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DEVELOPMENT OF MEASURING
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THE INNOVATIVE DIRECTION OF SCIENTIFIC INSTRUMENTATION — MÖSSBAUER SPECTROSCOPY AS A FACTOR OF IMPROVING THE BRANCHES OF THE RUSSIAN ECONOMY.
PART 2. CREATION OF NATIONAL RESEARCH EQUIPMENT IN THE FIELD OF MÖSSBAUER SPECTROSCOPY

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The main technical characteristics of the national research equipment for import substitution in the field of Mössbauer spectroscopy created by the IAI RAS are presented. The mass produced IAI RAS Mössbauer spectrometers SM 1101TER and SM 4201 TERLAB, which are widely used in the development of innovative materials, are presented. Mössbauer spectrometers SM 1101TER and SM 4201 TERLAB were in demand in the study of the magnetic and physico-chemical properties of innovative materials, the study of high-temperature superconductivity of compounds, studies of multiferroics and ferroelectrics.

Keywords: innovations, import substitution, Mössbauer spectrometer, gamma-resonance spectrum, Doppler energy modulation, gamma optical scheme

1. IMPORT SUBSTITUTION IN SCIENTIFIC INSTRUMENT MAKING, IN THE PART CONCERNING MÖSSBAUER SPECTROSCOPY

Fundamental and applied research carried out by scientific organizations is related to the need to determine the nature of the substances, their identification and structure determination, determination of their quantities, physical and chemical properties. Conducting advanced scientific research requires the creation of fundamentally new sets of scientific instruments, new techniques and powerful modern computers for processing and storing large amounts of information. The obtained scientific results, experimental scientific instruments and new methods of research constitute a set of new knowledge on the basis of which devices for research and practical use in other areas of the Russian economy are developed [1].

Advanced science can not exist and develop without modern instruments and materials, and at the same time is the creator of new ones. Scientific instrument making is a branch providing scientific research that turns research results into a high-tech product with high added value. In the modern world, scientific instrumentation creates an innovative base for other advanced industries. The level of development of scientific instrument making in the country determines the potential of its competitiveness in high-tech industries.

Today in Russia the institutions subordinate to the Federal Agency of Scientific Organizations have the greatest competence and scientific and educational potential in the field of scientific instrument making.

The introduction of the latest research results into production is one of the key tasks facing the Russian economy today. Scientific instrumentation is an exception in this sense, since there may not be a significant gap in time between the receipt of research results and their introduction into production. Scientific instrumentation allows in one system to create, produce, and use the results of intellectual activity.

The Institute of Analytical Instrumentation of the Russian Academy of Sciences (hereinafter IAI RAS) creates scientific instruments, the rights to which are protected by patents and certificates of Russian ministries, which allows them to be in demand both on the domestic market and on the foreign market of scientific instrumentation.

Traditionally, the Russian Academy of Sciences allocated funds for the renewal of the instrument park. Basically, these funds went for the purchase of imported equipment, which adversely affected the development of domestic scientific instrumentation. In the conditions of anti-Russian sanctions, the renewal of the instrument park is mainly based on domestic scientific instrumentation, which positively affects the development of the industry [2].

The creation of a national sector of the economy that implements competitive research and development, the results of which are in demand by various sectors of the Russian economy, presupposes the availability of a developed scientific infrastructure. A key element of the scientific infrastructure is research equipment, created on the basis of domestic scientific instrumentation.

The Instrument Making Council of the Federal Agency of Scientific Organizations of Russia (the Instrument Engineering Council of the Russian National Academy of Sciences) as a result of the analysis revealed about 30 Russian organizations and enterprises that produce analytical instruments competitive at the world level. These included enterprises that are subordinate to the FAO of Russia.

Given the long-term period of the anti-Russian sanctions, as well as the fact that Russia participates in global production and technological alliances, the issues of import substitution in scientific instrument making have become most significant. We are not talking about replacing imported products with domestic analogues, replacing imported products with domestic analogues will be implemented taking into account the international division of labor in the field of scientific instrument making.

The most significant part of both the Russian and foreign instrument fleet is equipment for studying the structure and composition of substances and properties of materials. In total, this equipment is 52 % of the total number of instruments, 24 % of which is Russian equipment.

FANO Russia together with the Russian Academy of Sciences develops breakthrough directions for the further development of instrument engineering, taking into account Russia's priority areas for the development of science and technology.

IAI RAS traditionally owns information about the main world trends in the development of scientific instrument making. Mössbauer spectroscopy is one of the promising directions for the development of domestic instrument making, replacing imported scientific instruments with domestic scientific instruments.

2. SM 1101TER MÖSSBAUER SPECTROMETER PRODUCED BY THE IAI RAS

SM 1101TER Mössbauer Spectrometer (hereinafter referred to as Spectrometer) realizes a new method of Nuclear Gamma Resonance — Grazing Incidence Mössbauer Spectroscopy (GIMS).

GIMS combines the capabilities of two physical phenomenon: Mössbauer effect and the effect of total external reflection of resonance radiation.

Spectrometer allows simultaneous and indepented recordings of nuclear gamma resonance spectra of specularly reflected and secondary radiation in a wide range of incidence angles.

Spectrometer is designed to study physics and chemical states of surfaces, interfaces and artificial multilayer structures containing Mössbauer isotopes as a native or foreign element with unparalleled depth resolution.

The most effective applications of the Spectrometer are basic (fundamental) research *n* surface phenomena in condensed media, quantum electronics physics and technology, X-ray and synchrotron optics.

Commercial Mössbauer spectrometers do not allow realization of the potentials og GIMS since their optics supports only the Mössbauer effect.

Photo 1. The SM 1101TER Mössbauer Spectrometer

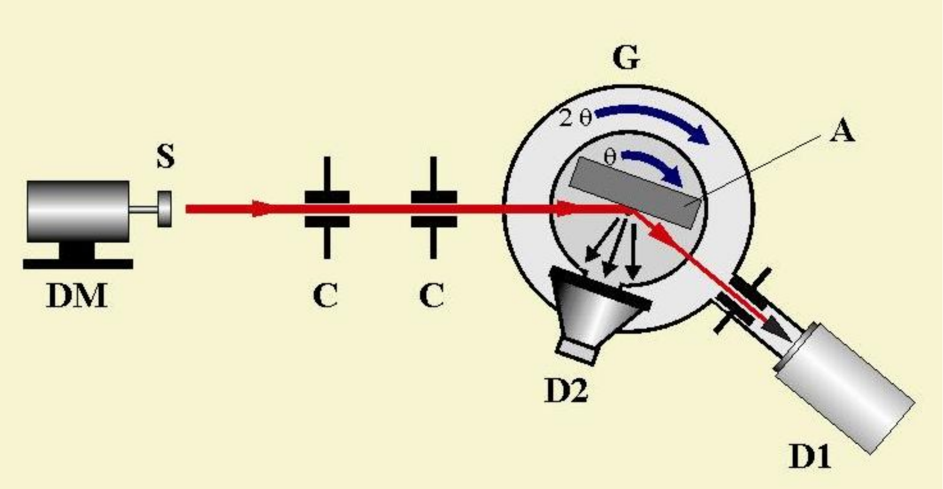


Fig. 1. The gamma optics of the Spectrometer

The proposed Spectrometer provides Mössbauer spectra measurement in four indepented channels simultaneously:

- gamma-radiation specularly reflected by the nuclei and electrons of atoms;
- conversion- and Auger electrons re-emitted by atoms;
- characteristic X-ray radiation and
- gamma radiation scattered by nuclei.

