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Transnational Call 2012

7-081/2013 M-ERA.NET project
New doped boro-phosphate vitreous materials, as nano-powders and nano-structured thin films, with high optical and magnetic properties, for photonics
MAGPHOGLAS

PRESENTATION BROCHURE

24.11.2016

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New doped boro-phosphate vitreous materials, as nano-powders and nano-structured thin films, with high optical and magnetic properties, for photonics
MAGPHOGLAS

Consortium members

Organisation	Activity type[1]	Contact Person (Name & email)	Country/ Region	Applying for funding
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The general objectives of this project are:

i) The design and development of vitreous BPG nano-materials by the sol-gel method, coprecipitation and coacervate techniques; (ii) Nano-structured powders and thin layers obtained by RFMS deposition, from BPG, at lower temperatures; (iii) High field (G-TW) and very short pulse (fs) laser irradiation on nano-structured BPG coatings for the preparation of meta-materials; (iv) Complex vitreous materials with negative refractive index in bulk; (v) Design and development of nano-structured thin films using RFMS and PLD/Ag-thin films on nano-structured BPG surface with negative refractive index; (vi) Modeling of nano-structure in nano-structured BPG; (vii) Structural and morphological characterization of the obtained materials and correlation with desired properties; (viii) Design and manufacturing of new Faraday rotator/ ultrafast opto-magnetic switch prototype using these materials with functional surfaces.

The novelty of the project consists of: (i) eco-design of sol-gel nanotechnology to obtain doped BPG; (ii) development of methods for using wastes and subproducts; (iii) design and modeling of inter-dependence structure/properties with process parameters; (iv) RFMS deposition of thin films in doped boro-phosphate systems; (v) high field and very short pulse laser nanostructured BPG films; (vi) meta-boro-phosphate glass materials design and development; (vii) modeling and design of negative refractive index BPG doped films; (viii) design and manufacturing of new Faraday rotator/ ultrafast opto-magnetic switch prototype based on BPG.

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[1] **HE – Higher Education, RES – Research, IND – Industry, SME, OTH - Others**

WP no.	Work Package Title	Main content (keyword)	Total effort (Person-months)	Work package leader	Participating Project Partners
1.	Design, modeling and development of sol-gel and co-precipitated boro-phosphate materials as powder and coatings: Thin nano-structured boro-phosphate glass films by sol-gel method using spin-coating method; Measurement of properties for nano-structured boro-phosphate glass powders and thin films. Correlation structure-properties	Sol-gel; Boro-phosphate glass thin coatings; Spin-coating; Nano-structured materials; Physico-chemical properties; Structure	24	CO	P3
2.	RFMS deposition of nanostructured doped boro-phosphate glass thin films: Development of doped nano-structured boro-phosphate glass thin films by RFMS; Thermal, structural, magnetic and optical properties measurements for the obtained thin films	Radio Frequency Magnetron-Sputtering; Nano-structured thin films; Structure; Thermal, magnetic and optical properties;	22	CO	P3
3.	PLD at high field (G-TW) and very short pulse (fs)(PLD-G-TW-fs) of nano-structured boro-phosphate glass thin films with functional surfaces: Properties measurements for the obtained thin films	Pulsed Laser Deposition; High field and very short pulse; Boro-phosphate thin films; Functional surfaces; Properties	20	CO	P3
4.	Modeling and design of meta-materials and negative refractive index nano-structured boro-phosphate glassy materials; Properties measurements for the obtained materials; dissemination	Meta-materials; Negative refraction index; Properties; Dissemination	18	P3	CO
5.	Patent elaboration related to the preparation method of new powders and thin films from vitreous materials	Patent deposition	5	CO	P2, P3
6.	Functionality demonstration for the obtained powders and thin films	Demonstration of functionality	5	P2	CO, P3
7	Design and prototype manufacturing of Faraday rotator /ultrafast opto-magnetic	Design of Faraday rotator /ultrafast opto-magnetic switch prototype	30	P2	CO, P3

	switch based on BPG	Modeling and simulation of opto-magnetic parts. Manufacturing of Faraday rotator /ultrafast opto-magnetic switch prototype			

Bulk glasses, powders and thin films were obtained by melting-quenching with wet raw materials preparation, sol-gel, magnetron sputtering - MS and pulsed laser deposition - PLD methods from Bi-Pb and Dy-Tb oxides co-doped boro-phosphate systems. The influence of precursors, pH, temperature, time, on the sol-gel process, and also the MS and PLD process parameters influence on films properties were investigated. The structure and properties of the obtained samples were analyzed by using Raman, FTIR and UV-Vis spectroscopy, thermal analysis and mechanical behavior at nano- and micro - scale under concentrated load action. The structure - properties correlation was studied by using XRD, XRF, SEM-EDS, and AFM measurements. The magneto- optical properties were analyzed using a modified Kerr device.

