MOSSBAUER SPECTROSCOPY OF LOCALLY INHOMOGENEOUS SYSTEMS

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Introduction

Substances with characteristic local inhomogeneities – with different from position to position neighborhood and properties of like atoms – gain recently increased scientific attention and wide practical application. We would call a system locally inhomogeneous if atoms in the system are in non-equivalent atomic locations and reveal different properties [1,2]. Such systems are, first of all, variable composition phases, amorphous, multi-phase, admixture, defect and other systems. LIS are most convenient model objects for studies of structure, charge, and spin atomic states, interatomic interactions, relations between matter properties and its local characteristics as well as for studies of diffusion kinetics, phase formation, crystallization and atomic ordering; all that explains considerable scientific interest in such LIS. Such systems find their practical application due to wide spectrum of useful, and sometimes unique, properties that can be controlled varying character and degree of local inhomogeneity.

Mössbauer spectroscopy is one of the most effective methods for investigation of LIS. Local character of obtained information combined with information on cooperative phenomena makes it possible to run investigations impossible for other methods. Mössbauer spectroscopy may provide with abundant information on peculiarities of macro- and microscopic state of matter including that for materials without regular structure. At the same time, analysis, processing and interpretation of Mössbauer spectra for LIS (that are sets of a large amount of partial spectra) face considerable difficulties. Development of computer technique is accompanied with development of mathematical methods used for obtaining physical information from experimental data. The methods make it possible to improve considerably, with some available a priori information, effectiveness of the research. Utilization of up-to-date mathematical methods in Mössbauer spectroscopy requires not only adaptation of these methods for specific physical tasks and their software realization, but development of the methods for application and physical interpretation of obtained results.

Present paper considers ways for obtaining information from Mössbauer LIS spectra. Application of LIS Mössbauer spectra processing and analysis methods are discussed. Ways for complex utilization of various methods are described along with the role of a priori information at all processing stages.

Locally inhomogeneous systems in Mössbauer spectroscopy

The entire notion *local inhomogeneity* directly relates to the state and, therefore, properties, of an atom at some location; these properties are first of all defined by the neighborhood and its characteristics. Characteristics of atomic neighborhood can be split up into the following categories (see Fig.1):

- *topological* characteristics (characteristics of spatial arrangement of neighboring atoms): elements with point (local) symmetry, spacings and angles between the atoms, coordination numbers, radii of coordination spheres, catenation angles of polyhedrons, etc.;
- *composition (chemical)* characteristics of the neighborhood: amount and arrangement of different type neighbor atoms;
- *physical* characteristics of the neighborhood (i.e. parameters that characterize state and interaction of neighboring atoms): electronic configuration, spin, orbital and magnetic moments, etc. External conditions such as temperature, pressure, field gradients and strengths, may influence considerably on both characteristics of atomic neighborhood and directly on physical characteristics of the atom at the given location (see Fig.1). Characteristics of the atomic neighborhood entirely define equivalence or non-equivalence of the atomic locations. So, local non-uniformity in atomic properties can be classified based on its origin as *topological, composition (chemical), physical* and *combined* ones [2].

