

Positron-annihilation monitoring of reduction processes in conducting glasses[☆]

Damian Pliszka^a, Bogusław Kusz^b, Maria Gazda^b, Konrad Trzebiatowski^b,
Grzegorz P. Karwasz^{c,*}, Roberto S. Brusa^c, Antonio Zecca^c

^a Institute of Physics, Pedagogical University of Słupsk (IF PAP), Arciszewskiego 22B, Słupsk 76-200, Poland

^b Instytut Fizyki, Politechnika Gdanska, Narutowicza 11/12, Gdansk 80952, Poland

^c Dipartimento di Fisica and INFN, Università di Trento, via Sommarive 14, Trento 38050, Italy

Received 19 September 2003; received in revised form 10 February 2004; accepted 16 February 2004

Abstract

Depth-resolved, Doppler-broadening positron spectroscopy has been used to study structural properties of semiconducting glasses. Bismuth- and lead-doped, silica and germanium glasses with five different compositions were studied. Conducting surface layers in these glasses were obtained by reduction in hydrogen atmosphere. Depth scanning with a slow-positron beam allows to follow the temperature and time evolution of layers modified during reduction processes. However, while reduction processes in inner layers (below a few tens of nm) are clearly seen in changes of the Doppler-broadened positron-annihilation line, the near-to-surface changes are more complex and not fully understood. We prove that the positron spectroscopy can help in optimising technological questions of reduction processes.

© 2004 Elsevier B.V. All rights reserved.

PACS: 73.61.Jc; 61.82.Rx; 68.37.Rt; 61.72.Qq

Keywords: Bismuth–silica glass; Bismuth–germanate glass; Lead–silica glass; Conducting glass; Reduction; Microscopy; Positron spectroscopies

1. Introduction

Bismuth- and lead-doped silicon and germanium-oxide glasses are used, among other applications, for electron continuous-dynode multipliers [1]. To make them conducting, reduction procedures in hydrogen atmosphere are applied. The depth of the reduced layer must be thoroughly controlled, in order to ensure a high secondary-electrons yield and a low surface conductivity (see Fig. 1). In Fig. 1 the surface conductivity versus time of reduction for samples of series “S” and “G” (see also Table 1) are reported. Furthermore, not only the secondary-electron emission coefficient varies with the reduction conditions, but also the ageing properties of the electron multiplier devices.

The lead-doped glass is the material traditionally used in electron channel multipliers. We have started exploiting a new class of bismuth-doped glasses, which necessitate thermal treatments at lower temperatures than lead-doped glasses [2]. Bismuth–germanate glasses in comparison with bismuth–silicate glasses may contain less bismuth so these materials are complementary to each other.

We have observed, using the atomic force and optical microscopy, some nanometer precipitations of metal inside the near-to-surface layer and accumulation of metal nano-drops on the surface, after long treatment times, 2 h for silica glasses and about 8 h for germanium glasses [2,3]. The metal drops are Bi and some Ge in the case of bismuth–germanate glasses and solely Bi in the case of silica glasses. The purpose of this work is to see the early stages of annealing when these reduced layers and drops on the surface start to appear, before these layers can be identified by standard microscopy techniques.

We have applied the positron-annihilation technique, which is depth-resolved and non-destructive, and permits

[☆] We dedicate this article to the memory of Professor A. van Veen, who has given important contributions to the development of positron physics.

* Corresponding author. Fax: +39-0461-881696.

E-mail address: karwasz@science.unitn.it (G.P. Karwasz).

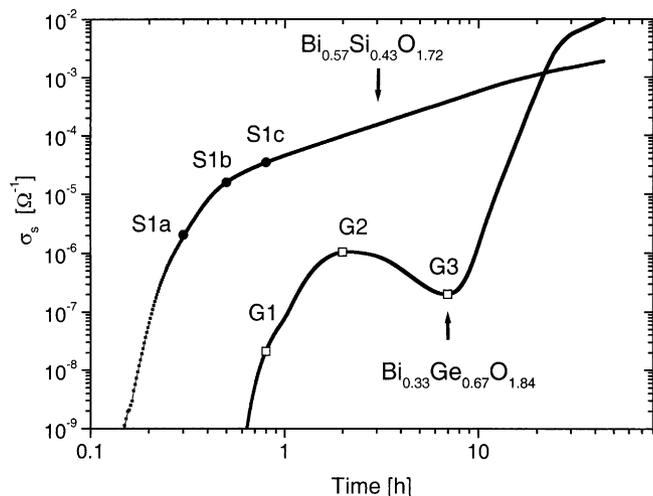


Fig. 1. Changes in the surface conductivity for silica-based (“S” series) and germanium-based (“G” series) bismuth-doped glasses (adapted from Ref. [2]) during heat treatment at 340 °C.

to study defects at sub-nano-scale, i.e. single vacancies, dislocations, and change in the structure of the materials. Glass samples, similar to those used in our study [3] were prepared and characterized at Gdańsk Technical University while positron measurements were done at Trento University. Partial experimental results of positron investigation have been already published [3,4] but the complete, comparative analysis for different glasses is performed only now.