I. Laboratory methods, vitreous materials obtaining and materials characterization - INFLPR

Bulk glasses obtaining and characterization

The vitreous matrix comprises network formers meaning phosphorus and boron oxide, together with modifiers and stabilizers like Li_2O and ZnO. For the opto-electronic and magnetic properties PbO, Bi_2O_3, CoO, Dy and Tb dopants were added – Table 1. The boro - phosphate glasses containing dopants in 1-3 molar % amount were prepared using p.a. reagents by melting in electric furnace at 1200-1250 ^oC for 2-4 hours.

Table 1. Oxidic composition (wt and mol. %) of studied glasses

Proba	**Oxid**	**B_2O_3**	**P_2O_5**	**Li_2O**	**Al_2O_3**	**ZnO**	**Dy_2O_3**	**Tb_2O_3**	**Bi_2O_3**	**PbO**	**CoO**	**Total**
BPM1	% molar	35	35	10	9	5	0	0	3	3	0	100
	% gravimetric	21.96	44.77	2.69	8.27	3.67	0	0	12.60	6.03	0	100
BPM2	% molar	20	50	10	9	5	0	0	3	3	0	100
	% gravimetric	11.43	58.27	2.45	7.53	3.34	0	0	11.48	5.50	0	100
BPM3	% molar	20.62	51.55	10.31	9.28	5.15	0	0	0	0	3.09	100
	% gravimetric	13.47	68.65	2.89	8.88	3.94	0	0	0	0	2.17	100
BPM4	% molar	50	20	10	9	5	0	0	3	3	0	100
	% gravimetric	34.77	28.36	2.98	9.17	4.06	0	0	13.96	6.69	0	100
BPM5	% molar adus la 100	21.17	52.91	10.58	9.52	5.29	0	0	0	0	0.53	100
	% gravimetric	13.72	69.92	2.94	9.04	4.01	0	0	0	0	0.37	100
BPM6	% molar	20	50	10	9	5	3	3	0	0	0	100
	% gravimetric	11.29	57.56	2.42	7.44	3.30	9.08	8.90	0	0	0	100
BPM7	% molar	19.42	48.54	9.71	8.74	4.85	4.37	4.37	0	0	0	100
	% gravimetric	10.36	52.82	2.22	6.83	3.03	12.49	12.25	0	0	0	100

The bulk materials obtaining method *includes the following steps*:

Wet raw materials preparation

The technological process of preparing the mixture of raw materials by wet method, used in BPM glass obtaining comprises next operations: Gravimetric and volumetric dosing of the raw materials; Introduction in predetermined order of raw materials in the homogenization vessel; Cold homogenization of raw materials, mechanically for 15 minutes; Partial drying of the mixture of raw materials, with partial elimination of water (in quartz crucibles with electrical mixing, with flexible shaft, on the electric stove) under constant stirring rate, until the temperature of 120-150oC is reached; Pouring the mixture while it is still fluid, into the melting high alumina crucible.

The melting of glass

The crucible with the mixture of raw materials was introduced into electrical furnaces equiped with SiC rods and with $MoSi_2$ elements, for the implementation of the program of themelting-forming-conditioning-refining-cooling of the rare-earth-doped phosphate glasses (Fig. 1a).

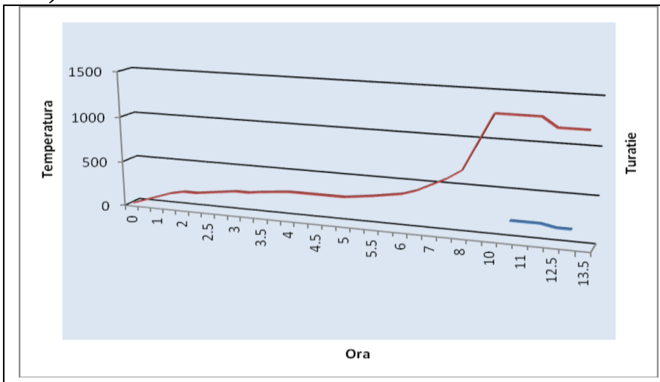


Fig. 1a) Melting program

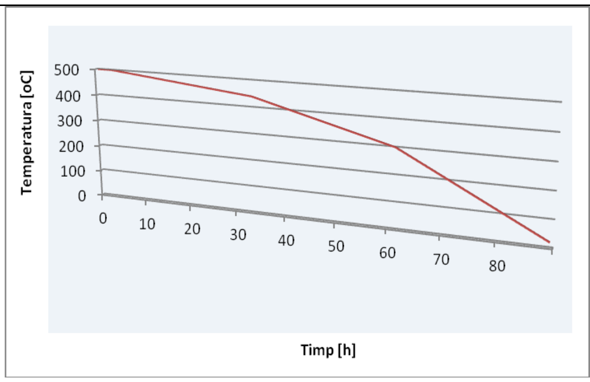


Fig. 1b) Annealing program

The rotation speed of the stirrer was set-up between 100-500 rpm, in dependency on melt viscosity, that in its turn depends on temperature. The first experiments were effectuated out without stirring, and then with continous stirring of the melt.