Simultaneous registration of all types of radiation accompanying the primary radiation – substance interaction considerably cuts the measurement times and ensures higher confidence level and reliability of experimental results because the entire information is obtained in a single run and the sample conditions have no time to change.

The results of such combined investigation complement each other and give fuller information both on the ultrathin layer structure of the surface studied and characteristics of grazing incidence radiation interaction with a substance.

Furnishing of laboratories with a SM 1101TER spectrometer will aid in achieving considerable progress in the fields of its application.

For the most part the experimental data obtained with the Spectrometer are of unique nature. Besides, these results will complement information on the surface state from other analytical methods such as, e.g., ESCA and Auger spectroscopy, with the data on microphase, structural and magnetic states.

The SM 1101TER spectrometer can be also used in experiments with the conventional Mössbauer gamma optics: transmission; emission; forward-, back-, and angular scattering; Rayleigh Scattering of Mossbauer Radiation (RSMR); Selective Excitation Double Mössbauer Effect (SEDM) and any combination of there of.

The grazing Incidence Mössbauer Spectroscopy is based on two physical phenomenon: Mössbauer effect and the effect of total external effect of gamma radiation.

At grazing incidence angles in the total external reflection angle range, apart from resonance, energy values, relative intensities and individual spectral linewidths typical of conventional Mössbauer spectroscopy a contribution to experimentally observed Mössbauer spectra are made by such properties of substances studied as electron and nuclear susceptibilities of a medium and their variation with depth, depth distribution of nuclei having different parameters of hyperfine interaction parameters, etc.

The selectivity of the method is defined by the expression for Mössbauer radiation penetration depth $d\perp$ in the vicinity of total external reflection angles:

$$d_{\perp} = \frac{\lambda}{4\pi} \frac{1}{\operatorname{Im}\sqrt{\sin^2\theta + \chi}}.$$

Here λ is the incident radiation wavelength, θ is the incidence angle, χ is generalized electronic and nuclear susceptibility of the medium: $\chi_{\mathrm{el}} + \chi_{\mathrm{nucl}}$.

Equation shows that for a specific sample by varying the resonance incidence angle to the surface of the sample, one can conduct depth selective studies.

The gamma optics of the Spectrometer is presented in Fig. 1.

A narrow plane-parallel γ-radiation beam from the source S placed into a Doppler modulator DM, which

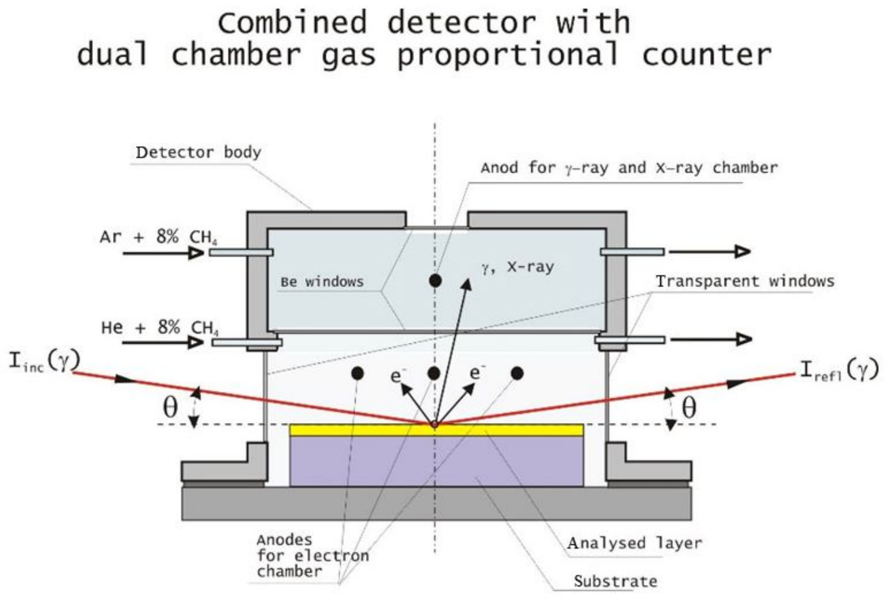


Fig. 2. Combined detector with dual chamber gas proportional counter

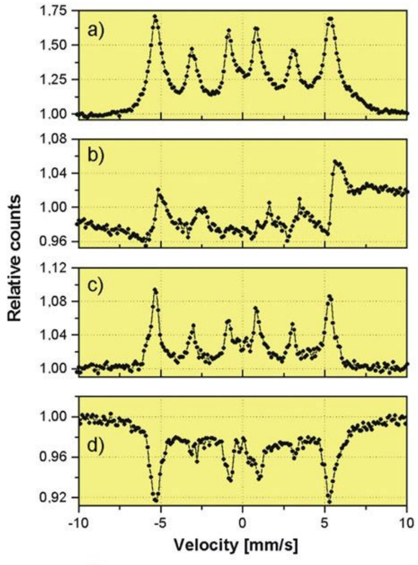


Table 1. Main technical characteristics of Mössbauer spectrometer SM1101TER

No.	Parameter	Value
1	Velocity range, mm/s	50
2	Nonlinearity, %	0.8
3	Velocity reproducibility, μm/s	3.5
4	Number of Channels for Storage	100, 200, 400, 600, 800
5	Channel Capacity	$2^{24}-1$
6	Standard sample linewidth, mm/s	0.23
7	Number of storage channels	4
8	Scanning angle range, mrad	1.5÷120
9	Grazing angle setting accuracy, mrad	0.25
10	Analysis depth, nm	0.2÷1000
11	Resolution in the depth range of 10 to 50 nm, nm	1

is shaped by collimators C, falls onto the sample under study A. A goniometer G is used for high-precision adjustment of the grazing incidence angle. The radiation specularly reflected from the sample id detected by a detector D1, while conversion and Auger electrons, X-rays and scattered gamma-radiation are detected by a combined detector D2 of original design.

The combined detector D2 intended for the grazing incidence geometry (Fig. 2) consists of a proportional two chamber gas counter. The characteristics X- and γ-rays emitted by the sample pass the lower chamber sensitive space for electron detection almost without loss, a beryllium foil 50 μm thick separating the chamber volumes, and detected in the upper chamber.

A distinctive feature of this detector is its transparent windows of Plexiglas 0.5 mm thick for coming and outgoing radiations. This makes it possible to adjust the incidence angle using laser radiation and filter out the X-ray component (6.4 keV) in the original flux.

The exit window allows detection of specularly reflected gamma radiation simultaneously with electrons and X-rays.