2. Experimental

Conducting lead glasses samples were produced from SiO_2 and PbO_2 oxides. Bismuth glasses were prepared from milled SiO_2 or GeO_2 powder and bismuth nitrate $4\text{BiNO}_3(\text{OH})_2 \cdot \text{BiO}(\text{OH})$. The mixture was decomposed at 700 °C and then the obtained mixture was ground again and melted at 1200 °C, see Ref. [2] for details. Five series of conducting glasses were produced: one lead–silica, two bismuth–silica and two bismuth–germanium glasses. Details on stoichiometry and thermal treatment are given in Table 1.

Thermal treatment in hydrogen atmosphere was done to create surface reduced layers. The annealing temperature was 300 °C for bismuth–silica glasses of series I; 340 °C for bismuth–silica glasses of series II and bismuth–germanate glasses of series III and IV, and 400 °C for lead–silica glasses of series V. During reduction both oxygen and water vapour were desorbed from the samples.

Positron-annihilation spectroscopy is a non-destructive technique permitting to determine essentially the presence of defects in solids. Defects, like vacancies and dislocations constitute trapping centres for positrons injected into samples and cause the rise of positron lifetimes and changes in the shape (broadening) of the annihilation gamma line, at 511 keV (see Ref. [5] for details about positron techniques). In the present measurements the Doppler-broadening of the annihilation line was studied and a variable-energy positron beam described elsewhere, was used [6]. Positron implantation energy E was changed from 50 eV to 25 keV. This,

Table 1

Stoichiometry, thermal treatments, and positron-annihilation data for surface-conducting glasses

Series	Stoichiometry (density)	Symbol	Thermal treatment	Reduced layer			
				From-to (nm)	S (surface)	From-to (nm)	S (bulk)
I	$\text{Bi}_{0.3}\text{Si}_{0.7}\text{O}_{1.85}$ (5.8 g/cm ³)	A0	As-received	–	0.516	–	0.489
		A1	300 °C/3.3 h	0–20	0.5252	–	–
		A2	300 °C/25 h	0–25 (?)	0.504	25–500	0.540
		A3	300 °C/91 h	0–8	0.54035	35–8	0.542
II	$\text{Bi}_{0.57}\text{Si}_{0.43}\text{O}_{1.72}$ (6.46 g/cm ³)	S0	As-received	–	0.492	–	0.469
		S1a	340 °C/0.3 h	0–8	0.490505	–	–
		S1b	340 °C/0.5 h	–	–	0–210	0.535
		S1c	340 °C/0.8 h	–	–	0–600	0.540
III	$\text{Bi}_{0.3}\text{Ge}_{0.7}\text{O}_{1.85}$ (5.65 g/cm ³)	B0	As-received	–	0.495	–	0.477
		B1	340 °C/0.4 h	0–12	0.484	–	–
		B2	340 °C/1.3 h	0–6	0.5084	15–300	0.519
		B3	340 °C/4 h	–	–	5–700	0.531
		B4	340 °C/5.5 h	–	–	3–1000	0.532
IV	$\text{Bi}_{0.33}\text{Ge}_{0.67}\text{O}_{1.84}$ (5.77 g/cm ³)	G0	As-received	–	0.493	–	0.471
		G1	340 °C/0.8 h	4–70	0.484	–	–
		G2	340 °C/2 h	–	–	15–120	0.507
		G3	340 °C/7 h	–	–	15–800	0.525
V	$\text{Pb}_{0.4}\text{Si}_{0.6}\text{O}_{2.0}$ (5.46 g/cm ³)	C0	As-received	–	0.515	–	0.497
		C1	400 °C/21 h	–	–	1–700	0.536

Thickness of the reduced layers (in nm) and S-parameter are determined using VEPFIT program [11]. Surface and bulk S-parameters are given in sixth and eighth columns, respectively.

according to a simplified formula [5]: $d = (40/\rho)E^{1.6}$ (where ρ is the sample density in g/cm^3 , d is expressed in nm and E the energy in keV) gives the positron implantation range down to $1.0 \mu\text{m}$ for the heaviest glass at 25 keV implantation energy.

