SINTEZA STICLELOR FOSFATICE DOPATE CU PĂMÂNTURI RARE CU PROPRIETĂȚI OPTICE ÎMBUNĂTĂȚITE▲

SYNTHESIS OF RARE-EARTH-DOPED PHOSPHATE GLASSES WITH IMPROVED OPTICAL PROPERTIES

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Glasses from lithium-aluminum-phosphate system doped with rare-earth oxides were studied. The following molar oxide composition was proposed: 17.88 Li_2O 8.93 Al_2O_3 6.31 BaO 1.27 La_2O_3 63.72 P_2O_5 1.89 RE_2O_3, RE= Tb, Eu.

The glass was melt in an electrical furnace equipped with superkanthal heating resistance, in alumina crucibles, at 1250°C, for minimum 2 hours. In order to improve the optical properties of the final glass a special device was used, consisting in a mechanical device equipped with alumina stirrer for mixing the melt. The rotation speed was varied between 100 and 500 rot/min. The glass was casted in graphite mould and then annealed in an electrical furnace with kanthal heating wire. The glass samples were annealed at the high annealing temperature for minimum 4 hours in order to obtain improved properties.

The light transmission, structure and homogeneity of the obtained glass were characterized by ultraviolet-visible (UV-Vis) spectrophotometry and scanning electron microscopy–energy dispersive X-ray (SEM-EDX) analysis, respectively. Transmission in the visible range was found to be above 90% and the chemical homogeneity was satisfactory.

The optical quality of the glass was enhanced by stirring the melt so that the cords and threads were much more reduced in the glass. The number and dimension of bubbles were minimized by the selection of an appropriate melting-refining thermal program, based on a proper selection of the temperature, duration, type and speed of stirring.

Au fost studiate sticle din sistemul litio-alumino-fosfatic dopate cu oxizi de pământuri rare. A fost propusă compoziția molară oxidică: 17.88 Li_2O 8.93 Al_2O_3 6.31 BaO 1.27 La_2O_3 63.72 P_2O_5 1.89 RE_2O_3, RE= Tb, Eu.

Sticlele au fost topite într-un cuptor electric echipat cu rezistențe din superkanthal, în creuzete de alumină, la 1250oC, pentru minim 2 ore. Pentru a îmbunătăți proprietățile optice ale sticlei obținute a fost utilizat un dispozitiv special, constând dintr-un agitator mecanic cu agitator din alumină, pentru amestecarea topiturii. Viteza de rotație a fost modificată între 100 şi 500 rot/min. Sticla a fost turnată în matriță de grafit şi apoi recoaptă într-un cuptor electric încălzit cu rezistențe de kanthal. Probele de sticlă au fost recoapte la temperatura superioară de recoacere pentru minim 4 ore pentru a obține proprietăți îmbunătățite.

Transmisia luminii, structura şi omogenitatea sticlelor obținute au fost caracterizate prin spectrofotometrie în ultraviolet-vizibil (UV-Vis) şi respectiv, microscopie electronică de baleiaj cu analiză de raze X cu energie dispersivă (SEM-EDX). Transmisia în domeniul vizibil a fost de peste 90%, iar omogenitatea chimică a fost satisfăcătoare.

Calitatea optică a sticelor a fost crescută prin agitarea topiturii, astfel încât striurile au fost mult reduse în sticlă. Numărul şi dimensiunea bulelor au fost minimizate prin selectarea programelor termice potrivite de topire-afinare, pe baza unei alegeri juste a temperaturii, duratei, tipului şi vitezei de agitare.

Keywords: *phosphate glasses, rare-earth oxides, thermal treatment, EDAX, electronic microscopy, optical spectroscopy*

1. Introduction

Glass is an essential material in the context of current civilization. Some solutions to unpleasant side effects in production of glass, at industrial scale and in laboratory are shown by Balta P. [1]. New methods of Spectral Basicity Distribution - SBD – calculation are described in a recent paper continued by showing the possibilities of representing the distribution of structural properties of nano-aggregates [2]. SBD was recognized as a leading property of oxide glasses, which is correlated with all other properties, offering thus general information. Based on the new information on the glass structure, a new concept of nano-heterogeneous structure of the glass melt and solid was formulated [2].

Recent studies showed that it is possible to correlate various properties, especially those related to glass composition (viscosity, expansion coefficient, refractive index, etc.) [3]. Associated numerical values characterizing basicity of glasses, theoretically calculated or experimentally determined, are not always in agreement with experimental results and discrepancies between different theoretical values are not unusual. Models

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of different chemical species and distribution of their chemical bonds in crystalline oxides, in an oxide ionicity approach, were developed on the basis of electronic energy levels and bands of energy in crystalline solids [4].

