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LT10 program for solving basic problems connected with defect detection

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Abstract

Features of LT10 software for positron annihilation lifetime (PAL) spectra analysis are presented. It is a completely new version of well known LT9.x series with newly designed intuitive user interface. The program allows to significantly reduce the number of freedom degrees in the numerical analysis of PAL spectra. It is accomplished by simultaneous analysis of a series of spectra with regard of the correlation between related parameters of each spectrum in the series. A few examples of the program application are presented in the paper.

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Keywords: PAL spectra, numerical analysis, source correction

1. Introduction

It is well known that PAL spectra usually can be fitted in many ways (i.e. using many various sets of parameters and their values) with similar quality of fits. Therefore, extraction of the reliable ones from the PAL data requires introduction of a different type of constrains during their numerical analysis. ‘False’ fits may be, of course, partially excluded if the parameter values are unphysical, but often there are still many probable parameter configurations which cannot be discarded with certainty. That is why reducing free parameter count is so desirable. Another uncertainty in PAL spectrum analysis is connected with the choice of the theoretical model fitted. Sometimes, there is a necessity of applying a new, user-defined

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model. All of those problems are taken into account in LT10 – the latest version of the well known LT series [1]. This paper shows how even very complex tasks can be solved thanks to the LT10 software.

2. Program main features

The biggest advantage of LT programs, including the newest version, is the ability of reducing the number of free parameters. This may be achieved in LT10 in many ways. The easiest one, already available in previous versions of LT (especially LT9.x), is to use a combination of two most important program features: a) multiple spectra analysis, and b) so called *parameter statuses* which define various kinds of constrains imposed on the parameters. Analyzing multiple spectra brings the advantage of finding common values for chosen parameters. Such processing makes the resulting value more reliable in the case when one parameter should have the same value for all spectra. The available parameter statuses as well as the parameter values organization in LT10 are shown in Table 1. In the program, each parameter is represented by a single column to which the user assigns the *parameter status*.

Table 1. Possible *parameter statuses* used in the LT10 program where ‘v’ stands for a free value and ‘c’ – for a constant value of a parameter in the analysis process.

parameter statuses	<i>local free</i>	<i>local fixed</i>	<i>common free</i>	<i>common fixed</i>	<i>partially common free</i>	<i>partially common fixed</i>
spectrum ₁	v ₁	c ₁				
spectrum ₂	v ₂	c ₂			v _{1,2}	c _{1,2}
spectrum ₃	v ₃	c ₃	v _{ALL}	c _{ALL}		
spectrum ₄	v ₄	c ₄			v _{3,4}	c _{3,4}
spectrum ₅	v ₅	c ₅			v ₅	c ₅

In LT10, a series of spectra can be analyzed not only with one theoretical model but also with several theoretical models simultaneously. Each model is assigned to a separate part of the analyzed series. Such part of spectra is called “document”. In the case of multi-document analysis, for further reduction of free parameter count, some of the parameters (so called *interdocument parameter*) or even whole groups of parameters (*shared group*) may be set as common. For example, parameters related to the positron source contribution can be chosen as a *shared group* since usually all spectra are measured with the same positron source.

Besides the standard theoretical models already defined in the present release of LT10¹, the program is open for other theoretical models in a plug-in-like form which may be even designed and built by end-users.

3. Examples of LT10 use

3.1. Source correction

It is recommended to divide every analysis of spectra into two steps. First, the parameters independent on the measured samples should be determined, i.e. positron lifetimes and fractions of particular annihilation processes in the source, and the parameters related to the spectrometer resolution. Then in the

¹ In the time of writing this paper, current version of LT10 is 10.1.5.3. Standard theoretical models are as follows: multiexponential, multiexponential with continuous lifetime distributions, and three kinds of simple trapping models.

second step, in which the analysis of the samples of interest is performed, each of these parameters is treated as a known constant. The first step is often accomplished with use of some reference samples characterized by as few as possible well known lifetime components. It is suggested [2-4] that c_s depends on atomic number of sample, therefore such approach is used in the following example.