A high-purity germanium detector with 1.3 keV resolution and 24% detection efficiency was used to monitor the shape of the annihilation line. We have evaluated this energy resolution on the basis of FWHM of the 356 keV ^{133}Ba line (FWHM was 1.08 keV). The stability of the energy resolution of the detector and the stability of the positron beam was controlled thoroughly by methods described elsewhere [6–8].

The S -parameter, defined as the ratio between the number of counts in the central part of the broadened 511 keV line and the total number of counts in this line is commonly used to monitor the presence of defects in solids. In presence of open volume defects, this parameter rises as compared to a defect-free material, due to the enhanced probability of annihilation with valence (i.e. low-momentum) electrons, see [5]. In the present measurements the S -parameter is defined as the number of counts in the $|511 \text{ keV} - E_\gamma| \leq 0.85 \text{ keV}$ window compared to the total number of counts in the $|511 \text{ keV} - E_\gamma| \leq 4.25 \text{ keV}$ peak. The W -parameter defined as the ratio of positrons in the “wing” $1.6 \text{ keV} \leq |511 \text{ keV} - E_\gamma| \leq 4.0 \text{ keV}$ range to the overall area of the peak describes annihilation of positrons with high-energy electrons (i.e. core). The S versus W curves permit to extract partial information about chemical elements surrounding an open volume defect.

The experimental uncertainty mainly depends on the counts accumulated in the annihilation line. Several spectra, up to typically 2×10^5 counts have been accumulated per point. Lower total counts characterise points at positron implantation energies below 1 keV, see Ref. [8]. As a conservative number we give a 0.5% statistical error on S -parameter values above 1 keV implantation energy and somewhat higher uncertainty at lower energies.

Present spectra have not been deconvoluted for the energy resolution of the detector: as the reference value we quote the value of $S = 0.523$ from our laboratory for the bulk of defect-free, p -type, floating-zone grown silicon, which we used previously [9,10] for normalization of silicon-related spectra. With this normalization, the S -parameter in bulk quartz, as given in review [5] would amount to 0.465 and for SiO_2 layers thermally grown in dry deposition processes it would be 0.51.

The S versus E curves have been analysed with the variable-energy-positron fit (VEPFIT) [11] program that models the diffusion and trapping of positrons after their implantation and thermalization. The program permits to determine the depth of up to six layers with relative S -parameters and the positron diffusion length in each layer. Additionally, it determines the S -parameter for the sample surface and the S -parameter for epithermal positrons (injected to the sub-surface layer).

3. Results

Results of S -parameter measurements and VEPFIT analysis of the samples reported in Table 1 are presented in Figs. 2–7. In Fig. 2 we present the S -parameter data for non-reduced samples (for clarity we show only the “smoothed”, VEPFIT results) and we compare with the data from other laboratories for natural quartz [12] and for

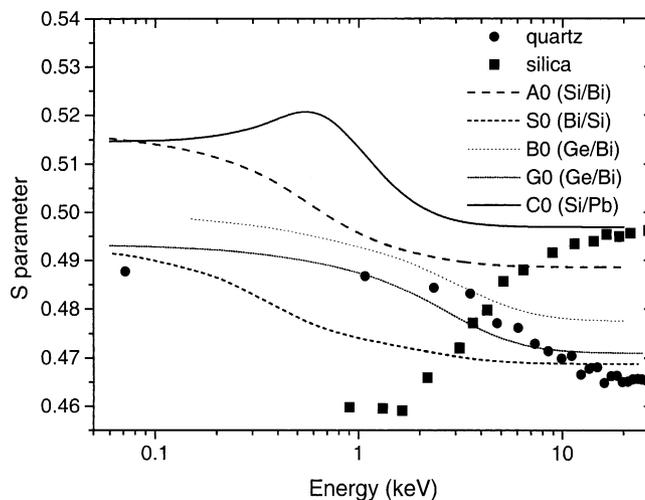


Fig. 2. The S -parameter dependence for as-obtained glass samples, see Table 1 for samples description. The fit on the experimental curves obtained by the VEPFIT [11] programme. Quartz data were taken from measurements of Fujinami et al. [12]; data for synthetic silica glass are taken from measurements of Anwand et al. [13]. Brazilian quartz samples, as measured by Anwand et al. [13] (with energy resolution for the S -curve of 1.09 keV) show a similar dependence as the data of Ref. [12] but are much lower (0.42 in bulk) — out of scale on this figure. The quartz and silica data have been rescaled on abscissa scale, in order to account for the difference in the sample density.