In order to obtain homogenous glasses there were studied several technics, such as mixing the melt, gas bubbling, introducing special gas atmosphere and adding small amounts of different oxides. Homogenization of melts is precursive to self-organization of glasses [5]. A new method to evaluate the chemical homogeneity of glasses is based on statistical analyses of the volume distribution of crystals developed through thermal treatments [6]. Homogenization of glass melts is made sometimes simple such as using mechanical stirring of glass melts. In practice the manipulation of glass melt flow is mainly based on free convection, which is caused by density differences resulting from the inhomogeneous temperature distribution in the melt. The effects of external Lorentz forces on glass melt flow in crucibles were studied using a special experimental facility. The equipment enabled the direct electric heating of the glass melt via two electrodes and the generation of Lorentz forces using an external magnet system [7]. Some traditional methods for the optimisation of melting processes and the improvement of quality level are presented in Table 1 [7].

Phosphate glasses become very interesting to study in past decades, due to their different properties and application, according to their composition, such as materials for biomedical purposes, agricultural uses, optoelectronics, sensors etc. [8-12]. The presence of rare-earth oxides assures desired optical properties [13].

The efect of crucible, melting conditions and melt mixing (time and speed) on glass homogeneity and on optical properties of some phosphate glasses, doped with rare-earth oxides in this work were studied. This first part presents the influence of crucible on glass bubbles and cords. The analizes were made by using UV–Vis spectroscopy, energy-dispersive X-ray spectroscopy microanalysis - EDAX and scanning electronic microscopy - SEM.

2. Experimental

Phosphate glasses doped with Tb and Eu trivalent ions have been obtained by a non-conventional wet preparation of raw materials fallowed by melting-quenching, using analytical grade reagents: Li_2CO_3, $BaCO_3$, Al_2O_3, La_2O_3, H_3PO_4, Tb_2O_3 and Eu_2O_3.

The raw materials composition for doped phosphate glasses corresponds to the following molar formula:

$56.66LiPO_3$ $28.33Al(PO_3)_3$ $10Ba(PO_3)_2$ $2La_2O_3$ 3 RE_2O_3 (RE= Tb, Eu).

There were prepared two doped samples coded 4 and 5, containing 3 mol.% Tb_2O_3 and Eu_2O_3, respectively, melted in Romanian alumina crucibles and two undoped samples coded 6 and 7, first melted in Romanian alumina crucible and the second in Alcoa crucible. Sample 8 has a similar composition to sample 5, but melted in Alcoa crucible.

The reagents were introduced in the necessary amount of H_3PO_4 solution under continuous stirring. The obtained mixture was heated and dried on an electrical heating plate until it was solidified. The dried raw material was introduced in alumina crucible and thermal treated in two steps. First step comprises the elimination of residual water and volatiles from room temperature to 700°C. This step was run with low rising temperature speed, of 100°C/h. The second program had higher rising temperature speed, of 250°C/h until 1200 or 1250°C. The glass samples were melted at 1200 - 1250°C for 12 to 14 hours programs and then annealed at 450°C for 6 h. The glass was melt in in alumina crucibles put in a Nabertherm type electrical furnace equipped with superkanthal heating elements.

The thermal treatments of melting and annealing are presented in Figures 1 and 2, respectively. Table 2 presents the stirring program.

Table 1

Methods for optimisation of glass homogeneity / *Metode de optimizare a omogenității sticlei*

Method/ *Metoda*	Characteristic/ *Caracteristica*	Improvement/ *Îmbunătățire*
construction of the melt system *construcția sistemului de topire*	modification of convection, outlet flow *modificarea convecției, curgerii*	formation of vortices *formare de vârtejuri*
bubbling *introducere de bule*	flow manipulation *manipulare curgere*	formation of vortices, residence time *formare de vârtejuri, timp de liniștire*
electric boosting *încălzire electrică*	direct electric heating via electrodes, Joule heat effect *încălzire electrică directă prin electrozi, efect Joule*	improvement of melting, formation of vortices *îmbunătățirea topirii, formare de vârtejuri*
mechanical stirring *agitare mecanică*	local flow manipulation *control local al curgerii*	chemical homogenisation *omogenizare chimică*
drainage *curățare sticlă*	elimination of polluted glass melt *eliminarea topiturii de sticlă murdară*	prevention of cat scratches and striae *prevenirea formării de striuri*

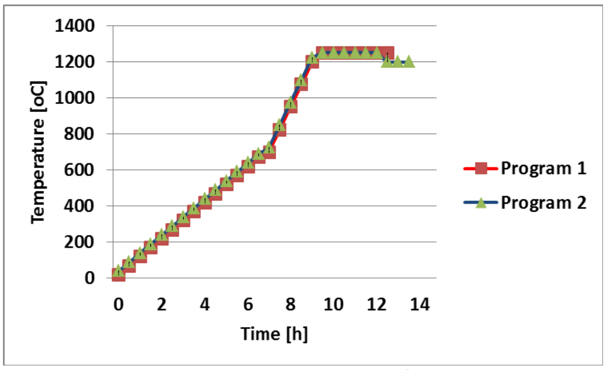


Fig. 1- Melting programs: program 1 used for sample 4 and 5 and program 2 used for samples 6 and 7 / *Programe de topire: programul 1, folosit pentru probele 4 şi 5 şi programul 2 folosit pentru probele 6 şi 7.*