The annealing of the glass samples was made in an oven equipped with kanthal heating wires as presented in Fig 1 b)

The characteristic temperatures and thermal expansion coefficients obtained with the differential dilatometer are presented in Table 2.

Table 2. Thermal expansion coefficients and characteristic temperatures of BPM glasses

Glass	Thermal expansion coefficient (α_{20}^{300}x10^{-7}/K)	Straining point (°C)	Vitreous transition temperature (°C)	Annealing temperature (°C)	Softening dilatometric temperature (°C)
BPM2	8.12x10^{-6}	413	466.6	483.5	511
BPM3	8.63x10^{-6}	433	498.5	517	541.3

The optical transmission and structure of obtained materials was investigated by using UV-Vis-NIR, FTIR and Raman spectroscopy – Fig. 2 for BPM2 glass. The vitreous network is specific to metaphosphates, as proved by symmetric and asymmetric stretching vibrations of P-O-P bonds in Q2 and Q1 units, at 770 and 870-890 cm-1 and of PO_3^{2-} Q1 units, at 1030 and 1220 cm^{-1} respectively. Vibration of -O-BO_4-O_3B links can be identified at 770 cm^{-1} and asymmetric vibration links of O_3B-O-B-O in triangular borate units (BO_3 and BO_2O^-) from pyro and ortoborate groups at 1030 cm^{-1}

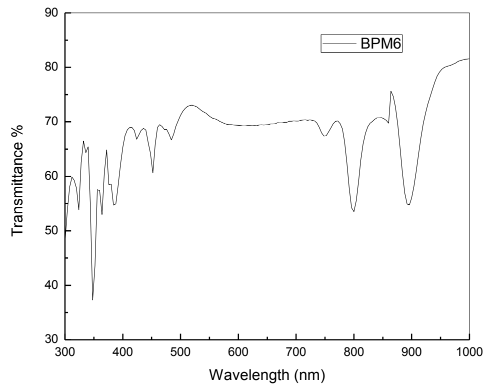
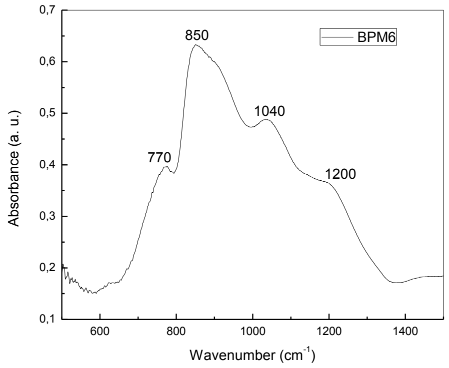
	

Fig. 2a). Curba de transmisie UV-Vis pentru proba BPM6	Fig. 2b). Curba de absorbție FTIR pentru proba BPM6

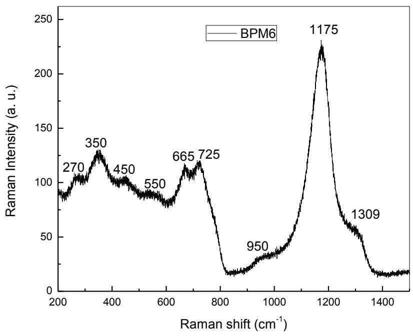
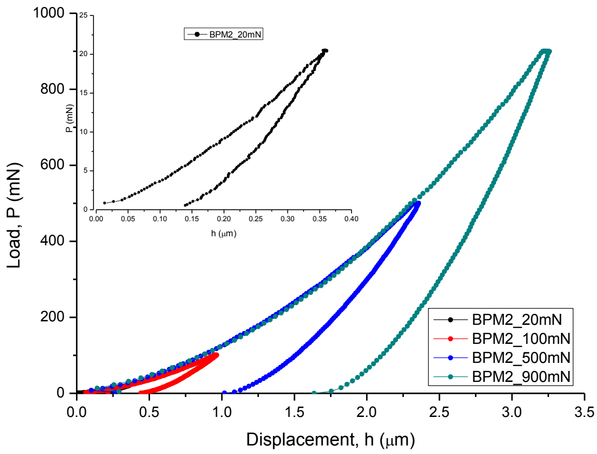


Fig. 2c). Spectrul Raman pentru proba BPM6

Mechanical properties investigation by means of indentation method

The typical nanoindentation curves showing the load-penetration (P-h) dependences for each of the four used loads (20, 100, 500, 900 mN) are presented in Fig. 3 (a). The aspect of obtained imprints visualized in optical microscope is shown in Fig. 3 (b).