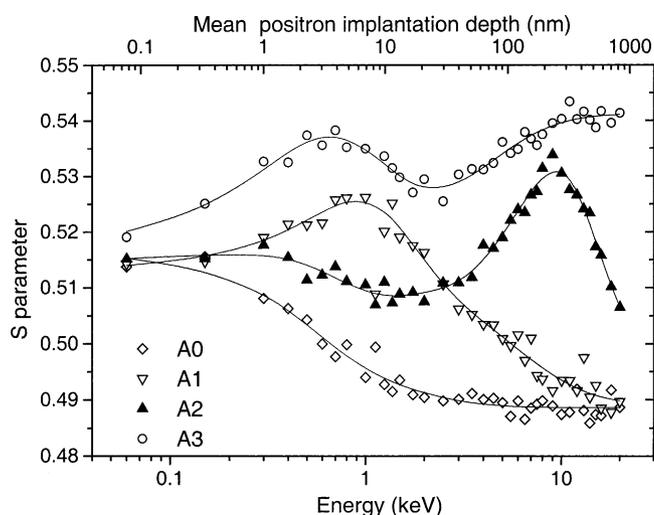


Fig. 3. Positron-annihilation S -parameter vs. positron implantation energy for bismuth-silica glasses (series I, with Si molar contents higher than Bi). Modelling of the S -parameter curves, has been performed using the VEPFIT [11] program. Increase of S -parameter in the formed reduced layer with rising reduction time is clearly seen.

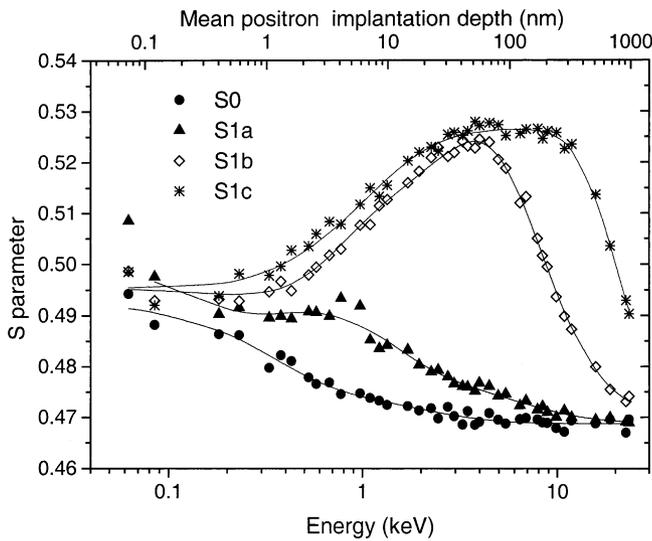


Fig. 4. Positron-annihilation S -parameter vs. positron implantation energy for bismuth-silica glasses (series II, with majority of Bi). Modelling of the S -parameter curves has been performed using the VEPFIT [11] program.

silica glass [13]. Please, note that due to different energy resolutions between our and other [12,13] experimental set-ups, only general shapes of curves can be compared and not their absolute values.

The values of S -parameter show rather big differences between non-reduced and reduced glasses — reduction processes generally rise the S -parameter, compare Fig. 2 with Figs. 3–7. It seems that for the S -parameter, the changes in the chemical composition of glass are less important than reduction processes. For reduced glass samples, the S -parameter shows non-monotonic dependencies, rising with the depth for higher reduction times.

The S -parameter for all as-received glasses falls monotonically with the positron implantation energy. (A bump for

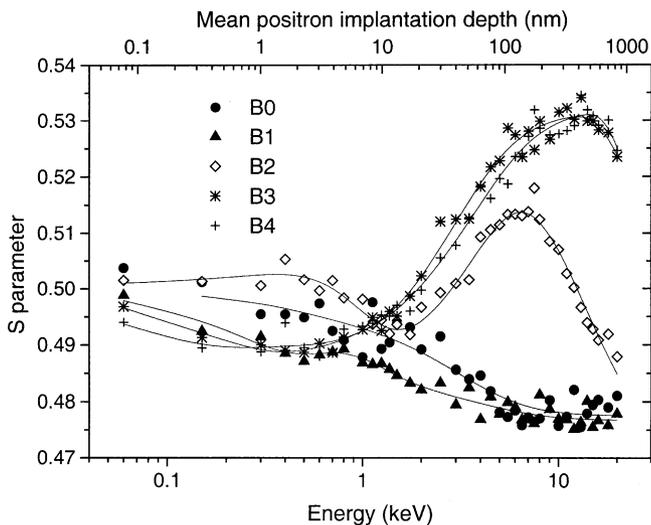


Fig. 5. The S -parameter for bismuth-germanium glasses (series III). Note a complex change of the S -level with the reduction time for the first 10 nm depth.