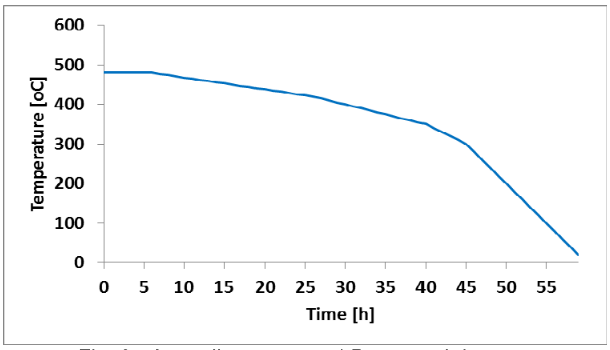


Fig. 2 - Annealing program / *Programul de recoacere.*

Table 2

Stirring program / *Programul de omogenizare*

Time [h] *Timp*	Temperature *Temperatura* [°C]	Rotation speed *Viteza de rotație* [rot/min]	Observations *Observații*
0	1250	100	Refining temperature
0.5	1250	200	
1	1250	500	
1.5	1250	500	
2	1250	350	
2.5	1250	200	
3	1250	150	
3.5	1200	100	
4	1200	100, stop	Moulding of glass

The optical quality of glass samples was low because of several bubbles and cords. In order to improve the glass quality it is necessary to use homogeneous raw materials, as we proceeded in our obtaining method. For the improvement of glass melt quality it is useful to homogenize it. Homogenization of melt can be done in several ways: Mechanical mixing using a ceramic stirrer; melting in helium atmosphere; inert gas bubbling; using electromagnetical field; using electrode temperature variation.

For the studied phosphate glasses we choosed to use the mechanical stirring, with an alumina stirrer. The rotation speed was varied between 100 and 500 rot/min. There were performed several experiments, first melting without mixing and next by using the mechanical stirring.

The glass samples were casted in graphite molds and then annealed in an electrical furnace with kanthal heating wire. The blocks were annealed at the high annealing temperature for minimum 4 hours in order to obtain improved optical best properties.

Glass samples were cut and optical polished in plates of 25x10x2 mm dimensions, using silicon carbide and cerium oxide.

In order to study of glass defects caused by the crucibles quality, the used crucibles were cut and studied by SEM-EDAX.

The light transmission, structure and homogeneity of the obtained glass were characterized by UV-Vis spectroscopy and SEM-EDAX analysis.

UV-Vis spectra were plotted with a spectrophotometer LAMBDA 950 UV/Vis/NIR type, with measurement range between 175 and 3300 nm, UV/Vis resolution of under 0.05 nm, and wavelength reproducibility of under 0.02 nm for UV/Vis.

SEM-EDAX investigations were made with a scanning electronic microscope QUANTA INSPECT F type, using a field emission gun - FEG with resolution of 1.2 nm and an X-ray energy dispersive spectrometer (EDS) with resolution at MnK of 133 eV.

3. Results and Discussion

The UV-Vis transmission curve of the obtained undoped glass sample coded 7 is presented in Figure 3.

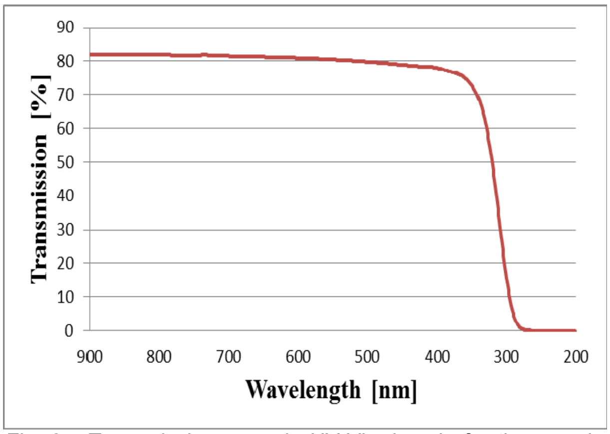


Fig. 3 - Transmission curve in UV-Vis domain for the sample coded 7 / *Curba de transmisie în domeniul UV-Vis, pentru proba 7.*

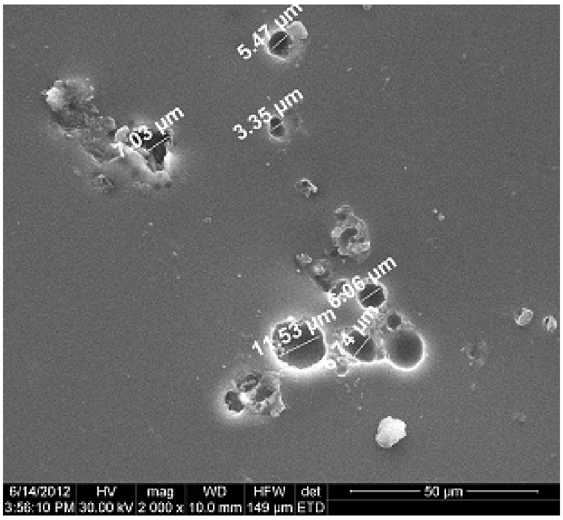


Fig. 4 - SEM photo of the glass sample coded 7 (2000x) / *Fotografia SEM a probei de sticlă cod 7.*

The SEM photo for the obtained coded 7 glass is presented in figure 4.