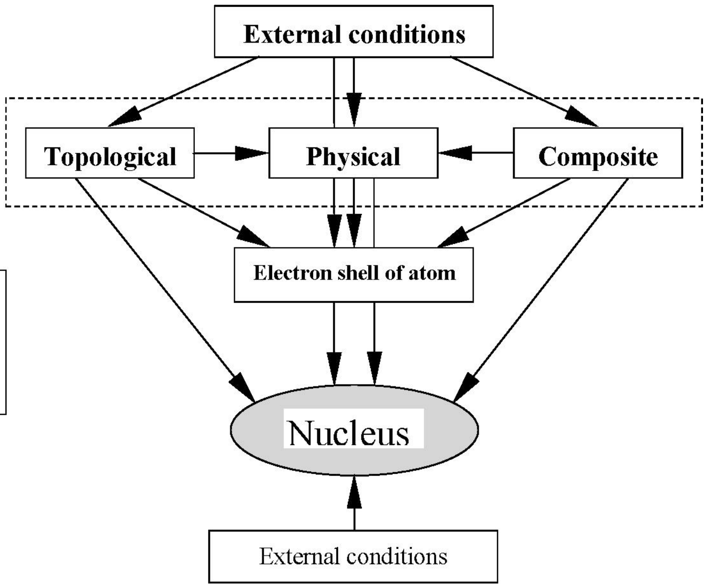

Fig1. The main factors determining local inhomogeneity of hyperfine interactions.

Since characteristics of the neighborhood and physical properties of an atom in a given position define hyperfine interactions (HI) of its nucleus (fig. 1), then using HI one can investigate local inhomogeneity of atomic properties and one can speak in terms of *local inhomogeneity of HI* (LIHI). In this case classification of LIHI remains the same as for local inhomogeneity of atomic properties.

In order to describe LIHI it is convenient to introduce the following notions: *type* and *degree* of LIHI [2], which enable us to compare different HIs. LIHI type is defined by the type of HI (electric monopole, magnetic dipole, and electric quadrupole interactions along with any combination of them) for which energy is considerably different from position to position compared to natural resonance line width. As a rule, there is realized a combined type of LIHI with considerable simultaneous change of several different types of HI. At that their change may be both correlated (due to the same physical factors) and non-correlated (due to different physical factors). Each type of LIHI is associated with its HI parameter (hyperfine parameter of Moss Bauer spectrum): electric monopole – shift δ of Moss Bauer line, electric quadrupole constant of quadrupole interaction e^2qQ and asymmetry parameter η (or quadrupole shift ε of the spectrum components), magnetic dipole – hyperfine magnetic field $\vec{H}_n$ in the region of nucleus. Shift of the Moss Bauer line δ is due to the isomer (chemical) δ_I and temperature δ_T shifts. The temperature shift δ_T is defined by vibration spectrum of the nuclei and the isomer shift δ_I – by the charge density of electrons in the nucleus' vicinity. At that electronic density on a nucleus is directly related to electronic configuration of an atom and to electronic structure of the system, including the character of the atoms' chemical bonds. Shift of the line δ is mainly defined by the direct neighbors of the atom. Influence from the atoms in higher-order coordination spheres is delivered, as a rule, through location and electronic state of the atoms in the first coordination sphere (see Table 1).

Table 1. Sensitivity of hyperfine parameters.

Hyperfine parameters		δ	ε	H_n
Number of coordination spheres		1÷2	1÷10	1÷5
Distance, Å		2÷4	2÷20	2÷10
The characteristics of an atom environment	Topological	+/–	+	+/–
	Composite	+/–	+	+
	Physical	–	+/–	+

"+" – high, "+/-" – appreciable and "-" - low sensitivity.

Interrelation of the isomer shift δ_I and electron charge density in vicinity of a nucleus stipulates notable sensitivity of the δ line shift to topological and composition local in homogeneities (Table 1). In case of LIS there is change in spacings from the atoms in the first coordination sphere of replacement of some atoms in this sphere by other-type atoms from position to position. For instance, ^{57}Fe nuclei may reveal the following shift change with the change of distances in different systems: $\frac{\partial\delta}{\partial \ln r} \cong 1 | 5$ mm/s, and with replacement of one atom - ~ 0.01 mm/s. Shift of the Moss Bauer line δ makes it possible to identify valence state of atoms, obtain information regarding both phase composition and peculiarities of electronic and crystalline structures of LIS under investigation.