Table 2. Screenshot from LT10 project showing the lifetimes and intensities of source components and total source contributions for all spectra analyzed. The key values are atomic numbers of samples used in the analysis

Spectrum	Key value	Model	Fit	int ₁	τ_1	int ₂	τ_2	Contrib.
					Commo...	Commo...	Commo...	mixed
Si109IX	14	IX2009	1.1067	45.7925	0.1522	54.2	0.3710	38.69
Si11V	14	V2011	1.2595	-	-	-	-	-
Si209IX	14	IX2009	1.0490	-	-	-	-	-
Ni11V	28	V2011	1.0911	-	-	-	-	42.0
Cu11V	29	V2011	1.1659	-	-	-	-	43.45
Cu09IX	29	IX2009	1.2168	-	-	-	-	-
In109IX	49	IX2009	1.1716	-	-	-	-	48.75
In209IX	49	IX2009	1.6061	-	-	-	-	-
Sn09IX	50	IX2009	1.1822	-	-	-	-	49.1
Sn11V	50	V2011	1.1102	-	-	-	-	-

In our case, the positron source was $^{22}\text{NaCl}$, deposited between two layers of 3.6 mg/cm^2 ($25.5 \text{ }\mu\text{m}$) Kapton foil [5]. The spectrometer resolution was approximated by two Gaussians with average value of FWHM (full width at half of maximum) equal to 246 ps. The reference samples were well annealed Si, Ni, Cu, In, and Sn, all of high purity (at least 99.99%). Positron lifetimes in bulk of those samples were fixed at *local* values of 218 ps for Si [6, 7], 95 ps for Ni [8], 120 ps for Cu [6], 192 ps for In [9], and 199 ps for Sn [10]. All the reference spectra were analyzed simultaneously and the lifetimes and related intensities in *source group* were set as parameters of the *common free* status. Due to the dependence of the source contribution on the type of sample, c_s had individual value for each sample used. Since each reference material was measured several times, c_s related to the same material was grouped using the *partially common free* status (see table 1). Table 2 shows the parameters grid which plays role as the

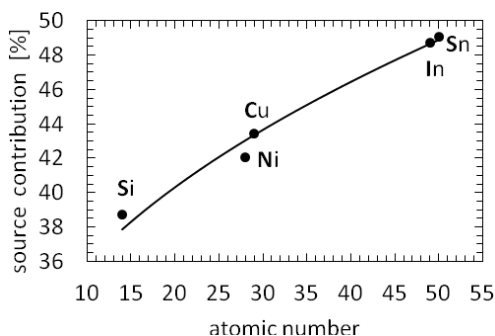


Fig. 1. Source contributions for reference samples calculated with LT10 (●) and fitted curve proposed by Płotkowski et al. in [2] (—).

input-output control. To distinguish between different *parameter statuses*, different colors of cell boundaries are used (e. g. green for *common free*). Additionally, in the case of the *partially common* statuses small rectangles appear in the cells, as it is seen in the “Contrib.” column.

Usually one lifetime component in Kapton is reported by authors. In our case though, the best fit were obtained with two components in source of lifetimes 152 ± 1 and 371 ± 1 ps. This is in good agreement with values reported by Dauwe et al. [11], however intensities of the respective components are different. The determined values of c_s for the reference samples, we approximated using the relation proposed by Płotkowski et al. (eq. 11 in [2]), with modified formula for the backscatter coefficient [12], i.e. $p_+ = 0.1513 Z^{0.4724}$, where Z is atomic number of the sample material. Similar studies were performed in [3], with indium spectra measured between Kapton layers of various widths. In the case of $25.5 \mu\text{m}$ Kapton, the estimated value of c_s was 41.8% which is a bit lower than our value (48.8%).

From the theoretical curve (Fig. 1), one can find the source contribution for other sample materials. For example, for $\text{Fe}_{75}\text{Al}_{25}$ samples (average $Z=22.75$) c_s can be estimated to 41.3%. This value of c_s , as well as positron lifetimes in source and their relative intensities were used as constants of *common fixed* statuses in the analysis described below.

3.2. Defects in Fe-Al

This example illustrates the procedure of determination of defect concentration using the two state trapping model². Samples of interest are, $\text{Fe}_{70}\text{Al}_{25}\text{X}_5$ ($X = \text{Fe}, \text{Ni}$) after various heat treatment. During analysis of spectra of these samples, all parameters related to the positron source were constant (*common fixed*), which greatly decreased the number of free parameters. Assuming that all the samples are characterized by one value of positron lifetime in defect (τ_D) and one value in the bulk (τ_f), these two parameters had *common free* status. Only the trapping rate (κ_D) was treated as a *local free* parameter. An overview of such an analysis is presented in Table 3.

Table 3. LT10 project screenshot with resulting sample parameter values. Positron lifetime in defect τ_D as well as lifetime in bulk τ_f were *common free* parameters. Only κ_D parameter was *local free*.