The detector housing is made of aluminum, has a sectional construction with seals of vacuum rubber. The sample is placed in the chamber for electron detection, so that its surface to be analyzed faces the working volume of the chamber through which a He + 8 % CH_4 gas mixture flows at flow rate of 2 sm^3/min. The anode in this chamber consists of three tungsten filaments 20 μm in diameter attached on an insulating support 3 mm apart from sample surface. To reduce the noise component from the scattered radiation, the inner walls of the electron chamber are closed with plexiglass inserts so that the working volume of the chamber is only between the sample surface and beryllium window separating the upper chamber volume and corresponds to the sample surface area.

The working gas in the chamber for detection of X- and γ-rays is the Ar + 8 % CH_4 mixture. The additional upper beryllium window permits using the detector in the conventional backscattering geometry.

The detection chamber design of the combined detector assures fast, stable and high-quality measurements of Mössbauer spectra even with relatively weak radiation sources.

As an example, in the same figure is shown the result of simultaneous recording of Mössbauer spectra obtained on conversion electrons (a), specularly reflected radiation (b), X-rays (c) and scattered gamma radiation (d) from a thin metallic iron film at grazing incidence angle of 5 mrad. Main technical characteristics of Mössbauer spectrometer SM1101TER are represented in Table 1.

3. EXAMPLES OF APPLICATION MÖSSBAUER SPECTROMETER SM1101TER

The grazing incidence Mössbauer spectroscopy mode provides unique chances to investigate the spatial dynamics of chemical reactions starting at the sample surface, processes initiated by various influence on the surface (e. g. laser irradiation, gas annealing), processes of implantation, corrosion catalysis, etc.

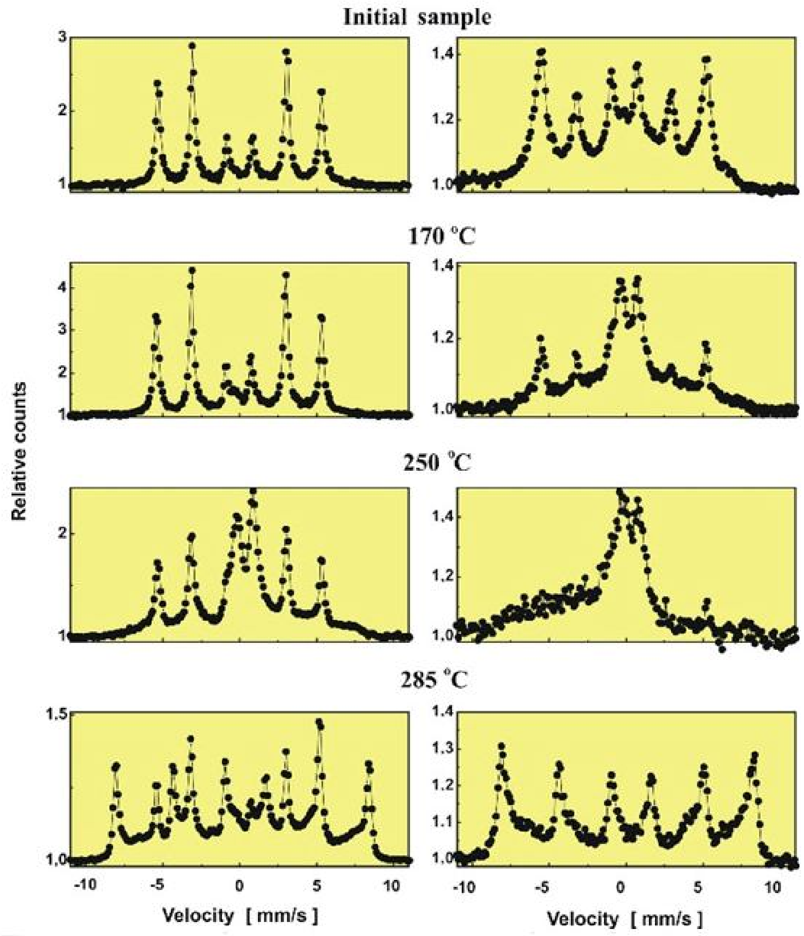


Fig. 3. The high surface sensitivity of the method

The high surface sensitivity of the method is illustrated in Fig. 3, showing two series of experimental spectra of a Armco steel film 20 nm thick subjected to treatment at different temperatures measured in the normal incidence ($\theta = 90^{o}$) geometry, when the spectrum is formed from the whole film (left) and at a grazing angle $\theta = 2.2$ mrad (right) when the radiation penetration depth does not exceed 2÷3 nm.

The spectra show a dramatic difference between the film subsurface layer parameters and parameters averaged over the entire film thickness, which are obtained by the backscattering Mössbauer spectroscopy method.

The Mossbauer spectrometer SM1101TER is used to study the corrosion of metals.