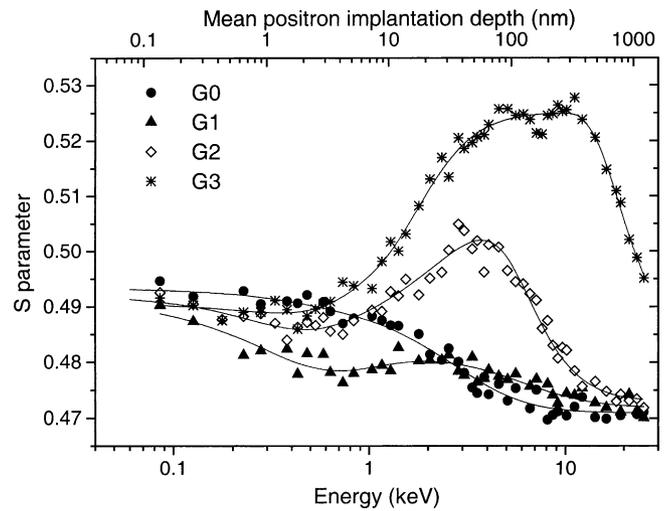


Fig. 6. The S -parameter for bismuth-germanium glasses (series IV, similar to series III).

Si/Pb glass (sample “C0”) is probably a VEPFIT artefact — experimental data are much scattered, due to a lower counting rate in those measurements.) This behaviour resembles the dependence for defect-free quartz [12], see Fig. 2. Also for the synthetic quartz, the S -parameter falls monotonically with the positron implantation energy, see Ref. [13]. From Fig. 2 one notes that the surface S -value are almost the same, 0.515 for glasses “A0” and “C0”, in which silicon is the majority component over bismuth or lead, respectively. On the other hand, in sample “S0” containing more Bi than Si and in samples Ge/Bi (“B0”, “G0”) the surface S -value is lower (about 0.493). Note that both for quartz [12] and for the native oxide on Si from Trento laboratory [8] the S -values at the surface are low, about 0.495.

The bulk S -parameter value for non-reduced samples does not correlate with solely one of the metallic components: the highest bulk value (0.497) has been measured for Si/Pb

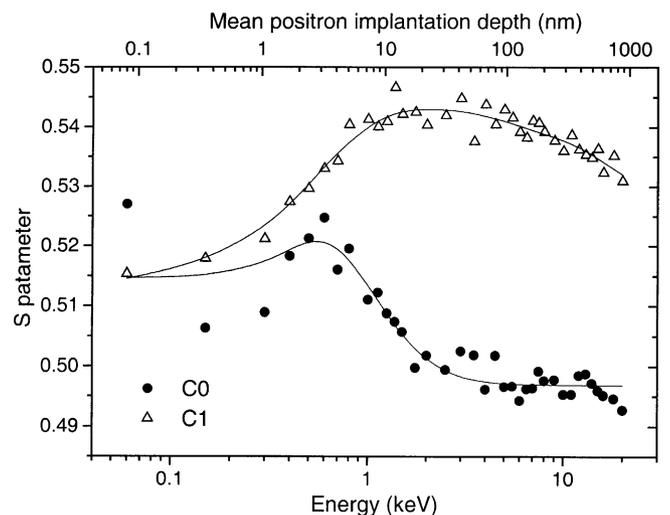


Fig. 7. The S -parameter for lead-silicon glasses (series V). Modelling of the S -parameter performed using the VEPFIT [11] program.

glass, a lower (0.489) for Si/Bi glass “A0” and the value of 0.47 for Ge/Bi. The S bulk value in Si and Ge glass decreases as the content of Bi increases. What seems to be dependent on the glass matrix type is the diffusion length, which amounts to 15–20 nm for Si-based glasses (samples “A0”, “S0” and, less evident, also “C0”) and to 35–40 nm for Ge-based glasses.