The obtained glass contains very small bubbles and micro-crystals, ranged in the dimension domain of 3 to 12 µm, as it can be seen from the figure 4. The image puts in evidence the crystallization tendency of the glass. No other defects such as threads or cords are to be seen. It appears that the stirring process had good effects consisting of the elimination of defects like threads and cords from the glass.

The micro-crystals are introduced most of them from the crucible wall but some of them can be provided by the apparition of some crystals in the glass. These crystals were identified as aluminium phosphite. In order to eliminate such defects the refining program was shortened at upper annealing temperature from 6 to 4 hours.

The bubbles may be due to the crucible quality or to the melting parameters. In order to study the crucible influence we used 2 kinds of crucible, one made from alumina supplied by ICEM Bucharest (coded I), and the other from Alcoa, Canada (coded II). The bubbles must be removed by using special methods, such as vacuum method, chemical methods, and gas bubbling method. The removal of bubbles will be presented in a future paper.

Table 3 presents the investigated samples regarding the type of used crucible, the glass dopant and the performing or not of the stirring program.

Table 3

The code of glass samples / *Cod probe de sticlă*

Sample code *Cod proba*	Crucible *Creuzet*	Doping *Dopant*	Stirring *Omogenizare*
4	ICEM type	Tb doped	No
5	ICEM type	Eu doped	No
6	ICEM type	Undoped	Yes
7	Alcoa type	Undoped	Yes
8	Alcoa type	Eu doped	Yes

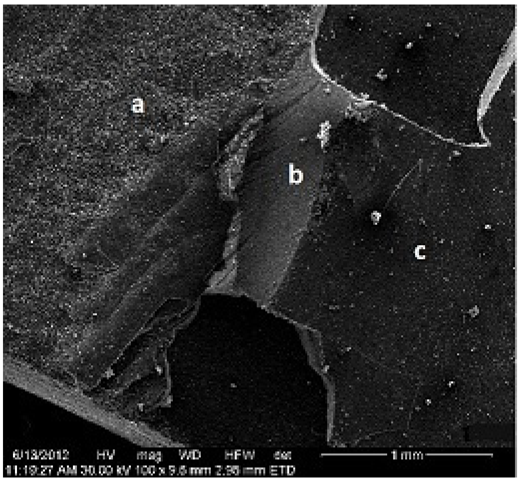
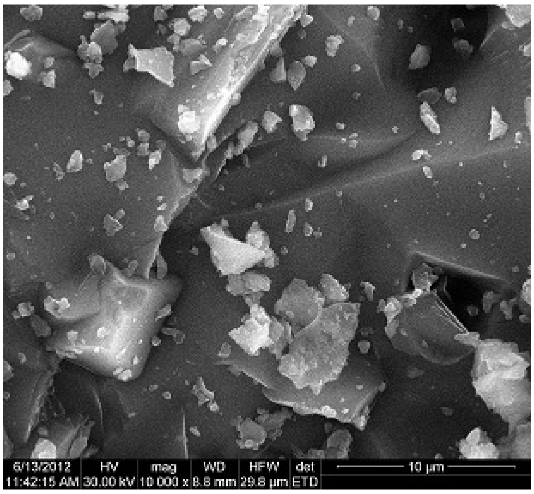
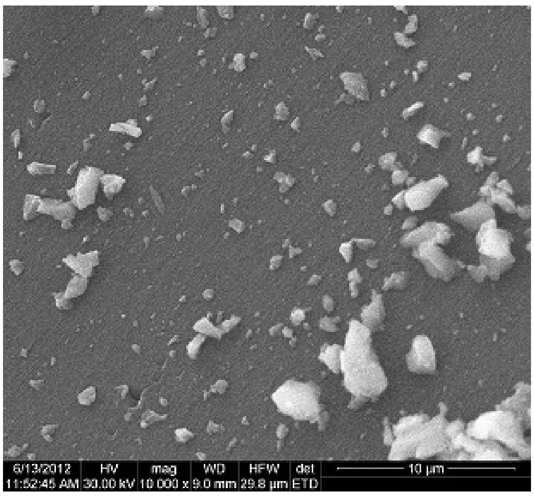