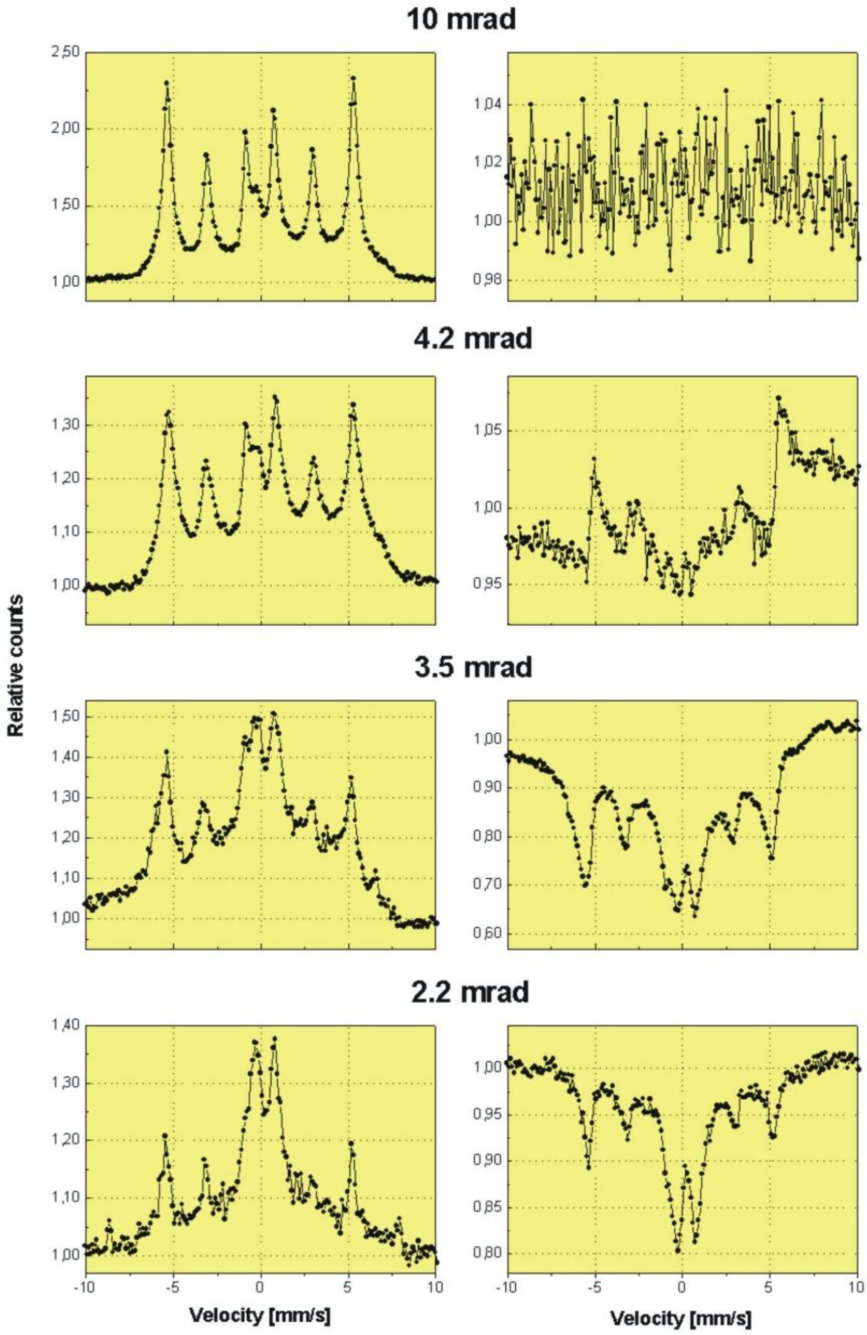


Fig. 4. Mössbauer spectra measured on conversion electrons (left) and specularly reflected radiation (right)

The Spectrometer possibilities can be demonstrated by the example of studying corrosion products in an ultrathin iron film 20 nm thick oxidized at various temperatures. Already at the early stages of corrosion a multiphase system is formed in the film. Mössbauer spectra measured at various grazing angles in the region of total external reflection angles can give information on the depth dependence of phase distribution.

Fig. 4 presents an example of Mössbauer spectra measured on conversion electrons (left) and specularly reflected radiation (right) of an iron film oxidized at 170°C for several grazing angles.

The interdepented processing of those spectra will help to recover the depth distribution profile of hyperfine interactions and hence of iron-containing phases to which the hyperfine interactions correspond.

Fig. 5 gives the depth distribution profiles of iron-containing phases of films oxidized at various temperatures.

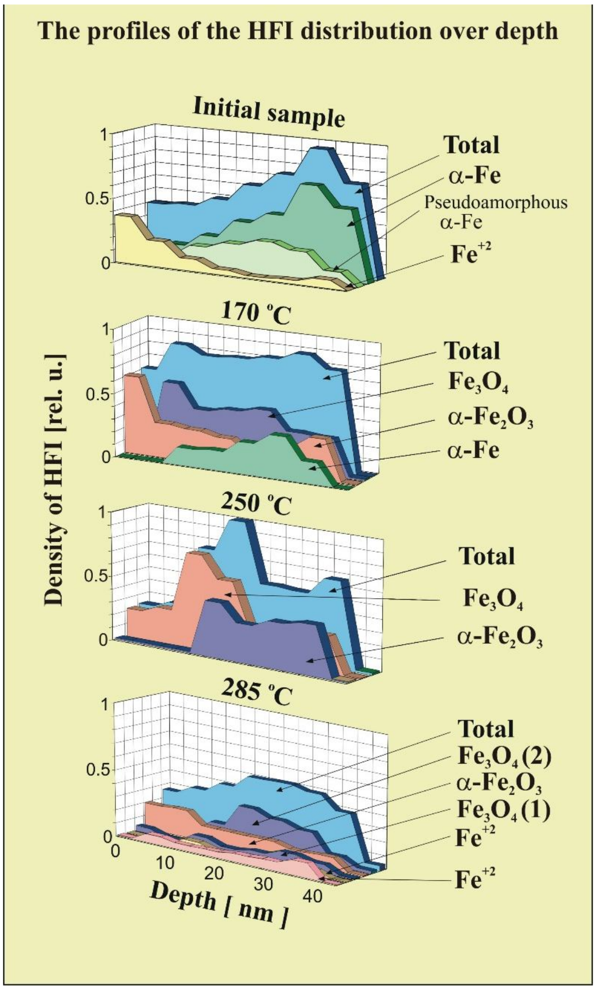


Fig. 5. The depth distribution profiles of iron-containing phases of films oxidized at various temperatures

The Mossbauer spectrometer SM1101TER is used to study the Spin Structure Studies.

It is known that the intensities of hyperfine interaction lines in Mössbauer spectra depend on the relative orientation of the quantization and incidence radiation axes.

Another example of Spectrometer application may be investigation of the spin texture, i.e., magnetic moment distribution relative to the sample surface in a Fe/V [3] artificial multilayer structure (Fig. 6).

The Mössbauer spectra for that structure were measured for different radiation orientations relative

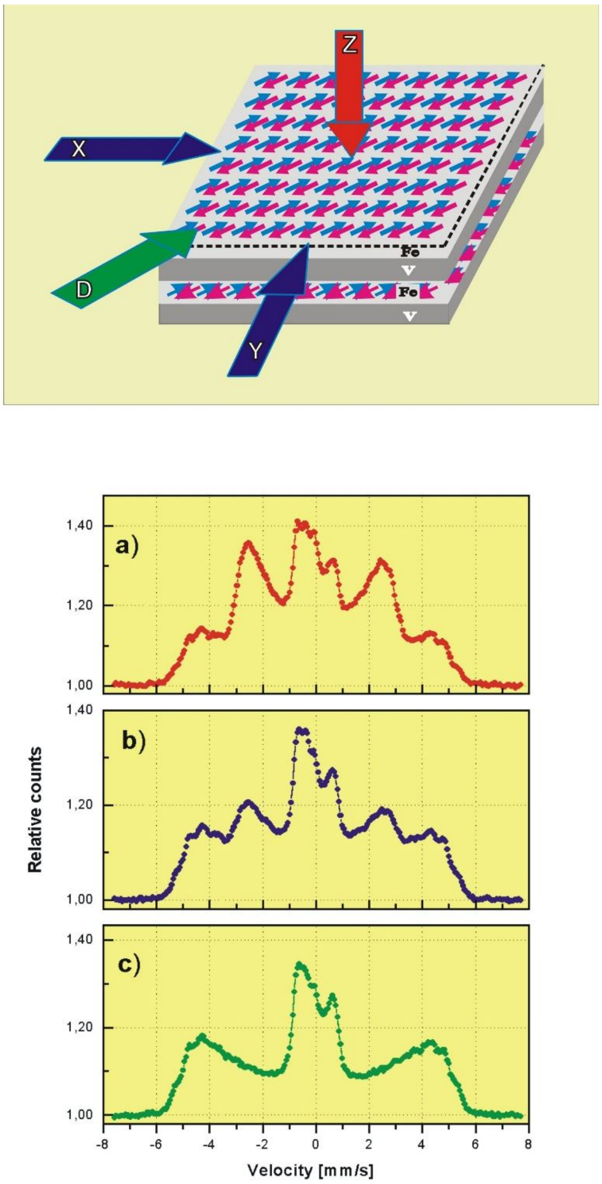


Fig. 6 Magnetic moment distribution relative to the sample surface in a Fe/V and artificial multilayer structure

to the sample surface. The measurements were performed in the backscattering geometry (a) and that of grazing incidence along the film sides (b) and diagonal (c).

The spectra show that in the diagonal direction, the second and fifth lines disappear, which points to the high magnetic anisotropy along that direction.