Structural changes in reduced samples (of series II and IV) were previously studied [2,3] by the atomic force microscopy, optical microscopy and X-ray diffraction methods. For example, after 44 h reduction time in Si/Bi samples (series II) rather big, about 10 μm , microcrystals of rhombohedral Bi on the surface and a 50 μm thick layer of Bi-granules embedded in the glass were observed. For the same long reduction time, Ge/Bi samples show a thin, 4 μm layer of embedded granules and nano-drops of Bi with some admixture of Ge metallic phase on the surface [2]. In positron measurements we observe, with long reduction times (see Table 1), a general rise of the S -parameter, to about 0.53–0.54, almost independently on the kind of glass. Such high values have been measured for highly defected SiO_x non-stoichiometric layers, see [9] (we reported there the S -parameter values normalized to silicon bulk — without normalization the S -parameter would amount to 0.53–0.54, depending on oxygen deficiency in SiO_x). In the reduced layers nano-precipitates of Bi in GeO_x and SiO_x matrices are formed. In such a structure positrons are expected to mainly annihilate in defects at the interfaces of nano-precipitates and the open structures of SiO_x [9] and GeO_x , giving the observed increase of the S -parameter.

Let us consider first Si/Bi glasses: the two series “A1–A3” (Fig. 3) and “S1a–S1c” (Fig. 4) show some essential differences. Samples of series “A”, with 30% molar contents of Bi (relative to Si), require very long (up to several days) reduction times in order to obtain satisfactory high surface conductivity and secondary-electron emission coefficients [3]. From S -curves one notes that 3 h reduction time (sample “A1”) do change this glass, apart from the very superficial, 20 nm deep layer — a slight rise of the S -parameter is observed, see Table 1. In sample “A2”, heated for 25 h, the near-to-surface layer seems to disappear if compared to “A1” — a modified layer with a high value of S -parameter (0.54) extends from 25 to 500 nm depth. However, in order to reproduce the S -curve in sample “A2” we had to assume two layers and a non-modified bulk. The near-to-surface layer extends down to 25 nm and shows the S -value (0.50) slightly higher than that in non-reduced bulk (0.49); the positron diffusion length in this layer is about 5 nm, much shorter than in the bulk. In sample “A3” (heated for almost 4 days) both, the near-to-surface (first few nm) and the deeper (below 35 nm) layers have the same, high (0.54) S -parameter indicating a new structure with more open volumes.

In samples “S1a–S1c”, silica-based with a higher contents of bismuth than series in “A”, we do not observe such a complex reduction dynamics as in series “A”. For

the whole series only one reduced layer is seen. In sample “S1a” it resembles the thin layer in “A1” and extends down to about 10 nm, with the S -parameter only slightly higher (0.49) than in the non-reduced bulk. In samples “S1b” and “S1c” — a single layer with a high S -parameter (0.54), extending down to 200 and 600 nm, respectively, is observed.

Germanium-based bismuth glasses (Figs. 5 and 6) show a somewhat more gradual rise of the S -parameter value in the reduced inner layer than it was seen for “S1b–S1c” samples. For example, both for “S1b” and “B2” samples the modified inner layer extends down to 200–300 nm but for “B2” sample the S -parameter is lower, about 0.52, than the “saturation” value (0.532 for sample “B4”). Similarly, in samples “G”, the S -parameter rises gradually in the (inner) reduced later, from 0.47 for non-reduced bulk (“G0”) to 0.507 in sample “G2” and 0.525 in sample “G3”.

The changes of S -parameter in the first 10 nm in Bi/Ge glasses are quite complex. Reduction times shorter than 1/2 h (samples “B1” and “G1”) cause a decrease of the S -value in the first 10 nm, below the values for “as-obtained” glasses, see Figs. 5 and 6. In sample “B2”, treated for 1.2 h we observed a rise of the S -parameter in the first few nm. With longer reduction times, when the inner modified layer is well developed, the near-to-surface S -parameter is again low, below the S -received value (samples “G2”, “G3”, “B3”, “B4”). Somewhat similar changes can be seen also for “A” Si/Bi series, but for Ge/Bi glasses they are more evident. We recall the conductivity curve, Fig. 1, in which lowering of the surface conductivity is observed for Ge/Bi glasses for 1–2 h annealing time.

VEPFIT analysis hardly gives any quantitative insight on these near-to-surface changes of the S -parameters. Modelled S -values depend on several input data, like epithermal positron scattering length and the diffusion length, and these vary with changes in the glass structure (and density). Qualitative analysis would indicate different type of changes in inner and outer layers. In the inner layer a more defected structure, resembling SiO_x [10], while in the outer — a more compact (S rises but remain lower than in SiO_x), metal-enriched composition is observed. For example, Then and Pantano [1] observed a silicon rich zone in the outer 20–50 nm layer of the surface layer. Other techniques, more element-specific than S -parameter measurements, must be applied for understanding differences between the two layers observed in our present and previous [2] measurements.