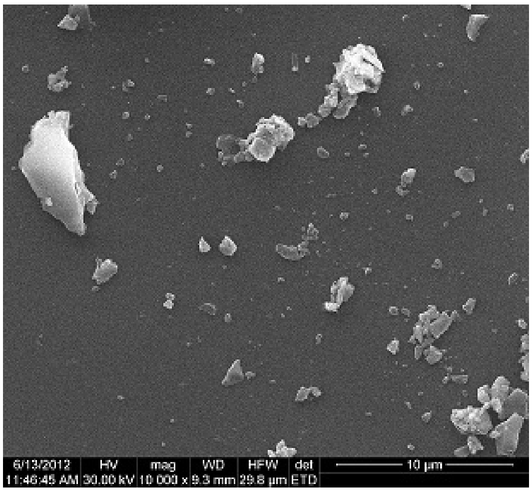
Fig. 5 - Crucible-glass contact zone for the sample 4: a. upper glass zone; b. glass surface zone; c. under glass surface zone; zoom 100x / *Zona de contact creuzet – sticlă pentru proba 4: a. Zona superioară sticlei, b. Zona suprafață sticlă, c. Zona de sub suprafața sticlei.*



5a. Upper glass zone of the sample 4 (10000x) / *Zona superioară sticlei proba 4.*



5b. Glass surface zone of the sample 4 (10000x) / *Zona suprafață sticlă, proba 4.*



5c. Under glass surface zone of the sample 4 (10000x) / *Zona de sub suprafața sticlei, proba 4.*

The SEM images of the used crucibles wall for the samples coded 4, 7 and 8 are presented in the Figures 5, 6 and 7, respectively.

In the case of the sample 4, the observed zones, one above the glass, one at the glass surface and the other under the glass exhibit different textures. The above-glass zone presents a very non-homogeneous mixture of crystals, crucible wall inclusions and thin glass layer with big pores. The under glass zone as well as the surface glass zone present many small crystals in the range of hundreds of nanometers to 8-10 microns dimensions, but the glass seems to be homogeneous and only few bubbles appear. The texture is almost similar with that in the case of surface glass zone where some cracks and holes appear.

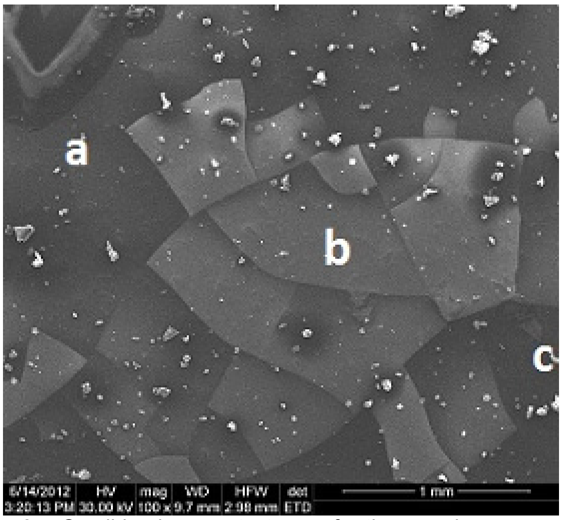
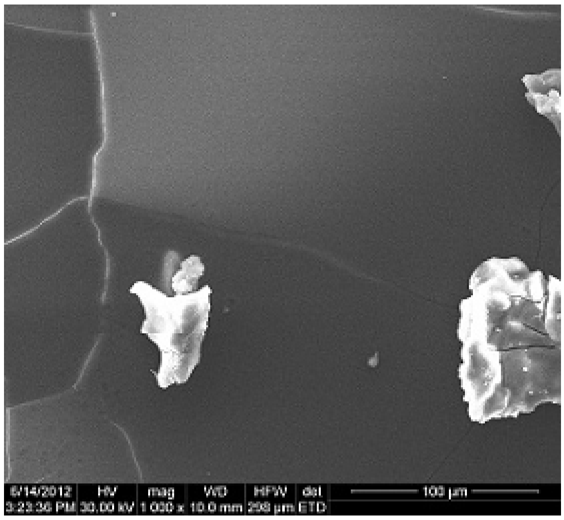
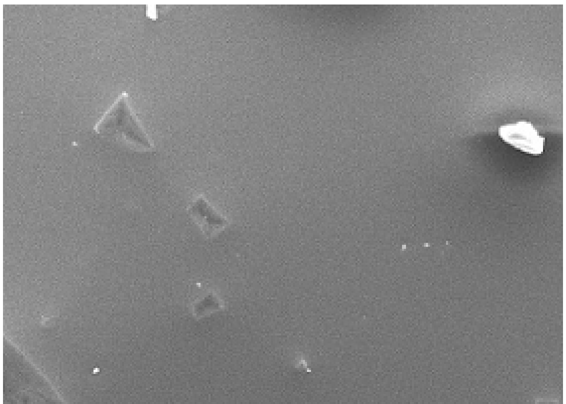