Quadrupole shift ε (splitting $\Delta=2\varepsilon$) of the spectral hyperfine structure components takes place in non-uniform electric field that partially eliminates degeneration of the nuclear level on the magnetic quantum number. In general case for solids the electric field gradient in vicinity of a nucleus is made by localized charges of ion bases of the neighboring atoms (contribution from the lattice), polarized conduction electrons and valence electrons of the Mössbauer atom. This means that the quadrupole shift ε is sensitive, first of all, to topological and composition local in homogeneities (Table 1).

Assessment of the change in ε due to lattice contributions only shows for ^{57}Fe that sensitivity region in case of quadrupole electric interaction is much wider than for the case of monopole electric interaction. This region has ~10 coordination spheres and can expand to the radius of up to 20 Å (Table 1). In particular, appearance of an +3 ion at ~10Å results in change of quadrupole shift for ~0.006 mm/s what is quite enough for revealing using modern methods for processing and analysis of Mössbauer spectra. If such ion happens to appear in the first coordination sphere, it results in huge ~0.8 mm/s change in quadrupole shift that exceeds its natural line width for 8 times. Quadrupole shift ε describes peculiarities of electronic and crystalline structures of LIS and may provide with useful information both on symmetry of the closest vicinity of Mössbauer atoms and about their electronic configuration.

At investigations of magnetic-ordered systems, the most sensitive to local inhomogeneity among the Moss Bauer spectra parameters is effective magnetic field H_n. This field is generated by both an atom and its neighbors. Effective field H_n is particularly sensitive to composition and physical (magnetic) local in homogeneities (Table 1) [1]. It is known that replacement of one magnetic atom with another one in the close vicinity may change the field at the ^{57}Fe nuclei for 20 – 30 kOe mainly due to change in Fermi contribution H_F. Assessment of dipole-dipole contribution H_{dip} into H_n shows that sensitivity region may cover ~5 coordination spheres and expand in this case up to 10 Å in radius (table 1). In particular, an atom with magnetic moment $5\mu_B$ produces the field of H_{dip}~0.09kOe at distance 10Å what is quite enough for registration of the change in H_n, and the same atom produces the field of ~12kOe at 2Å. Data on effective magnetic field H_n may provide information on local peculiarities of atomic, magnetic and electrical structures of investigated magnetic-ordered LIS.

The degree of LIHI is defined by the change in HI energy from position to position of a Mössbauer atom compared to the component (resonance line) width of the Moss Bauer spectrum. At LIHI there are observed well-defined partial spectra or considerable widening of the components in the spectrum. Difference in hyperfine parameters of these partial spectra or degree of the component widening defines the degree of LI for corresponding HIs. In case when the spectrum is described by hyperfine interaction distribution function the degree of LIHI is defined by the distribution function parameters such as integral width, spacing between the modes and width of each of the mode. Changes in the type and degree of LIHI may be observed in such processes as crystallization and atomic ordering, phase formation and phase transitions (structural, magnetic and ferroelectric ones), implantation and sputtering, laser and thermal annealings, doping, etc.

Presence of a considerable amount of somehow non-equivalent positions in Mössbauer atoms (nuclei) in the investigated sample is typical for LH. Spectra of such samples in form of a superposition from many partial spectra may provide with various useful information on phase composition and peculiarities of crystalline, electronic and magnetic structures for each phase. There are, of course, some difficulties when extract this information. Complex structure of the spectra requires special spectrum analysis and processing methods involving modern mathematics and software.

At that, choice of information extraction method becomes of importance. To considerable extent this choice is defined by the particular task based on available a priori information about the sample or other considerations.

Classification of Moss Bauer spectra analysis and processing methods

Application of Moss Bauer spectroscopy for investigations of LIS requires solving of a set of tasks at analysis and processing of the Moss Bauer spectrum. Content of the tasks depends both on a specific object under investigation and on a priori information the investigator has [2]. In its turn, each of these tasks implies that there are specific methods available for processing of the Mössbauer spectra and that the methods meet the specific requirements of this task.