4. THE MOSSBAUER SPECTROMETER SM 4201 TERLAB

The SM 4201 TERLAB (Photo 2) automated multifunctional spectrometer (further referred to as Combined Spectrometer) is designed for depth selective nondestructive investigation of phase and element composition and physical and chemical state of condensed matter (crystalline and amorphous solids, artificial multilayer structures, nano-structures and nano-magnetics) [4–9].

The multifunctionality of Combined Spectrometer means that it combines features of Mössbauer and X-ray fluorescence spectroscopy, X-ray diffraction at normal and grazing incidence angles. The Combined Spectrometer also enables X-ray Reflectometry and X-ray or Mössbauer standing wave measurement.

The experimental data thus obtained will compliment information on the surface state from other analytical methods (mass spectrometry, ESCA, Auger spectroscopy) with the data on microphase, structural and magnetic states as well as surface roughness, density and thickness.

The Combined Spectrometer incorporates the latest achievement in the field of grazing incidence X-ray and Mössbauer optics of the surface, which suggest the combination of the total external reflection effect and various spectroscopic and diffractometry methods.

When some hard electromagnetic radiation I_o ($0.05\ \text{nm} \leq \lambda \leq 0.2\ \text{nm}$) is incident on a flat reflecting surface at an angle θ less than a certain critical value θ_{cr}, the total external reflection effect takes place. The depth d of the radiation penetration into a medium depends on the incidence angle of the primary radiation. By varying the incidence angle during experiments, one can carry on a depth selective investigation.

Interaction of the incident radiation with a medium leads to specularly reflected radiation, scattered gamma-rays, secondary X-rays, conversion electrons and Auger electrons (Fig. 7).

The optical scheme of the Combined Spectrometer is presented in Fig. 8.

A radiation beam from a Mössbauer source located

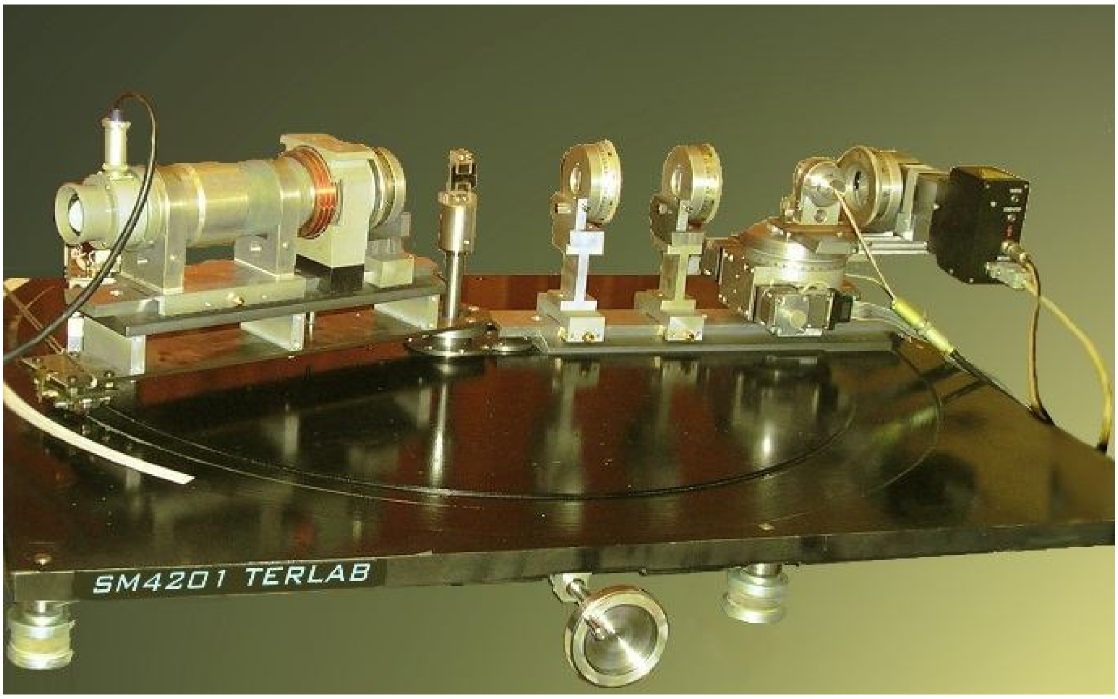


Photo 2. The SM 4201 TERLAB automated multifunctional spectrometer

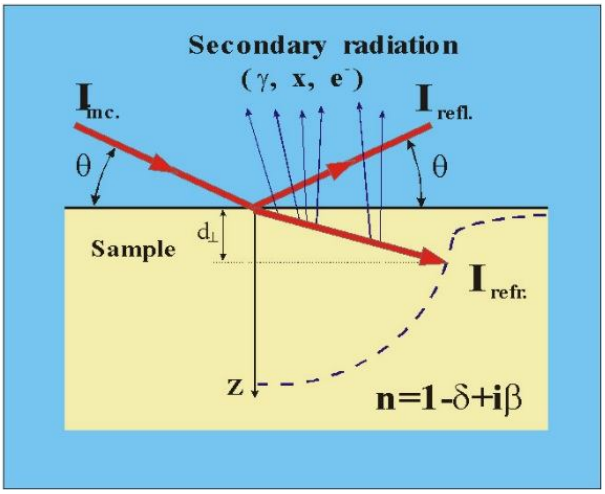


Fig. 7. Interaction of the incident radiation with a medium leads to specularly reflected radiation, scattered gamma-rays, secondary X-rays, conversion electrons and Auger electrons