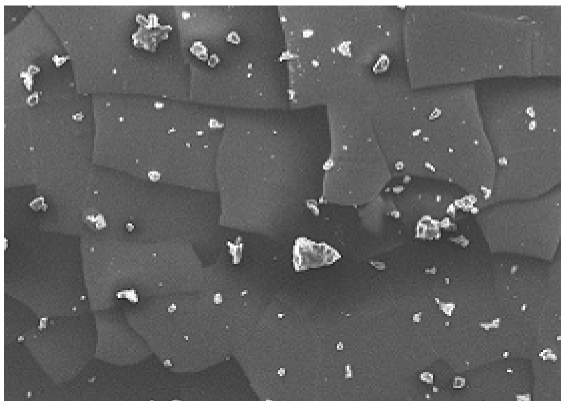
Fig. 6. - Crucible-glass contact zone for the sample 7: a. upper glass zone; *b. glass surface zone; c. under glass zone; zoom 100x / *Zona de contact creuzet – sticlă pentru proba 7: a. Zona superioară sticlei, b. Zona suprafață sticlă, c. Zona de sub suprafața sticlei.*



6a. Upper glass zone of the sample 7 (1000x) / *Zona superioară sticlei, proba 7.*



6b. Glass surface zone of the sample 7 (1000x) / *Zona suprafață sticlă, proba 7.*



6c. Under glass surface zone of the sample 7 (200x) / *Zona de sub suprafața sticlei, proba 7.*

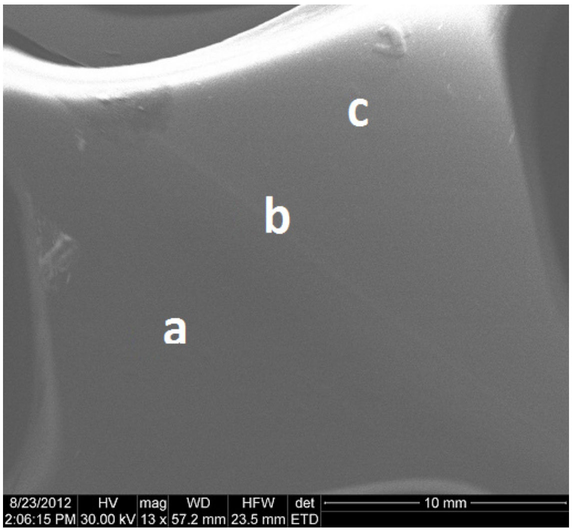
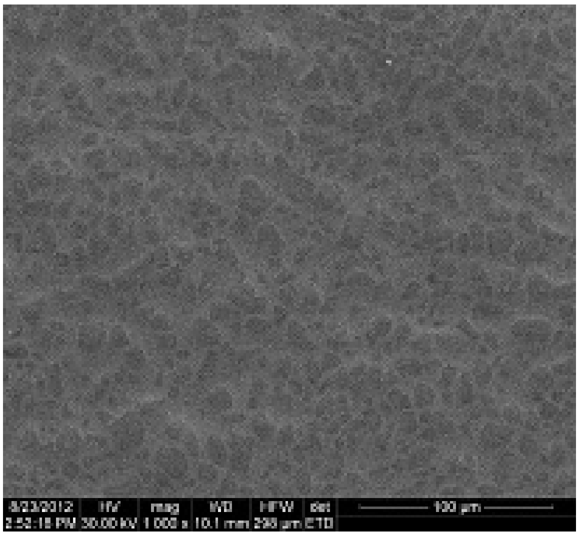
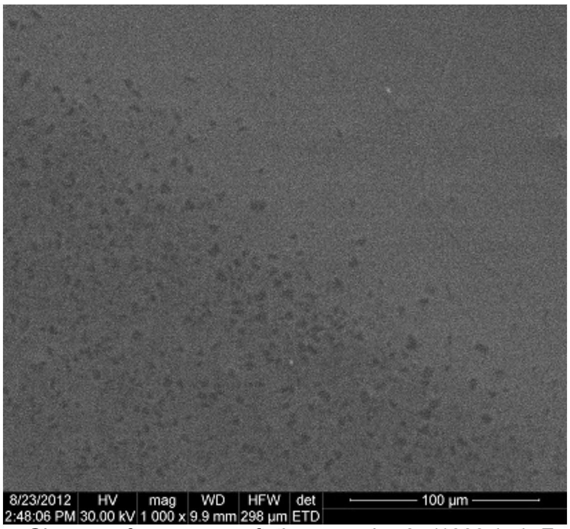