Let us consider specific tasks related to utilization of Moss Bauer methods in investigations of LIS and correspondingly to them classify the Moss Bauer spectra analysis and processing methods. We provide this classification with examples of the methods realized by us within the software package MSTools [2] designed for effective applications in Moss Bauer spectroscopy LIS (see Table 2.).

Table 2. Methods for analysis and processing of Mössbauer spectra realized within the software complex MSTools.

Analysis and processing method of Moss Bauer spectra	A priori information			MSTools
	S	A	ET	
Resolution enhancement and noise cancellation	+	-	-	RESOL, DISTRI
Model fitting	+	+	-	SPECTR
Distribution functions restoration	+	+	-	DISTRI
Comparative analysis with standard or modeling spectra	+	-	+	PHASAN
Simulation	-	-	-	RESOL, SPECTR, DISTRI, PHASAN

In some cases of Moss Bauer investigations of LIS it is necessary to improve spectrum quality by higher resolution or effective noise suppression. As a rule, such necessity arises with lack of a priori information about the object. Statistical noise in experimental spectrum of limited line width of the radiation source Γ_s may somehow “hide” peculiarities of the spectrum with desired information. The result of solving such task is a new, transformed spectrum with considerably increased resolution or effectively suppressed noise (Table 2).

Solving such task within the software package MSTools we realized linear methods for processing of Mössbauer spectra – filtration, regularization and “discrepancy” [2, 3]. A characteristic feature of these methods is absence of any valuable a priori information AI(A) about the object under investigation. But effectiveness and reliability of the processing depend greatly on reliability and completeness of a priori information AI(S) about the radiation source and peculiarities of the spectrometer hardware performance.

This processing method has been realized in a code RESOL [2, 3].

Spectrum quality may be improved with recovery of distribution function p(v) for the single resonance line shift (DISTRI code). In this case recovery of the function p(v) can be interpreted as a transformed spectrum of better quality (for more details see [2]).

Another task related to investigation of LIS relates to model fitting of spectra (table 2). This implies search for or specification of values for comparatively narrow set of physical parameters that, within the chosen model, uniquely describe state of Moss Bauer nuclei in a solid and, correspondingly, the Mössbauer spectrum. When applied to LIS such task statement is reasonable when two conditions below are met:

1. a researcher possesses (or thinks that possesses) complete a priori information about both the spectrometer AI(S) and the object AI(A), first of all, about its phase composition, atomic distribution, point symmetry, chemical bounds and valence;
2. number of independent parameters that describe spectrum within the chosen model is not too high (i.e. much less than the number of points in the experimental spectrum).

Such situation may take place if the substance under investigation is of regular crystalline and magnetic structure and the sample is homogeneous in its composition. The processing method is based on the least squares method to get the best fit for description of the experimental curve and superposition of so-called non-rigid bounds; it is also based on superposition of partial spectra valid for quite “fine” samples. The method has been realized in the SPECTR code [2] and designed for obtaining of qualitative information on hyperfine interaction parameters for Mössbauer nuclei.

Experimenters quite frequently deal with LIS of amorphous substances, variable composition phases of doped systems. Description of the system state with some discrete set of physical parameters’ values is difficult then as well as of Mössbauer nuclei in this system. When process Moss Bauer spectra of such systems it is necessary to recover distribution functions of the spectrum parameters. This problem has been solved in the code DISTRI using regularization method in its iterations variant [1, 2]. This variant of the method makes it possible to put physically grounded conditions both onto the values of recovered distribution functions and other variable using a priori information about the spectrometer AI(S) and the object under investigation AI(A). As a result there are obtained distribution functions for partial spectra parameters with evaluation of statistical errors and characteristics of these functions.

