 300 K. The increase of B_{hf} at ^{59}Co by ~3.5 T with respect to Co powder agrees with the effect of Fe atoms in the nearest neighbourhood. Similar effect is responsible for an increase of B_{hf} at ^{57}Fe nuclei due to Co nearest neighbours. Distribution of B_{hf} indicates a presence of amorphous phase and very small crystalline particles. This was confirmed by HRTEM. The films are composed of crystalline nanoparticles, 5-20 nm in size, embedded in amorphous matrix.

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NOTES

THERMAL DECOMPOSITION OF BARIUM FERRATE(VI) IN AIR

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Thermal decomposition of barium ferrate(VI) ($BaFeO_4$) in static air was investigated using ^{57}Fe Mössbauer spectroscopy, X-ray powder diffraction (XRD), thermal analysis and microscopic techniques (TEM, SEM). $BaFeO_3$ was confirmed to be the primary decomposition product above 190 °C. $BaFeO_3$ was found to be unstable in air at high and/or room temperature reacting with air-CO_2 towards orthorhombic $BaCO_3$ and speromagnetic amorphous Fe_2O_3 nanoparticles (< 5 nm). The room temperature Mössbauer spectrum of the product of decomposition at 300 °C consists of two components. A singlet ($\delta_{Fe} = -0.27$ mm/s) corresponds to tetravalent iron atoms in rhombohedral $BaFeO_3$ structure, while a doublet ($\delta_{Fe} = 0.35$ mm/s, $\Delta E_Q = 0.56$ mm/s) was ascribed to amorphous Fe_2O_3. At 600 °C, the solid state reaction between the as-formed Fe_2O_3 and $BaCO_3$ towards barium ferrite ($BaFe_2O_4$) nanoparticles (20-100 nm), takes place. The overall decomposition mechanism of $BaFeO_4$ in air can be described by the following chemical equations:

300 °C:

$$BaFeO_4 \rightarrow BaFeO_3 + 1/2\ O_2$$

$$BaFeO_3 + CO_2 \rightarrow BaCO_3 + 1/2\ Fe_2O_3 + 1/4\ O_2$$

600 °C:

$$Fe_2O_3 + BaCO_3 \rightarrow BaFe_2O_4 + CO_2.$$

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THERMAL DECOMPOSITION OF IRON(III) SULFATE

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Influence of conditions (temperature, particle size, diffusion conditions, layer thickness of the precursor and time of calcinations) on solid-state thermal decomposition of iron(III) sulfate in air towards iron(III) oxides and their polymorphous transformations are discussed. Mechanism of thermal decomposition and polymorphous transformations have been investigated mainly using ^{57}Fe Mössbauer spectroscopy, X-ray powder diffraction (XRD), and thermal analysis (DSC, DTA) methods. γ-Fe_2O_3 (maghemite) and β-Fe_2O_3 were identified as primary products of iron(III) sulfate decomposition. Particle size induced polymorphous transformations from maghemite to hematite via ε-Fe_2O_3 and simultaneously from β-Fe_2O_3 to hematite has a direct influence on thermal decomposition of residual iron(III) sulfate. This is related to a diffusion of gaseous SO_3 from the bulk of particle through iron(III) oxide surface layer. The overall decomposition mechanism is an unique exhibition of polymorphous transformations of iron(III) oxide.