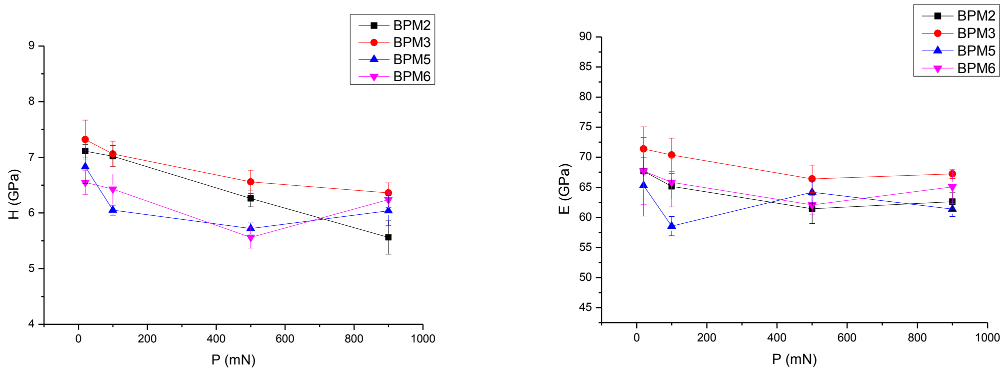


a)

b)

Fig. 3. Curves 'loading and unloading' ($P(h)$) obtained at different maximum loads for glass sample BPM2, the graph included shows an enlarged image of the curve $P(h)$ for P = 20 mN (a) and the aspect for fingerprints for P_{max}=100, 500 şi 900 mN (b)

Fig. 4 presents the dependencies of hardness (*H*) and Young's modulus (*E*) to the load value. For each of the loads were performed 5 tests (indentations) and the values of *H* and *E* were calculated as averages of these measurements.



a) b)

Fig. 4. The dependence of hardness (H) (a) and Young's modulus (E) (b) upon the value of the load for the BPM2 glass sample

To measure the coefficient of cracking resistance at indentation (K_{IC}) they were submitted and measured at least every five imprints, and the result is the average of these five measurements. Results of the measurements of K_{IC} are shown in Fig. 5.

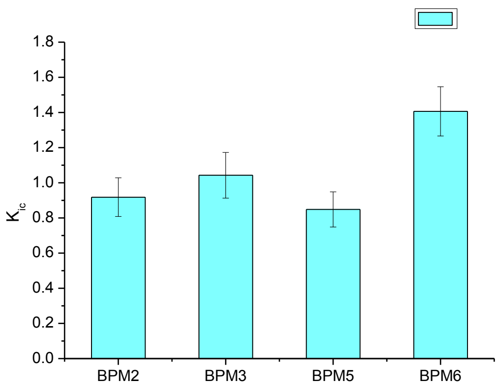


Fig. 5. The cracking resistance for sample BPM2

Generally it can be concluded that the boro-phosphate glasses have improved mechanical properties compared to the alumino-phosphate (SAP) ones. The NI and MI results showed that the doped BPM glass with a higher hardness (H) have also a higher cracking resistance (K_{IC}) higher as compared with SAP, which is an important feature for oxide glasses, usually characterized by an increased brittleness for the materials with a higher hardness.

Thin films from BPM glass targets deposited by Magnetron sputtering

Thin films from doped boro-phosphate glasses were obtained by magnetron sputtering deposition on different substrates, borosilicate and boro-phosphate glass, silicon, and quartz. We used a VARIAN ER3119 installation with the evaporation rate of 0.1 Å/s ÷ 10 Å/s and accurateness to control the thin film thickness of ± 1 nm.

MS parameters for depositions from BPM2 target experiments were: Quartz constant: 8.25; Target density (BPM2MS): 2.752 g/cm3; Ar pressure 6.5x10 -4 torr; Active power 136 W; Reactive power 0 W; intensity 0.2 A; Substrate: borosilicate glass with Ag deposition of 16 nm on one face; Deposition speed 0.7-1.1 Å/s; Deposition time: 2h 46 min; Thickness of deposited film: 10220 Å.

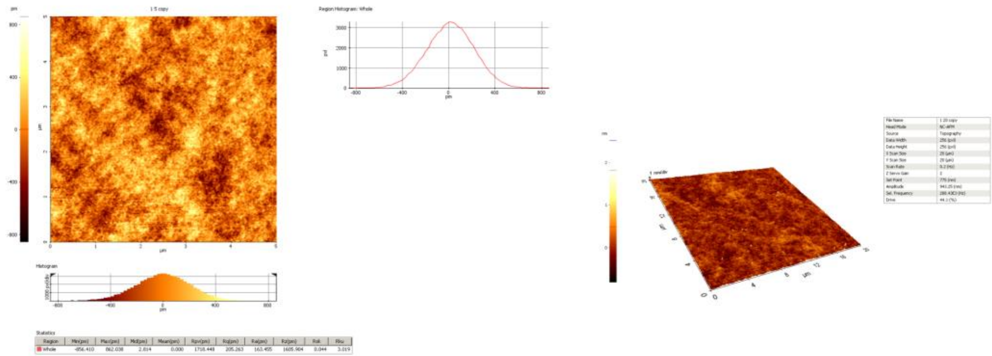


Fig. 6. AFM for MS deposited films from BPM2 deposited on quartz

The target noted BPM 6 is made from a boro-phosphate glass co doped with Dy and Tb, obtained by melting - quenching technology. Several experiments for MS deposition of thin films were made on borosilicate glass - BS and on Si, with the working parameters presented in table 3 and AFM investigation in Fig. 7.