The next considered task implies comparison of Moss Bauer spectrum obtained for a sample with spectra of standard samples. This task is of importance at analysis of multiphase systems when investigator has no information on each separate phase and it is necessary to make qualitative or quantitative phase analysis only. Almost no a priori information on a spectrometer AI(S) and a sample AI(A) is required in this case, but one needs spectra of standard samples with low noise levels, i.e. high-quality spectra. If the spectra of investigated sample and standard samples have been obtained at different spectrometers, their calibration and some experimental conditions are to be known. The core of the method is in searching for optimal combination of the standard samples spectra for description of the spectrum for the sample of interest; the method is based on application of the least squares method and superposition principle. Upon processing of the spectrum it is possible to obtain both weight (absolute and relative) and isotopic (absolute) content of the standards in the sample of interest. Within MSTools software the method is realized in the code PHASAN [2] and is designed for qualitative and quantitative spectra analyses independently on availability of information on their structure. This method of spectra analysis can also be used for revealing of slight changes in a spectrum at any directed impact on the sample such as heating, implantation, laser annealing, ageing, composition change and so on.

The last but still very important task is simulation of experimental Moss Bauer spectrum. It is possible to simulate experimental Mössbauer spectra with any of the codes SPECTR, DISTRI or PHASAN, but only within the framework of the models realized by these codes. As we can see, the considered methods realized within MSTools software package cover almost all possible types of analysis and processing of Moss Bauer spectra excepting only the case with relaxation effects. Each of these methods is most effective when dealing with a specific task it has been designed for, but they can also be used for other tasks adding and “helping’ each other.

Complex approach to processing and analysis of spectra

As a rule, at Mössbauer study of a specified LIS sample not one, but several from considered above tasks for processing of spectra emerge. First, a specific case rarely makes it possible to assign the object to a specific substance class. For instance, LIS under investigation may have regular crystalline and non-regular magnetic structures at the same time. Or, in some cases, variable composition phases may reveal their properties (with corresponding change in the Mossbauer spectra) as a small, but still existing complex of regular structures. In all these cases it is reasonable to set both tasks of model fitting and recovery of the distribution function for spectrum parameters. Second, peculiarities of the considered spectra processing methods make it possible to use these methods solving other tasks they were not designated to.

For example, in order to obtain weight and isotopic content of some phases in LIS sample one can use model fitting of the spectrum. One can also try to describe a Mössbauer spectrum, for instance, of a multi-phase sample with regular phase structures by restoring distribution functions for partial spectra parameters. Or vice versa, spectrum of a sample with non-uniform structure can be described with a large amount of partial spectra using model fitting.

Third, purposeful consequent utilization of various methods may be an advantage. Such approach is particularly effective when almost no a priori information is available about a LIS sample and its Mössbauer spectrum revealed complex multi-component structure. The need in simultaneous solution of several tasks related to analysis and processing of Moss Bauer spectra calls for complex utilization of the methods. Processing of a spectrum with several methods independently from one side adds up obtained information and, from the other side, improves reliability of the conclusions made.

As one can see, different methods for analysis and processing of Mössbauer spectra leave the possibility for their close interaction while application of a specific method, its effectiveness depends considerably on the results of application of other methods. Such interaction can be realized using information provided by one of the methods as a priori information for another one. Moreover, utilization of any of the methods is impossible without utilization a priori information in some form and to certain extent.

Role of a priori information

As early as at the stage of task formulation and choice of spectrum processing method, availability of a priori information, its character and completeness play crucial role. Really, information on crystalline and magnetic structures of a sample, short or long range ordering there, on isomorphic replacement of atoms, on admixtures in the sample may set up a task either of model fitting of the spectra, or of reconstruction of the parameter distribution functions. Even not high-quality and very reliable information on sample composition at real possibility to obtain standard spectra frequently explains the choice to compare the spectrum with spectra of such standard samples. Unavailability of a priori information about the object under investigation or its penury (even when this is accompanied by poor quality of experimental spectrum) forces to improve first the spectrum quality in order to obtain additional a priori information.

The same important role a priori information plays at direct application of the method for processing within a specific task. Reliable a priori information eliminates in some cases ambiguity of the processing result and improves its reliability.