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NOTES

^{57}Fe AND ^{119}Sn MÖSSBAUER EFFECT STUDY OF Fe-Sn-B AMORPHOUS ALLOYS

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Amorphous metallic alloys (the so-called metallic glasses) exhibit interesting magnetic properties which allow their use in many a practical applications (for example electric transformers, sensors, etc.). One of the first systems, which were prepared by the method of rapid quenching, is the Fe-B metallic glass. Recently, possibility of nanocrystallization of this system with incorporation of few at.% of Cu is investigated. Our interest is focused on the effect of Sn addition upon hyperfine interactions and structural features of the $Fe_{100-x}Sn_5B_x$ (x = 15, 17, 20) alloy. As-quenched ribbons are XRD amorphous for x = 15 and 17 whereas for x = 20 traces of quenched-in crystallites are revealed. Progress of crystallization is followed on samples annealed for 30 min at temperatures that cover the first crystallization peak of the DSC (differential scanning calorimetry) curve. Mössbauer spectra were recorded at room temperature. Using ^{57}Fe and ^{119}Sn Mössbauer spectrometry, inspection of both types of nuclei and their local arrangements is possible. The obtained Mössbauer spectra are evaluated by distributions of hyperfine magnetic fields in addition to crystalline components. The evolution of crystalline fraction as well as average values of hyperfine magnetic fields and isomer shifts is discussed as a function of the alloy composition and temperature of annealing.

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NOTES

MÖSSBAUER AND SANS STUDIES OF ANTI-INVAR Fe-Ni-C ALLOY UNDER MAGNETIC FIELD

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It was revealed that the f.c.c.-Fe-(20-25)%Ni-C alloys show abnormally enhanced thermal expansion coefficient (TEC) (~ 20 10^{-6} K^{-1}) within the temperature range between the Curie temperature and martensitic point and it is accompanied by almost temperature-insensitive behaviour that points to anti-Invar effect by opposite to Invar behaviour. Usually an essential differences between Invar and anti-Invar behaviour is considered in terms of the magnetically ordered-disordered states: in the former one the volume effect occurs essentially in magnetically ordered state, whereas in the latter it occurs in the magnetically disordered one. However, the Mössbauer analysis and SANS experiment in external magnetic field of 1,5 – 5 T have revealed an existence of magnetic inhomogeneities in the studied anti-Invar alloys below and above the Curie point. This is consistent with the ideas concerning ferromagnetic and antiferromagnetic exchange interspin interactions in the studied alloys and the earlier hypothetical model regarding moment-volume instability. The effect of carbon on anti-Invar behavior of the f.c.c.-Fe-(20-25)%Ni-C alloys is considered from the point of view of stabilization of austenitic phase at low temperatures and magnetic ordering.

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NOTES

STUDY OF VITAMINS AND DIETARY SUPPLEMENTS CONTAINING FERROUS FUMARATE AND FERROUS SULFATE USING MÖSSBAUER SPECTROSCOPY

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Mössbauer spectroscopy is a useful tool to study various materials including pharmaceuticals. Study of different iron containing vitamins and dietary supplements using the Mössbauer spectroscopy revealed various ferrous and ferric impurities. In the present work we consider results of the application of the Mössbauer spectroscopy with a high velocity resolution to study several new vitamins and dietary supplements containing ferrous fumarate ($FeC_4H_2O_4$) such as Elevit, Vitrum, Children's Multi Vitamin and Multi For Her, and ferrous sulfate ($FeSO_4$) such as Sorbifer Durules, Hemofer® and Falvit®. Mössbauer spectra of all samples were measured at 295 K in 4096 channels. The Mössbauer spectra of samples containing ferrous fumarate were considered for analysis in 2048 channels while those of other samples in 4096 channels. The analysis of the spectra revealed the presence of different components with various relative contributions. As the main component in the spectra of vitamins and dietary supplements containing ferrous fumarate was identified $FeC_4H_2O_4$ while that in the spectra of other samples as $FeSO_4$. The minor components were related to ferrous and ferric compounds which may be due to impurities or results of some transformation of ferrous fumarate or ferrous sulfate. It is interesting to observe small variations of Mössbauer hyperfine parameters for ferrous fumarate and ferrous sulfate in corresponding samples (Fig. 1). This fact may be a result of different preparation methods or/and purity of ingredients used by various manufacturers in the production process.