Spectrum	Key value	τ_D	κ_D	τ_{free}	int _D	τ_{avg}
		Common	Local Free	Common		
12302_2Alk_sum	12	0.168801	6.94261	0.113674	70.7309	0.13799
13302_3Alk_sum	13	-	5.93315	-	67.3757	0.135878
14302_4Alk_sum	14	-	24.2808	-	89.4198	0.15414
15302_5Alk_sum	15	-	1.51567	-	34.5366	0.121776
16302_6Alk_sum	16	-	11.703	-	80.2899	0.145145
32302_2NiK_sum	32	-	28.6688	-	90.8917	0.155857
33302_3NiK_sum	33	-	26.9092	-	90.3535	0.155219
34302_4NiK_sum	34	-	78.4859	-	96.4688	0.163245
35302_5NiK_sum	35	-	8.71101	-	75.1991	0.141102
36302_6NiK_sum	36	-	14.9409	-	83.8726	0.148371

² In this example, we are not going to discuss the details related to the particular problem (its full discussion can be found elsewhere [13]) but to propose a unique approach that can be useful in solving such types of problems.

3.3. Analysis of a set of simulated spectra

This example compares a constrained and non-constrained analyses performed for the same set of simulated spectra. For this purpose, 12 spectra have been generated using two state trapping model. All the spectra had the same defect ($\tau_D=165$ ps) and bulk ($\tau_f=120$ ps) lifetimes but different κ_D parameters. To bring this problem closer to reality, the spectra were generated with the source and resolution curve parameters equal to those obtained from the experimental data described in Sec. 3.1. The statistics of each

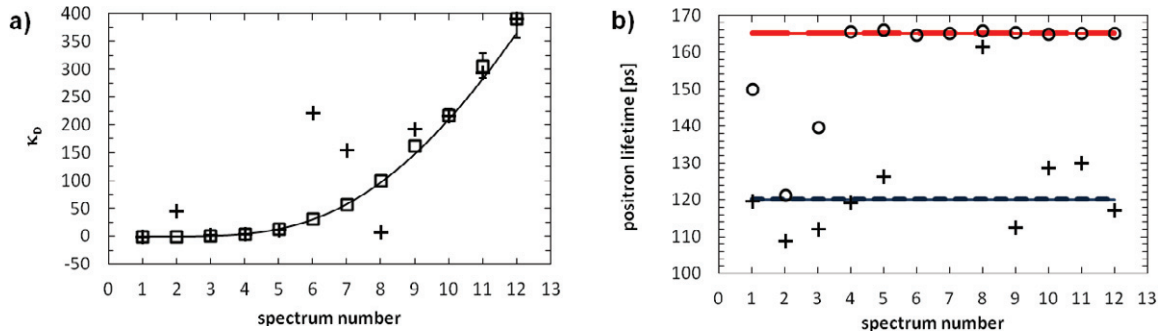


Fig. 2. Comparison of two approaches in PAL spectra analysis. a) κ_D determined using constraints (\square), without constraints ($+$) and values assumed in simulations (—). The bars show statistical errors estimated by the fitting procedure. Error bars for values obtained in non-constrained analysis are skipped for figure clearance due to their large values. b) τ_D determined from constrained analysis (---), non-constraints analysis (\circ), assumed in simulations (—) and τ_f from constrained analysis (---), from non-constrained analysis ($+$), assumed (—).

spectrum was 10^7 counts, a constant background was added and the points of the spectrum were scattered with a proper Poisson noise.

In the first approach, both lifetimes in the sample (defect and bulk) and all parameters of the resolution curve had the *common free* status, only κ_D was a *local free* parameter. In the second approach, all the spectra were analyzed once more with the same theoretical model but without any constraints imposed on τ_D , τ_f and κ_D . In both analyses, the parameters of source and the resolution curve were fixed at values used in simulations. Differences between these two approaches are presented in Fig 2.

One can see that values of κ_D determined in the constrained analysis are equal to the assumed ones within the range of statistical errors estimated by fitting procedure. Similarly, values of τ_D and τ_f obtained using constraints are also very close to the assumed ones, i.e. 165.1 ± 0.1 and 120.1 ± 0.2 , respectively. The non-constrained analysis, even if characterized by a bit better quality of fits (mean $\chi^2 = 0.9943$ for constrained analysis and 0.9939 for non-constrained one) resulted in much more scattered values of fitted parameters.

4. Conclusion

LT10 with many binding-oriented features makes analysis of PAL spectra easy and reliable. The program allows to draw one consistent result from information scattered throughout many different spectra due to various types of constraints imposed on parameters during the spectra analysis. In some cases, using constraints is crucial to obtain proper results as it is shown with help of the simulated spectra.

The program is available at <http://prac.us.edu.pl/~kansy>

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