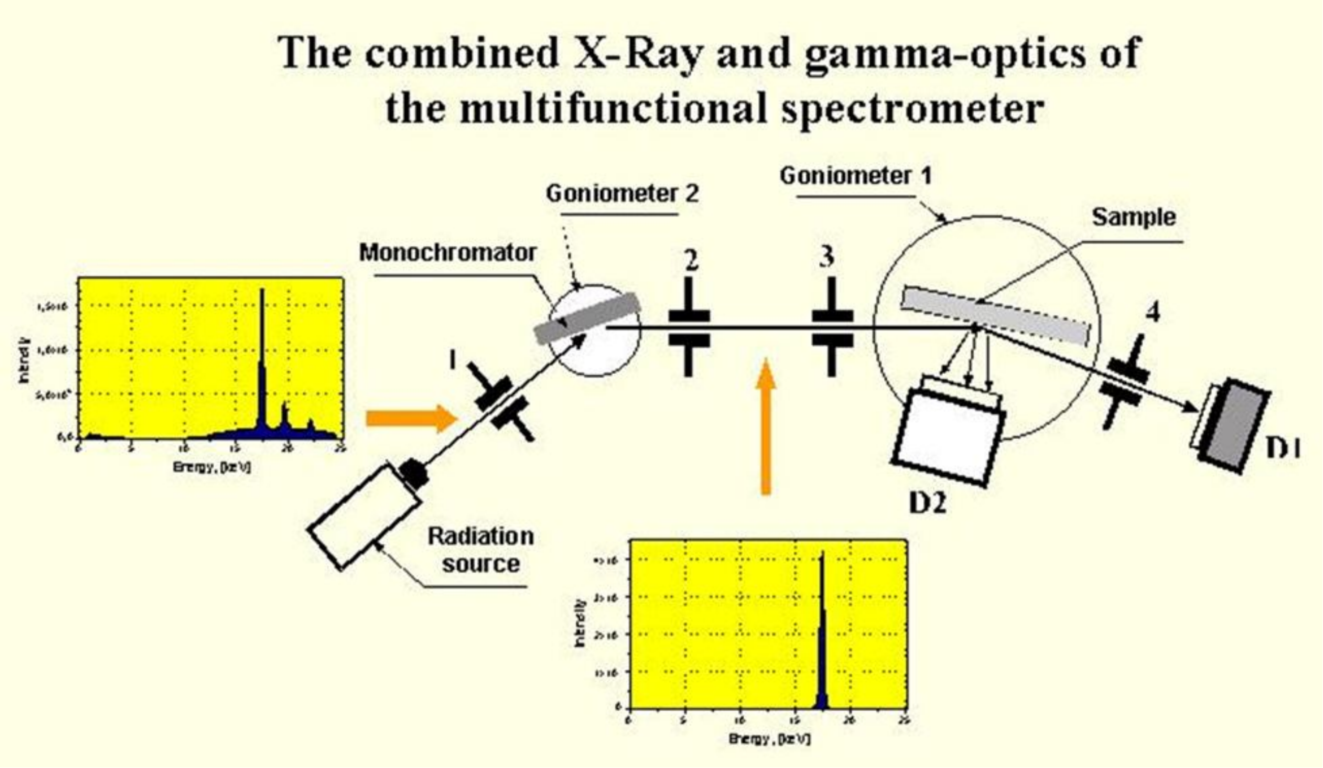


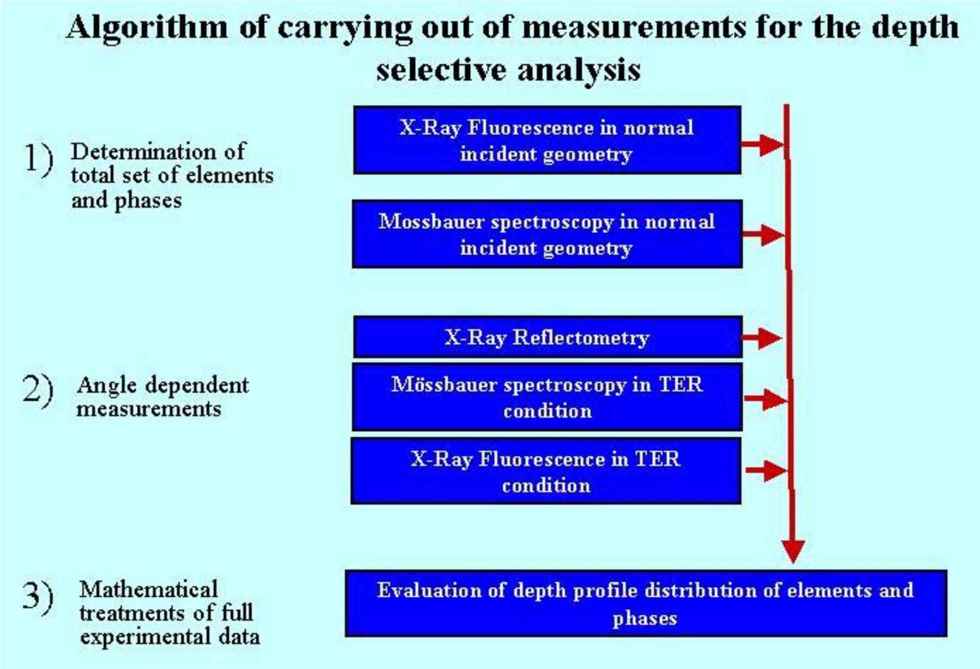
Fig. 8. The optical scheme of the Combined Spectrometer is presented

at Doppler modulator or X-ray tube mounted in its place is formed by slit collimator 1 and hits a monochromator M. The monochromator selects the required wavelength from a wide radiation spectrum. A narrow plane-parallel beam formed by slit collimators 2 and 3 falls on the sample under investigation.

Detector D1 records the radiation specularly reflected from the sample and detector D2 counts the respective secondary radiation.

Two-circle goniometer 1 allows high precision adjustment of grazing incidence angle, while goniometer 2 serves to set the monochromator to certain radiation energy.

For X-ray spectroscopy we used air-cooled tubes with a copper and molybdenum anodes of 50 W powers. For Mössbauer studies a radioactive ^{57}Co(Rh) source with an activity of 100 mC was used. The fluorescence spectra were recorded using Si-PIN diode,



Algorithm of carrying out of measurements for the depth selective analysis

1) Determination of total set of elements and phases
- X-Ray Fluorescence in normal incident geometry
- Mossbauer spectroscopy in normal incident geometry

2) Angle dependent measurements
- X-Ray Reflectometry
- Mössbauer spectroscopy in TER condition
- X-Ray Fluorescence in TER condition

3) Mathematical treatments of full experimental data
- Evaluation of depth profile distribution of elements and phases

Fig. 9. The final step consists in the interdependent experimental data processing to define the depth distribution of elements and phases

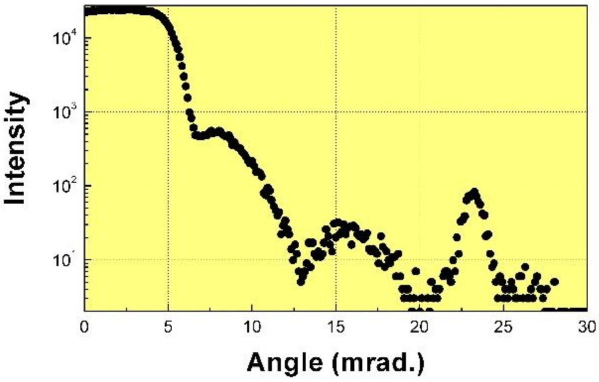


Fig. 10. A curve of specular reflection from artificial multilayer structure X-ray Fluorescence Spectroscopy Mode

while the specularly reflected Mössbauer radiation was detected by a scintillation detector with a NaJ(Tl) crystal 0.1 mm thick.

The Mossbauer spectrometer SM 4201 TERLAB can be used in physics, chemistry, geology, metallurgy, material science, quantum electronics, technology, biology, medicine, ecology, pharmacology, crime detection and emergency forensic medical examination.

Feasibility of using the Combined Spectrometer to determine element and phase compositions and their depth distribution is illustrated by the example of studying the artificial multilayer structure Zr (9.5 nm)/[Fe$_{0,65}$/Cr$_{0,35}$(1.4 nm)]•26/Cr (50 nm)/glass thin film.

The first step is measurement of X-ray fluorescence and Mössbauer spectra at normal incidence angles. This is necessary to determine the total number of elements and phases in the sample.

The second step involves measurement of the Reflectometry curve.

The third step is measurement of X-ray fluorescence and Mössbauer spectra in the range of total external reflection angles.

The final step consists in the interdependent experimental data processing to define the depth distribution of elements and phases (Fig. 9).