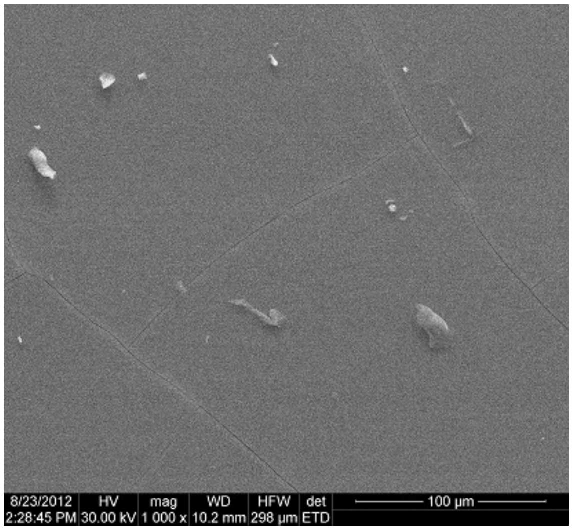
Fig. 7- Crucible-glass contact zone for the sample 8: a. upper glass zone; *b. glass surface zone; c. under glass zone; mag 13x / *Zona de contact creuzet – sticlă pentru proba 8: a. Zona superioară sticlei, b. Zona suprafață sticlă, c. Zona de sub suprafața sticlei.*



7a. Upper glass zone of the sample 8 (1000x) / *Zona superioară sticlei a probei 8.*



7b. Glass surface zone of the sample 8 (1000x) / *Zona suprafață sticlă a probei 8.*



7c. Under glass surface zone of the sample 8 (1000x) / *Zona de sub suprafața sticlei a probei 8.*

The sample 7 exhibits fewer crystals than the sample 4 but their dimensions are larger, up to 100 microns in all three zones. In this case the glass seems to be more homogeneous than in the case of the sample 4 and the number of bubbles or holes is decreased. This is certainly due to the stirring process. The fact that the crystals dimensions are bigger in this case is in accordance with the assumption that these crystals are due to the crucible wall rather than to the in-glass crystallization.

The sample 8 presents a less number and smaller dimensions of crystals than the sample 7 and a more homogeneous texture. The crystals dimensions are also variable from units up to tens of microns. Big pores or bubbles are not seen. The better quality of the glass in this case is due to the improvement of stirring process, according to the viscosity of glass. It is clear from the images of the samples 7 and 8 compared to those of the sample 4 that the Alcoa crucible is more resistant to glass attack than the ICEM type crucible and that is why it introduces in glass less crystals but of bigger size.

The EDAX results for the glass coded 7 and for the crucibles used to obtain glasses coded 4, 7 and 8 are presented in the Figures 8 to 11, respectively.

EDAX analysis identified all the elements introduced in the glasses, for all three samples coded 4, 7 and 8, in the glass film at the crucible wall. In the case of the sample 7, the maximum for aluminum content is smaller than in the case of the sample 4. This can signifies that the Alcoa-type crucible was more resistant to glass attack than the ICEM one, which was more dissolved in the glass film adherent to the crucible wall. The mapping shows that the homogeneity of glasses is

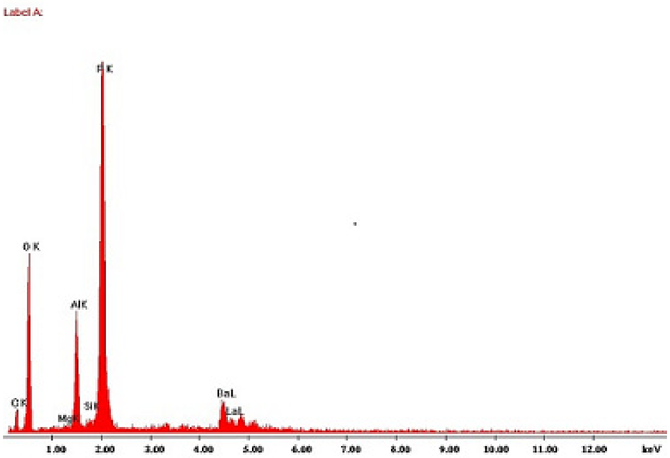


Fig. 8 - EDAX results for the glass sample coded 7 / *Rezultatele EDAX pentru proba de sticlă cod 7.*

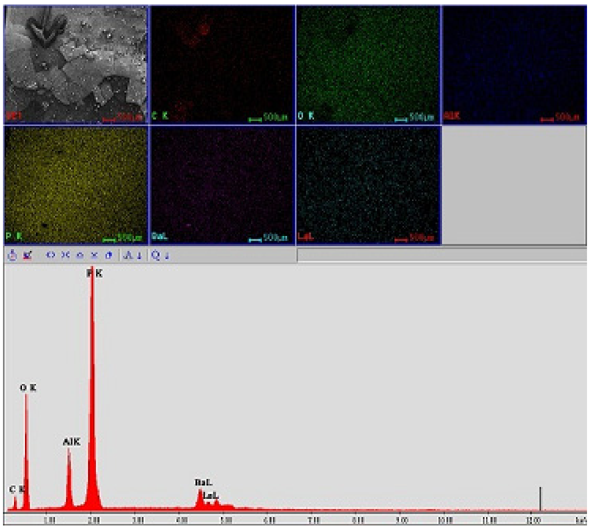


Fig. 10 - EDAX results for the sample coded 7 / *Rezultatele EDAX pentru proba 7.*

satisfactory and proves that the preparation method was well chosen. The zones where the glass was separated from the crucible wall appear in the map, in which the predominant element is aluminum, as expected, since the crucible is made from Al_2O_3.