Tabel 3. Deposition parameters for MS deposition from BPM6 target

Experiment	1	2	3	4
Target	Ag, BPM6 /BPM6	BPM6	BPM6 +Ag	BPM6 +Ag
Quartz constant	8.83	8.83	8.83	8.83
Pressure Ar [torr]	6.5 x10 -4	4.6 x10 -4	4.6 x10 -4	5.4 x10 -4
Active power [W]	100-150/61	150	30/150/3/150	150/31/150/30
Reactive power [W]	0-2/4	2-3	2/0/1/0	3/0/3/0
Curent intensity [A]	0.2	0.2	0.2	0.2/0.1/0.2/0.1
Substrate	BS/Si	BS/Si	BS	Si
Deposition rate [Å/s]	1-1.3	1.2-1.8	3 and 0.3 Ag and 1.2-1.8 BPM6	1.2-1.5 BPM6 and 1.2-1.3 Ag
Deposition time	1h 2 min/1h 17 min	1h 5 min	3h 55 min	2h 30 min.
Thickness of film [Å]	5400/5224	5000 over exp.1	1005Ag+5400BPM6+100 Ag+5400BPM6+100Ag	5352 BPM6 + 156 Ag + 5368 BPM6 + 104 Ag

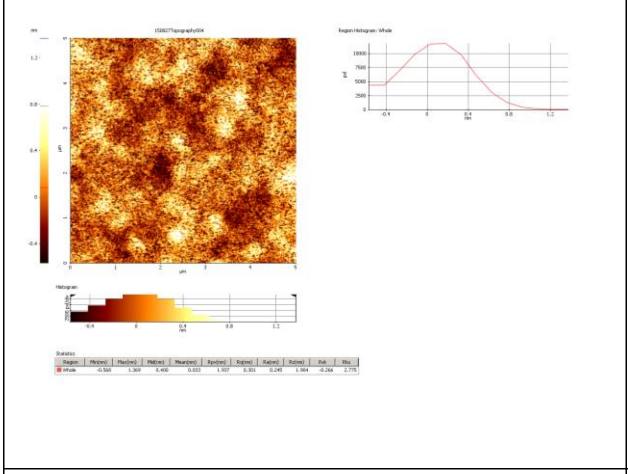


Fig. 7a) AFM of films obtained by MS on BS

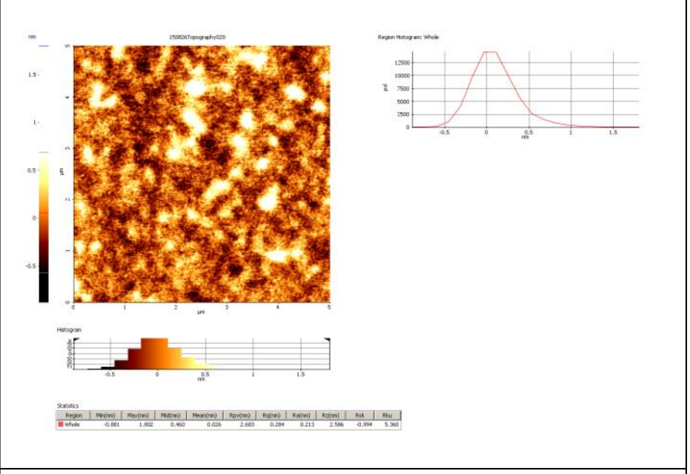


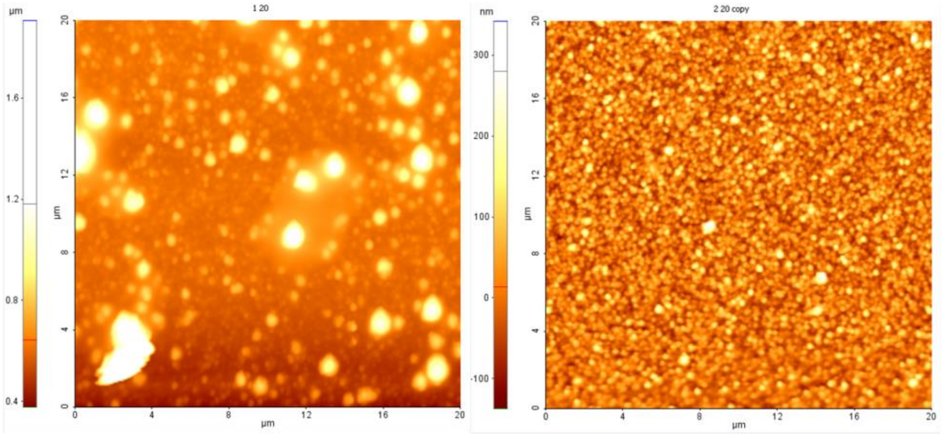
Fig. 7b) AFM of films obtained by MS on Si

It was identified that the films have the same composition as the glass target. Films with 0.5-1 microns thickness were obtained by magnetron sputtering deposition from boro-pohosphate glass target. The films were smooth as the AFM showed out.