Changes in lead glasses, series “C” are clear although experimental errors are somewhat higher than in series I–IV. The reduced layer in sample “C1” extends beyond the positron-implantation range of our apparatus.

It is important to note that in spite of the fact that S -parameters in non-reduced samples differ much between single glasses (from 0.47 to about 0.50), in all reduced glasses the S -parameter values are closer (0.53–0.54). The S -parameters in reduced layers prove to be almost

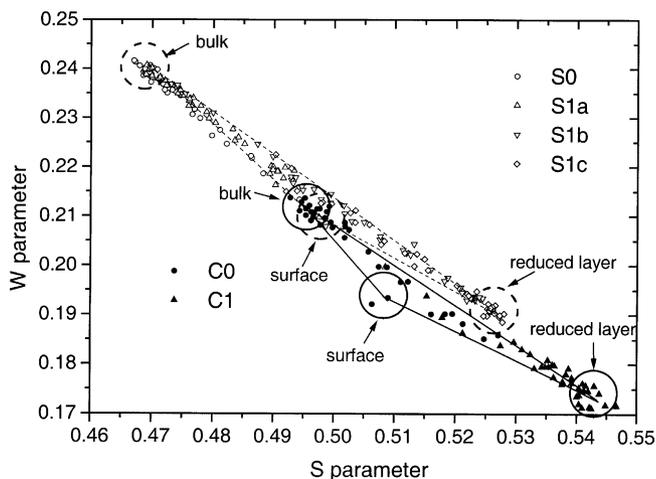


Fig. 8. Positron-annihilation parameters W vs. S plot for selected bismuth-silicon and lead-silicon glasses (series II and V). Experimental S/W points lay on two different triangles for the two glass series, with vertices signed by circles. In “as-received” “S0” glasses the (open) points lay on a segment between the upper (open) circle, corresponding to a surface-annihilation site and the middle (open) circle corresponding to a non-modified bulk. The lower vertex of the open-circle triangle corresponds to annihilation sites in the modified glass. Similar dependencies are observed of Si/Pb glass (full points and heavy-line triangle).

independent on the kind of glass (0.54 with experimental error for silica glasses, 0.53 for germanate glasses).

To get some more insight on the nature of positron-trapping sites, in Figs. 8 and 9 we show plots of the W -parameter versus the S -parameter for four series of samples. The W/S plots allow to determine how many types of annihilation centres co-exist in the glass. If the annihilation takes place only in the surface-related and in the bulk defect-free sites, all W/S points lay on one segment. If several kinds of annihilation centres co-exist in the material, surface, bulk and

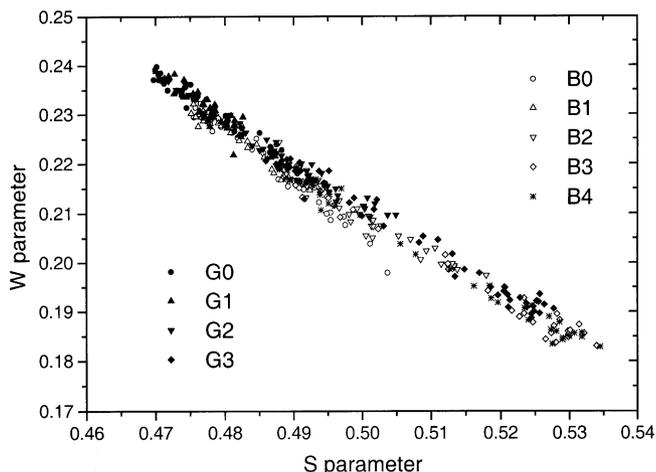


Fig. 9. Positron annihilation W vs. S plot for selected bismuth-germanium glasses (series III and IV). These data show a bigger scatter than the data from Fig. 8, but all of them lay in the same region of the S/W plane, indicating a similar nature of trapping centres in both samples.

defects, then the W/S plot shows some separated straight segments [14].

In Fig. 8 we compare two types of glasses, Si/Pb and Si/Bi. The points for these two glasses form two distinct triangles (with vertices signed by circles): annihilation sites are not the same in the two types of glasses. This confirms the conclusions from “as-received” curves, Fig. 2 — the annihilation sites are *glass-specific* and not related to one of the components, Si or Pb (Bi). For the non-reduced sample “S0” only one surface and one bulk annihilation-sites are present. For samples “S1a–S1c” another type of annihilation sites appear.