The X-ray Reflectometry mode is used to define the electronic susceptibilities component of thin surface layers, surface roughness and layer thickness of the multilayer structure. Fig. 10 presents a curve of specular reflection from artificial multilayer structure Zr (9.5nm)/[Fe$_{0,65}$/Cr$_{0,35}$ (1.4 nm)]•26/Cr (50nm) /glass measured in the 0–30 mrad angle range on the CuK$_{\alpha}$ radiation.

To determine depth profile of elements composition, a series of X-ray fluorescence spectra was measured for various incidence angles within the total external reflection angles range using MoK$_{\alpha}$ radiation (Fig. 11).

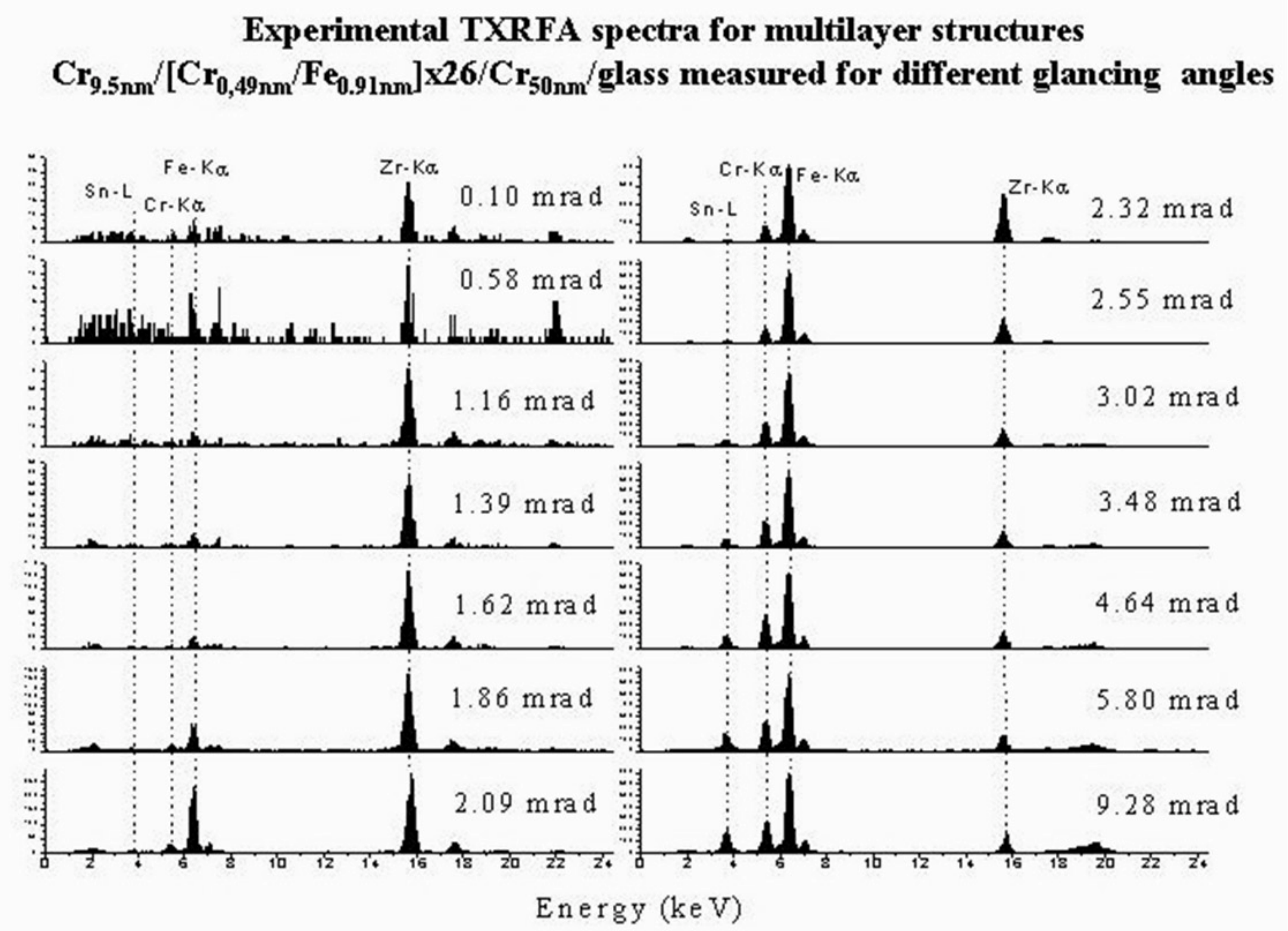


Fig. 11. To determine depth profile of elements composition, a series of X-ray fluorescence spectra was measured

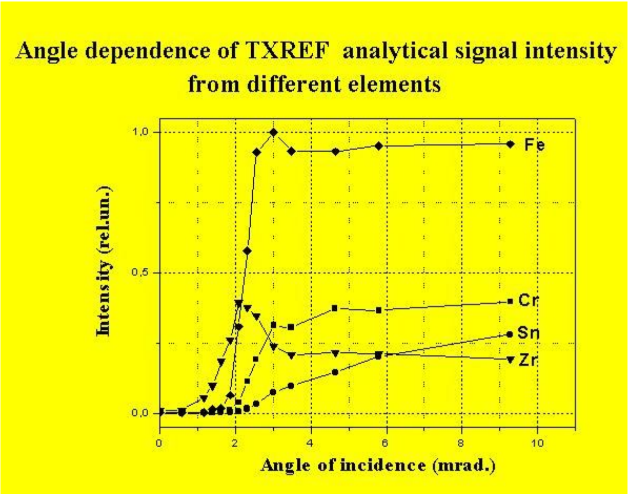


Fig. 12. The layer depth and relative content of elements in the layer

The clearly shows the spectral line of zirconium present at the surface, already at total external reflection angles. As the incidence angle increases, the radiation penetrates beyond the Zr layer and contributions from iron and chromium atoms to spectrum begin to grow, while the relative intensity of Zr line reduces.

Mathematical processing of spectra can restore the depth profile of an element composition. The layer depth and relative content of elements in the layer are indicated in the Fig. 12.

The phase state of iron atoms in the multilayer structure can be determined by Mössbauer spectroscopy (Fig. 13). The Mössbauer spectrum measured in the 90-degree geometry shows that iron is in a magnetically ordered state which correspond to single-phase α-Fe. However, we see that the spectral lines are broadened. This is because part of iron atoms are located in transition layers and their environment differs from that of iron in the single-phase state.