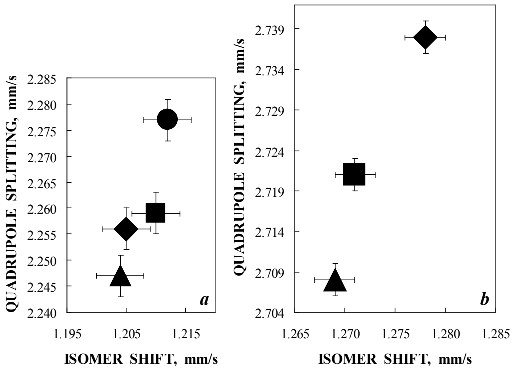

Fig. 1. Plots of Mössbauer hyperfine parameters for ferrous fumarate (***a***) in Elevit (◆), Vitrum (■), Children's Multi Vitamin (▲) and Multi For Her (●), and for ferrous sulfate (***b***) in Sorbifer Durules (◆), Hemofer® (■) and Falvit® (▲).

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NOTES

MÖSSBAUER SPECTROSCOPY OF MAGNETITE NANOPARTICLES FOR MAGNETIC FLUIDS: A PROBLEM OF SPECTRA FITTING AND INTERPRETATION

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Magnetic fluids based on iron oxide nanoparticles are used and developed for various biomedical applications. Therefore, Mössbauer spectroscopy is a useful tool for study of nanoparticles features. In this work we consider the results of the study of two samples: magnetite as prepared and dried magnetite nanoparticles dispersed in Copaiba oil using Mössbauer spectroscopy with a high velocity resolution (using 4096 channels) at 295 and 90 K. Mössbauer spectra of dried magnetite nanoparticles dispersed in Copaiba oil measured at 295 and 90 K are shown in Fig. 1. All spectra were fitted using discrete approach with various numbers of magnetic sextets and paramagnetic doublet (at 295 K only). It was observed that parameters of magnetite nanoparticles as prepared and dispersed in Copaiba oil were different. This fact may be explained as a result of surface interactions of polar molecules of Copaiba oil (for instance, kaurenoic acid) with iron. A large number of magnetic components is not clear yet and may be a result of complicated nanoparticle structure (surface and internal layers and/or domains). Some problems of complicated spectra fitting and interpretation are considered on the basis of the results of various spectra fittings with a large number of varied parameters.

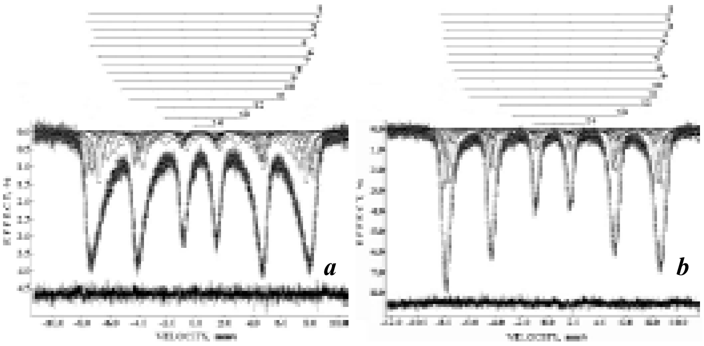

Fig. 1. Mössbauer spectra of dried magnetite nanoparticles dispersed in Copaiba oil measured in 4096 channels at 295 K (***a***) and at 90 K (***b***).

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CHANGES OF PHASE COMPOSITION OF $NaAlH_4$ BASED COMPLEX HYDRIDE

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The hydrogenation and dehydrogenation of complex hydride belong to the hot topics of hydrogen storage research. In this paper we present results of study of $AlNaH_4$ alloyed with Fe chloride. We have investigated influence of time of milling and an effect of ambient atmosphere on properties of this material. The complex hydride sample was prepared by dry milling of mixture of pure $AlNaH_4$ and 2 mol % $FeCl_3.H_2O$ powders in the protective atmosphere 90% Ar+10% H_2. The XRD and Mössbauer spectroscopy were applied for characterisation of the structure of the as-prepared (before milling) powder, and after 0.5; 1 and 2.5 hours of milling. Subsequently, changes during contact with ambient atmosphere were investigated.