For Ge/Bi glasses, see Fig. 9, the spread of points is somewhat large but essentially full points (“G” series) and open points (“B”) series occupy the same region of the S – W plane: annihilation sites are the same in both glasses. However, the univocal identification of the atoms near the positron-trapping sites would require investigation with positron-coincidence techniques [15].

4. Conclusions

Present analysis of positron measurements prove to be complementary to other methods used in characterisation of conducting glasses. The positron beam method allows to follow the growth of the modified layer with the reduction time, in particular at early stages of the process. For example, silica glass samples “A3” (Bi) and “C1” (Pb) show that the used reduction times (91 h at 300 °C and 21 h at 400 °C, respectively) caused “over reduction” of the samples: the reduced layer thickness extends over 1000 nm (see Figs. 3 and 7). Samples of series II–IV, annealed at slightly higher temperature (340 °C) have thinner reduced layers, so it is possible to observe them for the entire section (Figs. 4–6).

We observe also some changes in near-to-surface layers, like for samples “A2”, “S1a”, “B1”, “G1”. However, positron measurements are not able to show the presence of any metallic layers on the surface and indicate only a complex dynamics of the reduction processes in the near-to-surface layers.

The conclusion that the bulk values of S -parameter depends on the whole composition of the *glass* and does not correlate just to one metallic component is important. It indicates that positrons annihilate in sites surrounded by several different atoms — the glass seems to be homogeneous at a nano-scale.

Essential difference between silica-based and germanium-based bismuth glasses is that in Ge/Bi glasses more gradual changes of the S -parameter in modified layers are observed than in Si/Bi ones. Also, for similar treatments, like for samples “S1c” and “G3”, the reduced layer remains somewhat thinner for germanate glasses. Therefore, Ge/Bi types of glasses seem to be more appropriate for technological applications than Si/Bi glasses.

References

- [1] A.M. Then, C.G. Pantano, *J. Non-Cryst. Solids* 120 (1990) 178.
- [2] B. Kusz, K. Trzebiatowski, M. Gazda, L. Murawski, *J. Non-Cryst. Solids* 328 (2003) 137.
- [3] D. Pliszka, M. Gazda, B. Kusz, K. Trzebiatowski, G.P. Karwasz, W. Deng, R.S. Brusa, A. Zecca, *Acta Phys. Polon.* 99 (2001) 465.
- [4] B. Kusz, D. Pliszka, M. Gazda, R.S. Brusa, K. Trzebiatowski, G.P. Karwasz, A. Zecca, L. Murawski, *J. Appl. Phys.* 94 (2003) 7270.
- [5] P. Asoka-Kumar, K.G. Lynn, D.O. Welch, *J. Appl. Phys.* 76 (1994) 4935.
- [6] R.S. Brusa, G.P. Karwasz, M. Bettonte, A. Zecca, *Appl. Surf. Sci.* 116 (1997) 59.
- [7] A. Zecca, R.S. Brusa, M. Duarte-Naia, *Measur. Sci. Technol.* 5 (1994) 61.
- [8] A. Zecca, M. Bettonte, J. Paridaens, G.P. Karwasz, R.S. Brusa, *Measur. Sci. Technol.* 9 (1998) 1.
- [9] R.S. Brusa, G.P. Karwasz, N. Tiengo, A. Zecca, F. Corni, R. Tonini, G. Ottaviani, *Phys. Rev. B* 61 (2000) 10154.
- [10] R.S. Brusa, G.P. Karwasz, G. Mariotto, A. Zecca, R. Ferragut, P. Folegati, A. Dupasquier, G. Ottaviani, R. Tonini, *J. Appl. Phys.* 94 (2003) 7483.
- [11] A. van Veen, H. Schut, J. de Vries, R.A. Hakvoort, M.R. Ijpma, in: P.J. Schultz, G.R. Massoumi, P.J. Simpson (Eds.), *Positron Beams for Solids and Surfaces*, AIP Conference Proceedings, vol. 218, London, Ontario, 1990, p. 171.
- [12] M. Fujinami, N.B. Chilton, K. Ishii, Y. Ohki, *J. Appl. Phys.* 74 (1993) 4540.
- [13] W. Anwand, G. Brauer, M. Hasegawa, O. Dersch, F. Rauch, *Acta Phys. Polon. A* 99 (2001) 321.
- [14] M. Clement, J.M.M. de Nijs, P. Balk, H. Shut, A. Van Veen, *J. Appl. Phys.* 79 (1996) 9029.
- [15] W. Deng, D. Pliszka, R.S. Brusa, G.P. Karwasz, A. Zecca, *Acta Phys. Polon. A* 101 (2002) 875.