In order to improve spectrum quality the information on real source and spectrometer AI (S) used is of importance: form and width of radiation line, spectrometer performance quality (drifts in its electronic units, precision of continuous form of change in Doppler velocity v), about geometry of the experiment (angular aperture of the registered beam of γ-quanta and relative amplitude of the source oscillations).

In model fitting of spectra, utilization of a priori information on the investigation object AI(A) makes it possible to reduce dramatically the number of physically reasonable models within which the physical values of the parameters that describe the spectrum; these parameters help not only to identify the revealed phases or non-equivalent positions of atoms, but obtain new physical information. In such case very diverse information both qualitative (for instance, about magnetic structure colinearity, about the mechanism of exchange interactions, or presence of texture) and quantitative (number of non-equivalent positions, characteristic values of spectrum hyperfine parameters, specific cation distribution, orientation of easy direction, electronic configuration, etc.) can be used.

At reconstruction of distribution functions p(z) of one of the spectrum parameters z, besides information on the source and the spectrometer AI(S), there is of importance any information about object under investigation that enables making grounded guesses regarding the “behavior” of other spectrum parameters and, in particular, about their possible correlation with z. This can be information on the mechanism of hyperfine interactions, chemical bonds or, for instance, presence of spontaneous striction, about interrelation of cation distribution with the symmetry of closest vicinity of the Mössbauer nucleon, and so on. Information on standard samples AI(ET), their possible presence in the investigated sample, about the spectrometer where the sample spectra were obtained comprise the a priori information required for effective utilization of the method that compares spectrum of a sample under investigation with spectra of standard samples.

Peculiarities of the methods for analysis and processing of Moss Bauer spectra assure in case of LIS utilization of not only each method independently, but also their combination.

Role of a priori information becomes crucial at that. This interrelation is based on a quite obvious fact that new information obtained by any of the methods may be to certain extent used both for formulation of another specific task for analysis and processing, and for solving the same task with another method.
The sort of information that can bind different methods for analysis and processing of Mössbauer spectra is presented in a flow-chart at Fig. 2.