Thin films from BPM glass targets deposited by Pulsed Laser Deposition

Table 4. PLD depositions from BPM2 targets

Parameter	**1**	**2**	**3**	**4**
Substrate	Quartz glass thick	Quartz glass thick	Quartz glass thin	Quartz glass thin
Deposition	BPM2 boro-phosphate glass	BPM2 boro-phosphate glass	BPM2 boro-phosphate glass	BPM2 boro-phosphate glass
Temperature substrate at deposition	400°C	400°C	600°C	600°C
Pulses no	12500	25000	12500	25000
Fluence F	2.7 J/cm^2	2.7 J/cm^2	2.7 J/cm^2	2.7 J/cm^2
Distance target to substrate	8 cm	8 cm	8 cm	8 cm
Atmosphere	10^{-4} mbar O_2	10^{-4} mbar O_2	10^{-4} mbar O_2	10^{-4} mbar O_2



a) 400°C b) 600°C

Fig. 8. AFM images of BPM2 thin film deposited by PLD on silica glass

AFM investigations – Fig. 7 showed that the substrate temperature influence the thickness of the PLD thin films of BPM2. Using the AFM, we measured how thick the thin film deposited by PLD was: at 400°C, the thickness is almost double as the one at 600°C.

The roughness is affected by the number of pulses. The roughness increases from 60 nm to 150 nm at 400°C, respectively from 20 nm to 50 nm at 600°C when the number of pulses increases from 12.500 to 25.000.

The magneto-optical Faraday effect

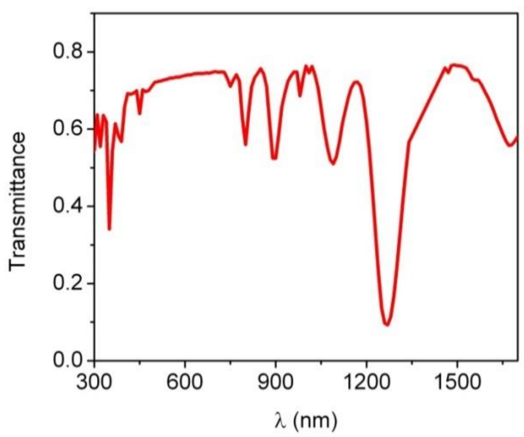


Fig. 9a) UV-VIS transmission curve for BPM6 sample

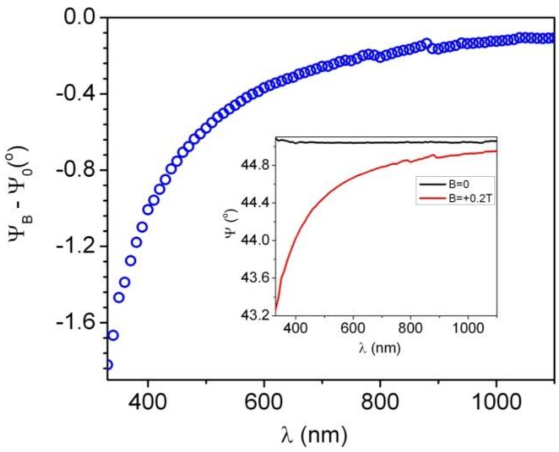


Fig. 9b) Rotation angles of polarised plane under influence of 0.2 T magnetic field, with wave length (ellipsometry) for BPM6 sample

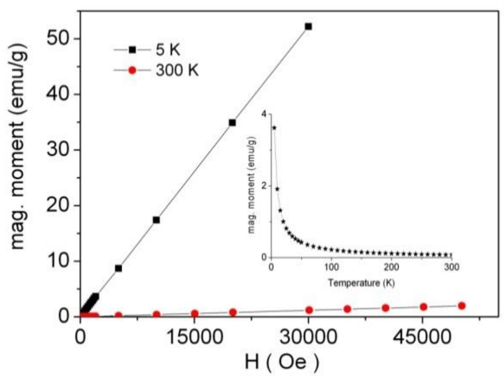


Fig. 9c) M(H) at 5 K and 300 K for BPM6 sample. In the insert is presented evolution of magnetization with temperature, under 2000 Oe magnetic field

At wavelength of 600 nm, Verdet constant for BPM6 samples is about 0,06 min/mm/Oe, and a rotation of 5 min with a glass of 1 cm length is obtained in a field of 90-100 Oe. BPM6 sample depass 3 times the BMP2 sample.