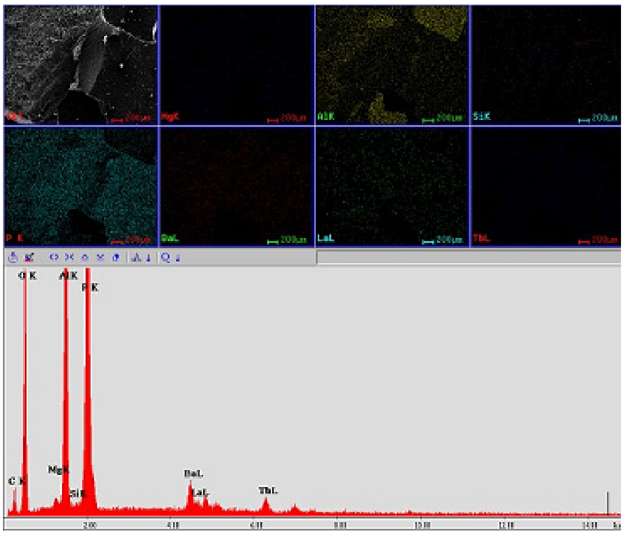


Fig. 9 - EDAX results for the sample coded 4 / *Rezultatele EDAX pentru proba 4.*

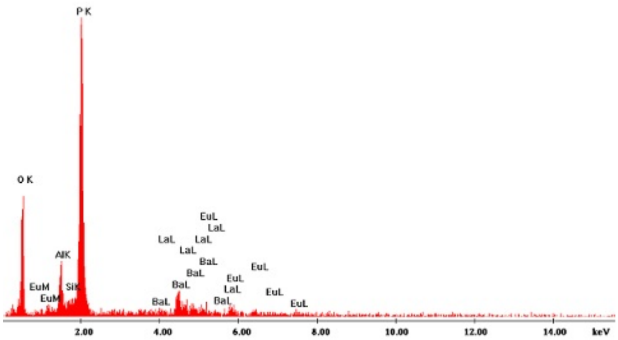


Fig. 11 - EDAX results for the sample coded 8 / *Rezultatele EDAX pentru proba 8.*

A semi quantitative analysis of the composition of the glasses coded 4, 7 and 8 was made by EDAX (Energy dispersive of X-ray spectroscopy analysis) method and the results of measured elemental composition and calculated equivalent oxide composition are presented in the Table 4.

As lithium can't be measured by this method, we calculated the oxide composition assuming that all the samples contain about 4 wt. % of Li_2O (which means about 8% volatilization loss). The obtained results are close enough to the raw materials composition. The phosphorus oxide

Table 4

Elementary and oxide composition of samples 4, 7 and 8 from EDAX data
Compoziția elementară şi oxidică a probelor 4, 7 şi 8 din datele EDAX

No. *Nr.*	Element	Wt.% in sample 4 *% grav în proba 4*	Wt.% in sample 7 *% grav în proba 7*	Wt.% in sample 8 *% grav în proba 8*	Oxide *Oxid*	Wt.% in sample 4 % grav în proba 4	Wt.% in sample 7 *% grav în proba 7*	Wt.% in sample 8 *% grav în proba 8*
1	P	27.48	31.94	34.61	P_2O_5	68.50	71.61	67.39
2	Ba	6.31	8.19	9.25	BaO	7.66	8.95	8.78
3	Al	6.24	6.18	6.19	Al_2O_3	12.83	11.43	9.94
4	La	1.34	3.5	4.86	La_2O_3	1.71	4.01	4.84
5	Tb	4.23	-	-	Tb_2O_3	5.3	-	-
6	Eu	-	-	5.13	Eu_2O_3	-	-	5.05
7	O	37.15	40.34	38.18				

results, as expected, have smaller value, due to the volatilization loss of above 8%.

The SEM-EDAX analysis of the crucible-melt contact zone established the role of the crucible type, the degree of corrosion, and the presence of micro pores that can cause defects in the form of seeds and bubbles.

There were studied the effects of duration and speed of rotation on the optical quality of obtained glasses. In the case of the glass samples where the stirring was applied, the seeds and cords in glass are not visible with naked-eye. There exist zones in the glasses where no cords can be observed, even at 5x magnification.

4. Conclusions

Alumino-phosphate glasses containing rare-earth oxides were made by a wet route of raw materials preparation and by melting at 1250^{o}C in electric furnace.

The undoped glass exhibit good transmission, of above 90% in visible domain.

The glass quality depends on melting, stirring and annealing programs: the optimum melting time at 1250^{o}C was 4 hours, the annealing time 4 hours and the stirring with a variable speed, depending on glass viscosity, between 100 and 500 rot/min. The Alcoa crucible gives a glass with fewer defects (bubbles, inclusions, crystals, seeds) than the ICEM one, but there was a bubble agglomeration in the glass in this case.

Acknowledgments

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