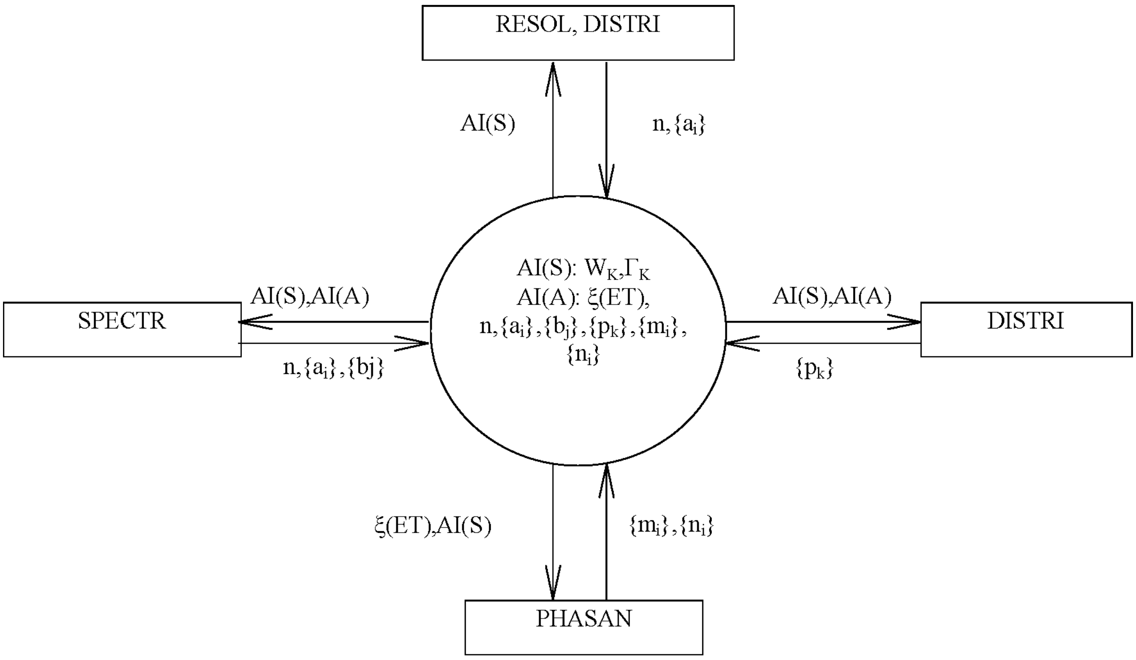

Fig 2. About the role of a priori information in interrelation of various methods for processing and analysis of Mössbauer spectra.

So, in case of spectrum quality improvement (codes RESOL and DISTRI) those are obtained at analysis of the transformed spectrum ξ', supposed number of components n in the original spectrum and initial values of the parameters $\{a_i\}$ that directly describe the Mössbauer spectrum: amplitudes $\{A_i\}$, velocities $\{v_i\}$, and widths $\{\Gamma_i\}$ of the spectrum components. When complete the task of model fitting (code SPECTR) those are optimal within the chosen model number of components n, values of the parameters $\{a_i\}$, and physical values $\{b_j\}$. At reconstruction of distribution functions for spectrum parameters (code DISTRI) those are obtained distribution parameters for nuclei presented at some discrete set of distribution function $\{p_k\}$ and their characteristics. At comparison with spectra of standard samples (code PHASAN) those are optimal values for weight $\{m_i\}$ and isotopic $\{n_i\}$ content of standard phases in the sample under investigation.

All this new information can be to certain extent used at consequent stages as a priori information about an object under investigation AI(A): either at choice of a model for spectrum fitting, or at establishing of conditions for reconstruction of the parameters' distribution function, or at choice of spectra obtained for standard samples. Therefore, complex approach to processing of Mössbauer spectra is an interrelated consequent application of various methods with specific combination of these methods defined, first of all, by available a priory information before and after application of each of the individual methods.

Interrelation of various analysis and processing methods

Let us consider in more details the interrelation of various methods for analysis and processing of Mössbauer spectra at investigations of LIS (see fig. 3).

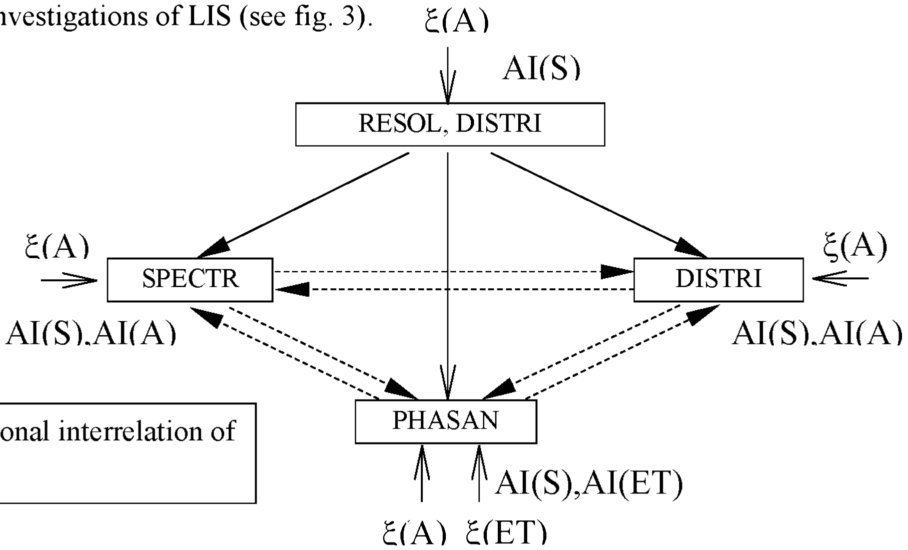

Fig 3. Possible functional interrelation of various methods.