M(H) plot and magnetization values from M(T) plot, indicate a magnetic susceptibility of 1,8*10-3 emu/g/Oe at 5 K and of 3,9*10-5 emu/g/Oe at T=300 K respectively (30 times the values for BMP3 cobalt sample). The behavior is tipically paramagnetic

Thin films deposited by sol-gel - spin coating technique

Table 5. Sol-gel doped thin films and powders composition

Compound Sample code	B2O3 g	TEP mL	DyCl3 6H2O g	TbCl3 6H2O g	C2H5OH mL	NH4OH 25% mL	HCl 1N mL	H2O mL	pH
BPMG2d1	0.36	7.1	1.72	1.71	8.56	-	-	2.2	2
BPMG2d2	0.36	7.1	1.72	1.71	8.56	-	1	2.2	1.5
BPMG2d3	0.36	7.1	1.72	1.71	8.56	1	-	2.2	3
BPMG2d4	0.36	7.1	1.72	1.71	8.56	0.5	-	2.2	2.5

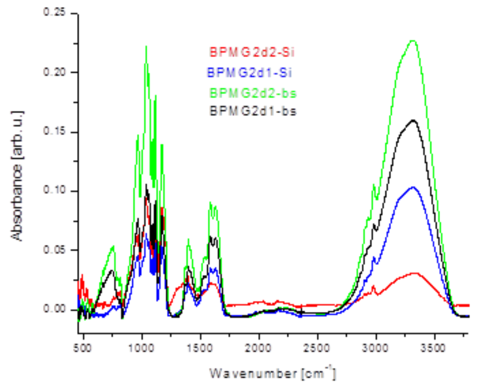


Fig. 10a) FTIR spectra of sol-gel thin films on Si and borosilicate glass - BS, dried at 120°C

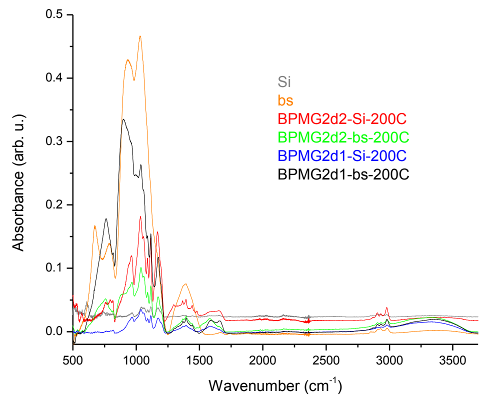


Fig. 10b)FTIR spectra of sol-gel thin films on Si and BS, treated at 200°C and of substrates