The dependence on the time of milling was obtained from XRD measurements. It shows presence of phases of both precursors and their progressive amorphisation with time of milling. The sample after 2.5 hours of milling shows an amorphous phase only. The amorphisation is explained as an effect of severe plastic deformation during the milling and as a role of hydrogen atoms released from the hydride precursor and from the protective atmosphere.

The original $AlNaH_4$ and $FeCl_3$ phases disappeared in the sample exposed to ambient atmosphere. They were replaced by new phases resulted from oxidation and hydrogen decomposition.

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MÖSSBAUER SPECTROSCOPY OF IRON-BASED SUPERCONDUCTOR FeSe

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Superconducting FeSe has been investigated by Mössbauer spectroscopy at various temperatures including strong external magnetic fields applied to the absorber. It was found that isomer shift exhibits sharply defined increase at about 105 K leading to the lowering of the electron density on iron nucleus by $0.02\,\mathrm{electron\ a.u.}^{-3}$. Above jump in the electron density is correlated with the transition from the *P4/nmm* to the *Cmma* structure, while decreasing temperature. Mössbauer measurements in the external magnetic field and for temperatures below transition to the superconducting state revealed null magnetic moment on iron atoms. The principal component of the electric field gradient on the iron nucleus was found as negative on the iron site. Superconductivity with the transition temperature T_c=8 K occurs for a compound being formed close to the FeSe stoichiometry. This compound has *P4/nmm* tetragonal structure at room temperature and transforms into *Cmma* orthorhombic phase between 100-80 K. It was found that T_c strongly depends upon applied pressure raising to 36.7 K at 8.9 GPa and subsequently dropping due to the induced phase transition into some hexagonal structure. Therefore, it is important to look upon phonon and electron density of states versus pressure in this unconventional compound to decide what kind of the boson field is responsible for the Cooper pairs formation. Calculations are performed for the stoichiometric compound within the density functional theory (DFT). Local density approximation (LDA) is used as better suited for the refinement of the crystal structure. A harmonic approximation is used to study lattice dynamics of the system. Results of the calculations are to be discussed in detail. Calculations show that the iron magnetic moment is zero in the ground state of both phases.

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NOTES

COMPARISON OF FEATURES OF STRUCTURAL TRANSITIONS IN $LaMnO_{3+\delta}$ AND $La_{1-x}Sr_xMnO_{3+\delta}$ (x = 0.05 – 0.2)

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The features of the structural transformations in $La_{1-x}Sr_xMn_{0.98}Fe_{0.02}O_{3+\delta}$ (x = 0.05÷0.20) depending on the Sr concentration have been investigated by Mössbauer spectroscopy and X-ray diffraction analysis. The features of the phase formation and suppression in the basic compound $LaMn_{0.98}Fe_{0.02}O_{3+\delta}$ and in $La_{1-x}Sr_xMn_{0.98}Fe_{0.02}O_{3+\delta}$ have been compared and both their common regularities and the essential distinctions between themselves have been revealed.

In undoped $LaMnO_{3+\delta}$ with the stoichiometric composition (*PnmaII* phase, $\delta = 0$) all lattice positions are occupied and only Jahn-Teller Mn^{3+} ions are present. Under oxidation a part of Mn^{3+} transfers to Mn^{4+} and additional oxygen ($\delta > 0$) occupies interstitial positions in the lattice notably distorting it. As the Mn^{4+} concentration increases, phase transitions appear.

Doping with Sr^{2+} occupying La^{3+} sites results in an appearance of vacant oxygen sites in the lattice. Therefore a part of Mn^{3+} ions transfers to Mn^{4+} ions already during synthesis and additional oxygen occupies vacant oxygen sites in the lattice. As the Sr^{2+} concentration increases, the concentration of Mn^{4+} ions and additional oxygen in lattice sites increases that leads to a decrease in an amount of interstitial oxygen and, correspondingly to a decrease in lattice distortions related to it and in the value of quadrupole splitting.