A method of spectrum quality improvement (codes RESOL and DISTRI) takes a particular place among the methods for analysis and processing. In this method, different to all other, no a priori information about an object under investigation AI(A) is used. Moreover, this method has been designed for obtaining of such a priori information by increasing resolution in a spectrum or by effective noise suppression. The entire quality improvement may result in completion of the LIS investigation. However, in reality the results of the method are very frequently used in application of other methods at consequent processing stages. Analysis of the transformed spectrum (or distribution functions p(v)) makes it possible to define with some reliability the number of lines in the spectrum n and reveal the number of partial spectra (i.e. number of supposed phases in the sample or of entirely non-equivalent positions of Mössbauer atoms). Results of such analyses can be used for creation of a specific model for consequent model fitting or reconstruction of distribution functions for the spectrum parameters.

As a rule, revealed values n and $\{a_i\}$ together with a priori information AI(A) obtained from other experimental methods or due to gained experimental experience, makes it possible to choose between the other three processing methods. If it is known that a sample is of regular structure and supposed number of components is not high, it is necessary to formulate a task for model spectrum fitting (SPECTR) involving all the available a priori information into the constructed model. If spectrum quality improvement indicated non-uniform character of crystalline or magnetic structures (when it was not known a priori), one should set a task for reconstruction of spectrum parameter distribution functions (DISTRI) using available a priori information to set corresponding conditions. In case when revealed values $\{a_i\}$ indicate possible presence in the sample of at least one standard sample, it is worth to complete the task of comparison with standard sample spectra ξ(ET) (PHASAN).

In the most simple cases solving of one of these three last spectrum processing tasks results, as a rule, in completion of the analysis and processing. If investigator has got complete enough a priori information about the sample or sample spectrum is of high quality, then need in spectrum quality improvement is eliminated and one can simply use one of the codes SPECTR, DISTRI or PHASAN.

However, there might be a situation when none of the considered methods used individually provides reliable analysis of LIS spectrum. This may be stipulated by wrong method choice at previous stage (due to incompleteness of failure of a priori information AI(A)) or by the complexity of the object under investigation that may contain, for instance, phases with considerably different structure regularity.

If, for example, reconstructed distribution function p(z) can be characterized by a set of local maxima, then, using this result as a priori information AI(A), one could set a task of model fitting.

While if the function p(z) is of a form characteristic for some of the available standard samples, then comparison with the spectra of standard samples would be an advantage.

Similar situation may happen at initial application of the model fitting of spectra. For example, close found optimal parameters $\{b_j\}$ and the natural desire to increase the number of components n in the spectrum for considerable decrease of the functional $\chi^2(\{b_j\})$ or unreasonably large widths of the spectrum components led to formulation of a task to find distribution functions for corresponding parameters.

From the other side, if the values of at least a part of the found parameters $\{b_j\}$, that define in their entity one or several partial spectra, correspond well to a spectrum of at least one available standard sample, then the chance to solve the task of comparison with the spectra of standard samples should be realized.

In its turn, comparison with spectra of standard samples may lead to the conclusion that not all the spectrum of investigated sample is described by this set of spectra of standard samples. In this case depending on available a priori information it becomes very natural to run model fitting of the non-described part of the spectrum or to reconstruct distribution functions for the parameters of this part.

Conclusions

Effectiveness of Moss Bauer spectroscopy for investigation of LIS depends greatly on adequacy of our physical understanding and adequacy of information extraction ways to the object of investigation. All the experience gained by the authors for the last years [4 – 17] in investigation of LIS showed fruitfulness of the presented above conception of hyperfine interaction local inhomogeneity, reasons for its appearance and mechanisms of its formation as well as high effectiveness of the complex approach to processing and analysis of Mössbauer LIS spectra using a priori information.

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