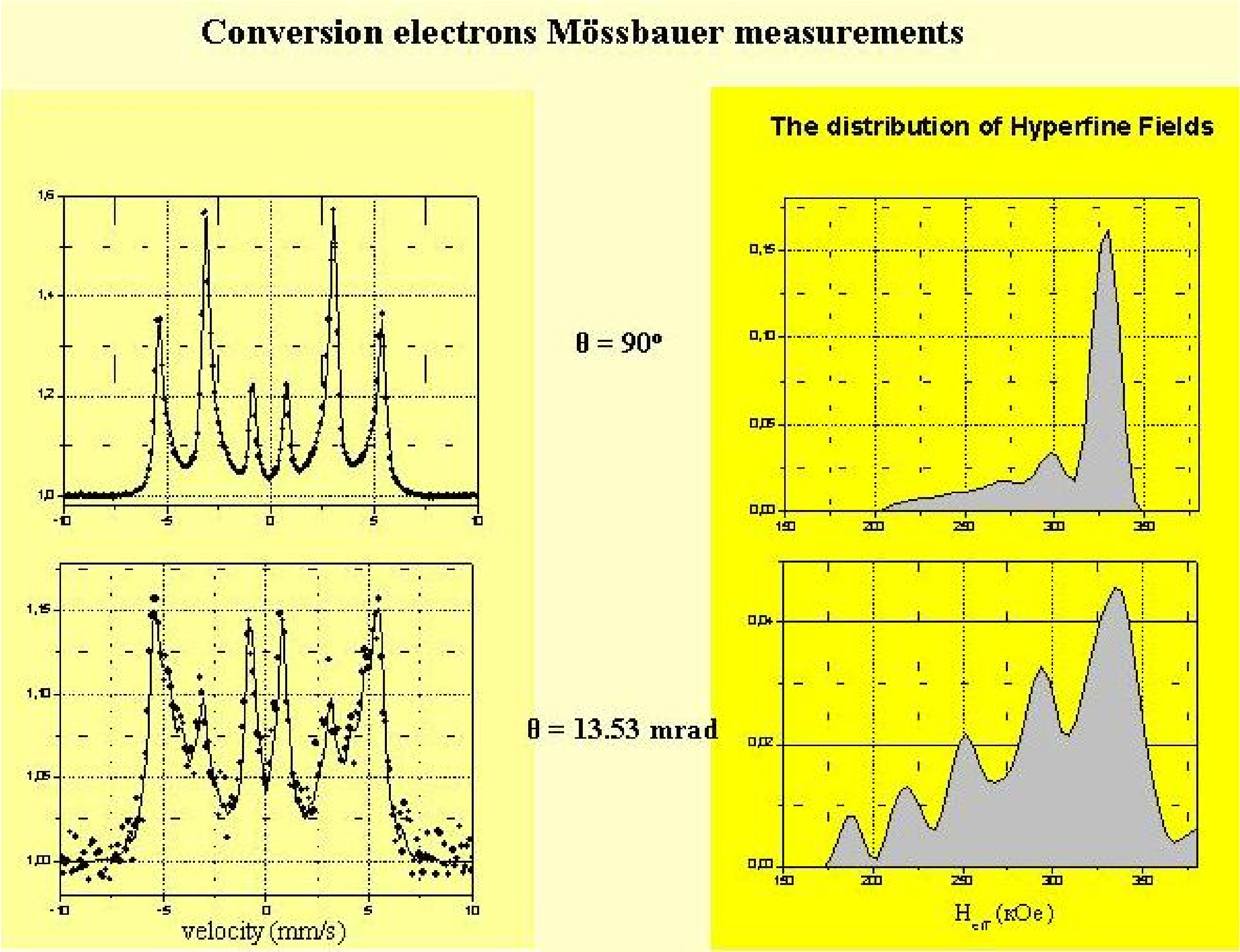


Fig. 13. The phase state of iron atoms in the multilayer structure can be determined by Mössbauer spectroscopy

The processing of the spectrum gave us the distribution function of hyperfine fields. The area under the intense peak corresponds to the iron phase present inside the layer, while the remaining peaks represent the iron in the interface region. It makes up about 40 %.

The same figure shows a Mössbauer spectrum measured in the Bragg maximum region. The spectrum quite clearly demonstrates increase in the signal from the interface region. This phenomenon is caused by the standing wave effect.

The standing wave is formed due to the interference of the incident and reflected radiations. In our case the wave field maximum corresponds to the interface region and hence its secondary radiation exhibits maximum intensity as compared to the layer itself. So the method sensitivity to the interfacial regions was experimentally increased, which is very important for analysis of multilayer structures. The same spectrum was used to obtain experimentally the distribution function of the hyperfine fields, which was allowed us to assess the composition of the interface region.

5. MÖSSBAUER SPECTROMETERS SM 1101TER AND SM 4201 TERLAB, PRODUCED BY THE IAI RAS IN THE MARKET OF SCIENTIFIC INSTRUMENTATION

Mössbauer spectrometer SM 1101TER and SM 4201 TERLAB, serially produced IAI RAS, compete successfully in the domestic and foreign markets of scientific instrument making with manufacturers Mössbauer spectrometers: Regional Centre of Advanced Technologies and Materials, Olomouc, Czech Republic [10] and Wissenschaftliche Elektronik GmbH (WissEl), Starnberg, Germany [11].

Russian Mössbauer spectrometers and SM

SM 1101TER TERLAB 4201 have been applied in research institutions, institutions of higher education and institutions of the Russian Academy of Sciences. Mössbauer spectrometer SM 1101TER and SM 4201 TERLAB are been exploited by the organizations working in the field of new construction materials and investigating the properties of innovative materials used in biomedicine, mining industry, the analysis of mineral raw materials and processed products.

Spectrometers SM 1101TER and SM 4201 TERLAB apply to the study of the magnetic and physical-chemical properties of innovative materials, high-temperature superconductivity research materials, ferroelectrics, multiferroics.

IAI RAS carries out the creation of scientific instruments that serve as import substitution in the market of scientific instrument making.

FANO Russia, defending the interests of subordinate institutions of science, implements one of the types of government economic strategy of import substitution, in relation to the industrial policy of the state aimed at protecting domestic producers by substituting imports of scientific instruments and scientific technologies of national production devices.

Implementation by FANO Russia of the import policy is conducted to increase the competitiveness of domestic products by promoting the modernization of production, increase of its efficiency and the development of high-tech, knowledge-intensive products with a relatively high value-added [12].

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РАЗРАБОТКА ПРИБОРОВ И СИСТЕМ

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ИННОВАЦИОННОЕ НАПРАВЛЕНИЕ НАУЧНОГО ПРИБОРОСТРОЕНИЯ — МЁССБАУЭРОВСКАЯ СПЕКТРОСКОПИЯ КАК ФАКТОР СОВЕРШЕНСТВОВАНИЯ ОТРАСЛЕЙ РОССИЙСКОЙ ЭКОНОМИКИ.

Ч. 2. СОЗДАНИЕ НАЦИОНАЛЬНОГО ИССЛЕДОВАТЕЛЬСКОГО ОБОРУДОВАНИЯ В ОБЛАСТИ МЁССБАУЭРОВСКОЙ СПЕКТРОСКОПИИ

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Представлены основные технические характеристики созданного ИАП РАН национального исследовательского оборудования в области мёссбауэровской спектроскопии. Представлены серийно производимые ИАП РАН мёссбауэровские спектрометры SM 1101TER и SM 4201 TERLAB, которые широко используются при разработке инновационных материалов. Мёссбауэровские спектрометры SM 1101TER и SM 4201 TERLAB оказались востребованными при исследовании магнитных и физико-химических свойств инновационных материалов, исследовании высокотемпературной сверхпроводимости соединений, исследований, связанных с мультиферроикой, сегнетоэлектрикой.

Кл. сл.: инновации, импортозамещение, мёссбауэровский спектрометр, гамма-резонансный спектр, доплеровская модуляция энергии, гамма-оптическая схема

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