The work was supported by the Russian Foundation for Basic Research (project no. 09--02--00767) and by the Program of Russian Academy of Sciences "Quantum Physics of Condensed Media".

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NOTES

FEATURES OF THE QUANTITATIVE ANALYSIS IN MOSSBAUER SPECTROSCOPY

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The Mössbauer spectroscopy technique belongs to few methods of defining the phase state or crystallographic sites of substance. The Mössbauer spectra bear information on various hyperfine interactions, many of which are indirectly related to the chemical nature of the Mössbauer atom and its nearest environment. Determination of parameters of hyperfine interactions that can be extracted from Mössbauer spectra and used for qualitative analysis is a routine task. In present work we studied the influence of various factors on experimental errors encountered in quantitative defining the phase composition or site populations of the substance under study. Such as measurements geometry, Lamb - Mössbauer coefficients, absorber thickness, efficiency and dead time of the detection system and spectral line shape. The absolute f measurements were made with "black" absorber method. Mössbauer measurements were carried out under carefully controlled of background intensities. Since the accuracy of f evaluation is directly depend on the measurement of background. The influence of a non-uniformity of samples on results of the quantitative analysis is discussed Data analysis we divided into two parts: removal of instrumental artifacts by folding and baseline correction and deconvolution to extract hyperfine parameters of individual local environments. In our approach calibration graphs were drawn by measuring the spectra of a series of analogous samples having different known concentrations. For the same purpose, internal standard method also was used. Experimental data are presented for phase analysis of different samples.

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NOTES

HYPERFINE INTERACTIONS IN NANOCRYSTALLINE $LiFePO_4$ PREPARED BY MECHANOCHEMICAL ROUTE

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Among the typical Li ion battery cathode materials, $LiFePO_4$ of the phospho-olivine family is particularly interesting due to its high energy density, low cost, and environmental compatibility. In the present work, nanocrystalline $LiFePO_4$ with an average particle size of about 10 nm was prepared via far-from-equilibrium mechanochemical processing of the bulk $LiFePO_4$ at room temperature. HR-TEM studies revealed a nonuniform structure of $LiFePO_4$ nanoparticles consisting of an ordered core surrounded by a disordered surface shell/interface region. A comparative ^{57}Fe Mössbauer spectroscopic study of bulk and nanocrystalline $LiFePO_4$ revealed that the near-surface layers of phosphate nanoparticles are disordered due to the strongly distorted geometry of the FeO_6 octahedra. Quantitative information on hyperfine parameters of the nonequilibrium $LiFePO_4$ phase is obtained.

The work is supported by the DFG in the framework of the Priority Program *"Crystalline Nonequilibrium Phases"* (SPP 1415), and the APVV (0728-07).

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NOTES

NUCLEAR FORWARD SCATTERING ON NICKEL AND GERMANIUM MÖSSBAUER ISOTOPES

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Nuclear forward scattering of synchrotron radiation became an important technique to study magnetic and electronic properties of solids. Observation of the nuclear forward scattering relies on the finite lifetime of an excited nuclear state and a pulse structure of synchrotron radiation. Nuclear resonant scattering, delayed in time, is separated from the prompt pulse of incident radiation by a fast detector. The huge intensity of the prompt pulse leads, however, to an overload of the detector which has to be avoided by decreasing the energy bandwidth of synchrotron radiation by appropriate monochromators. For low-energy nuclear resonances this is routinely achieved using special monochromator with high order reflections of perfect silicon crystals, which provide a narrow band pass (~meV) and high angular acceptance matching the angular divergence of synchrotron radiation. However, for nuclear resonances beyond 30 keV this approach is no longer applicable, because the angular widths of high-order reflections become very small.

Here we show how nuclear forward scattering at high-energies can be performed with x-ray optics based on silicon crystals in combination with fast multi element detectors. This was done using low-order reflections with a moderate energy resolution but sufficiently high angular acceptance. The method was applied to study 67.41 keV Mössbauer transition in ^{61}Ni, and 68.75 keV Mössbauer transition in ^{73}Ge. The method can be applied to a series of other Mössbauer isotopes with the energy of transition between 60 and 90 keV.

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NOTES

MÖSSBAUER STUDY OF IRON PHASES OF IRON THIN FILMS ON Si/SiO$_x$ SUBSTRATE AND AT DEPOSITED CARBON NANOTUBES

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Phase composition of ~10 nm thick Fe film on silicon substrates with 200 nm thick SiO$_x$ layer after annealing in vacuum and in CH_4+H_2 was investigated using Conversion Electron Mössbauer spectroscopy (CEMS), X-ray photoelectron spectroscopy, and SEM. CEMS spectra show that the original iron film was formed by Fe^{3+} phase. This phase is stable after the annealing in vacuum below 500 °C but formation of phases with Fe^{2+} and Fe^0 was observed after annealing at higher temperatures. Photoelectron spectroscopies detected changes in morphology after vacuum annealing above 300 °C where the continuity of the iron film was broken. Annealing of the Si/SiO$_x$/Fe samples in CH_4+H_2 atmosphere caused reduction of the original Fe^{3+} to Fe^{2+} and α-Fe. Analysis of CEMS spectra of the sample with grown multi-walled carbon nanotubes shows that the dominating part of iron atoms is present in Fe_3C and Fe_5C_2 phases. Besides their stoichiometric forms spectrum components which can be ascribed to the amorphous $Fe_{5-x}C_{2+x}$ carbides, γ-Fe, and Fe^{3+} were observed.

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CORROSION AT NUCLEAR POWER PLANT FROM MÖSSBAUER SPECTROSCOPY POINT OF VIEW

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Steam generators of four VVER-440 units at nuclear power plants V-1 and V-2 in Jaslovske Bohunice (Slovakia) were gradually changed by new original “Bohunice” design in period 1994-1998. Corrosion processes before and after these design and material changes in Bohunice secondary circuit were studied using Mössbauer spectroscopy during last 25 years. Innovations in the feed water pipeline design as well as material composition improvements were evaluated positively. Mössbauer spectroscopy studies of phase composition of corrosion products were performed on real specimens scrapped from water pipelines or in form of filters deposits. The corrosion of new feed water pipelines system (from austenitic steel) in combination to innovated operation regimes goes dominantly to magnetite. The hematite presence is mostly on the internal surface of steam generator body and its concentration increases towards to top of the body. In the results interpretation it is necessary to consider also erosion as well as scope an type of maintenance activities. The long-term study of phase composition of corrosion products at VVER reactors is one of precondition for the safe operation over the projected NPP lifetime.

Keywords: corrosion, VVER, Mössbauer spectroscopy, NPP lifetime

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PREPARATION AND PROPERTIES OF FeCo NANOPARTICLES

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Fe-Co nanoparticles were prepared from the double Fe- and Co- oxalate. The formation of FeCo metallic phase (solid solution) begun at 250 °C as determined from the X-ray diffraction spectra recorded during heating of the oxalate sample in reducing atmosphere. The reduction finished at 500 °C by formation of α-FeCo phase with the mean particle size of about 17 nm. The final product was investigated using Mössbauer spectroscopy, transmission electron microscopy (TEM), and magnetic measurements. Analysis of Mössbauer spectra shows the ferromagnetic α-FeCo phase represented by two six-line patterns corresponding to interiors and surfaces of the nanoparticles. The hyperfine inductions of these components are 34.06 for the first one and 35.03 T with hypefine field distribution 34.22 T for the second one. In addition to a weak paramagnetic component represented by the doublet with isomer shift 0.14 mm/s and quadrupole splitting 0.49 mm/s was found. The doublet was ascribed to fine particles in superparamagnetic state. The TEM has shown conglomerates of particles with composition about 50 at. % Fe and 50 at. % Co.

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