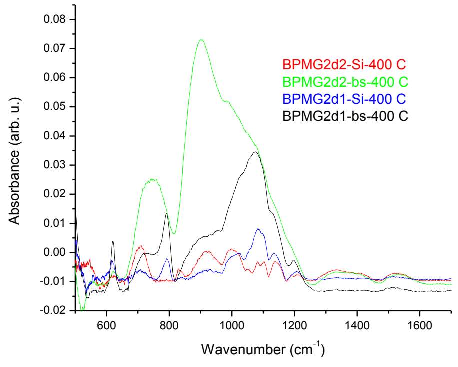


Fig. 10c) FTIR spectra of sol-gel thin films on Si and BS, treated at 400°C

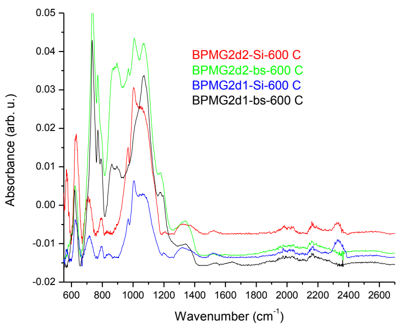


Fig. 10d) FTIR spectra of sol-gel thin films on Si and BS, treated at 600°C

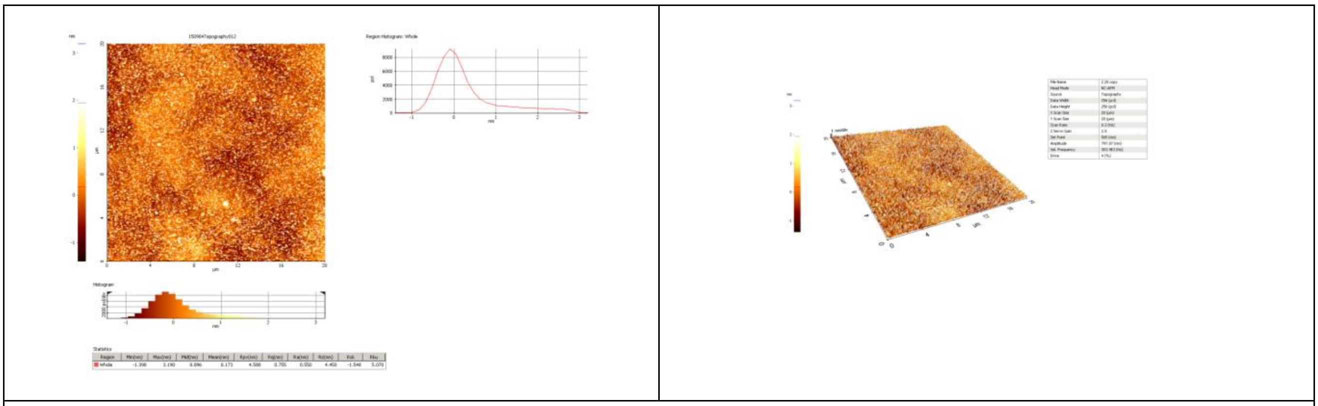


Fig. 11. AFM measurements for the BPMG 2d2 sol-gel sample on Si

Magnetic properties of sol-gel films

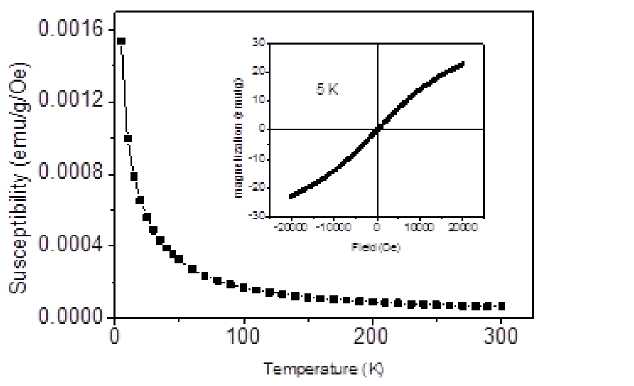


Fig. 12 a) Magnetic susceptibility versus temperature as obtained on sample BPMG 2d1.

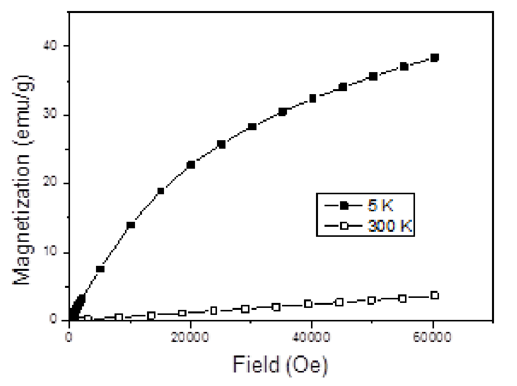


Fig. 12b) Magnetization versus applied field as obtained on sample BPMG 2d1, at 5 K and 300 K

The above results support a paramagnetic behavior up to the lower measuring temperature of 5 K , with a quite consistent magnetization of the 2d1 sample. In the case of 2d2 sample the magnetic susceptibility is four times higher comparatively to 2d1 sample, and the saturation magnetization is close to half of magnetite one.

II. Characterization of the obtained materials – CENIMAT - Portugal

A. Characterization of bulk glasses

i) Chemical composition (XRF); ii) Thermal characterization (DSC); iii) Structural characterization (XRD); iv) Microstructural characterization (SEM/EDS)

Table 6. Chemical compositions

	BPM2 wt%		BPM3 wt%	
Compound	*nominal	XRF	*nominal	XRF
Li_2O	2.45	2.45	2.89	2.89
Al_2O_3	7.53	8.77	8.88	10.3
P_2O_5	58.27	58.7	68.70	66.5
ZnO	3.34	2.92	3.93	2.14
B_2O_3	11.43	11.4	13.48	13.4
CoO			2.10	2.13
SiO_2		0.265		2.30
Bi_2O_3	11.48	5.03		
PbO	5.49	10.9		
K_2O				0.206
CaO				0.0522
Fe_2O_3				0.0757
NiO				0.0341
TiO_2				0.0245

Compositions determined by XRF were close to the nominal composition. Unexpected presence of SiO_2 was detected (BPM2: 0.265 wt% SiO_2 and BPM3: 2.30 wt% SiO_2). Source of SiO_2 probably attributed to refractory from the furnace.

The glass density was determined by Arhimede method – Table 7.

Table 7. Densities of BPM glasses

	BPM2	**BPM3**	**BPM5**	**BPM6**
Density (g/cm3)	**2.94**	**2.61**	**2.59**	**2.87**

BPM2 has the highest density because Pb and Bi are heavier than the dopants Dy and Tb presented in BPM6. The lightest glasses are BPM3 and BPM5 doped with Co (3 and 0.5 mol%, respectively).

Thermal characterization of the glasses by DSC

Thermal behavior of the glasses showed that : Glasses are thermally stable up to 450 ºC; Glass crystallization can be observed above 500 ºC; BPM6 glass shows a comparatively lower tendency to crystallize than the other glass compositions

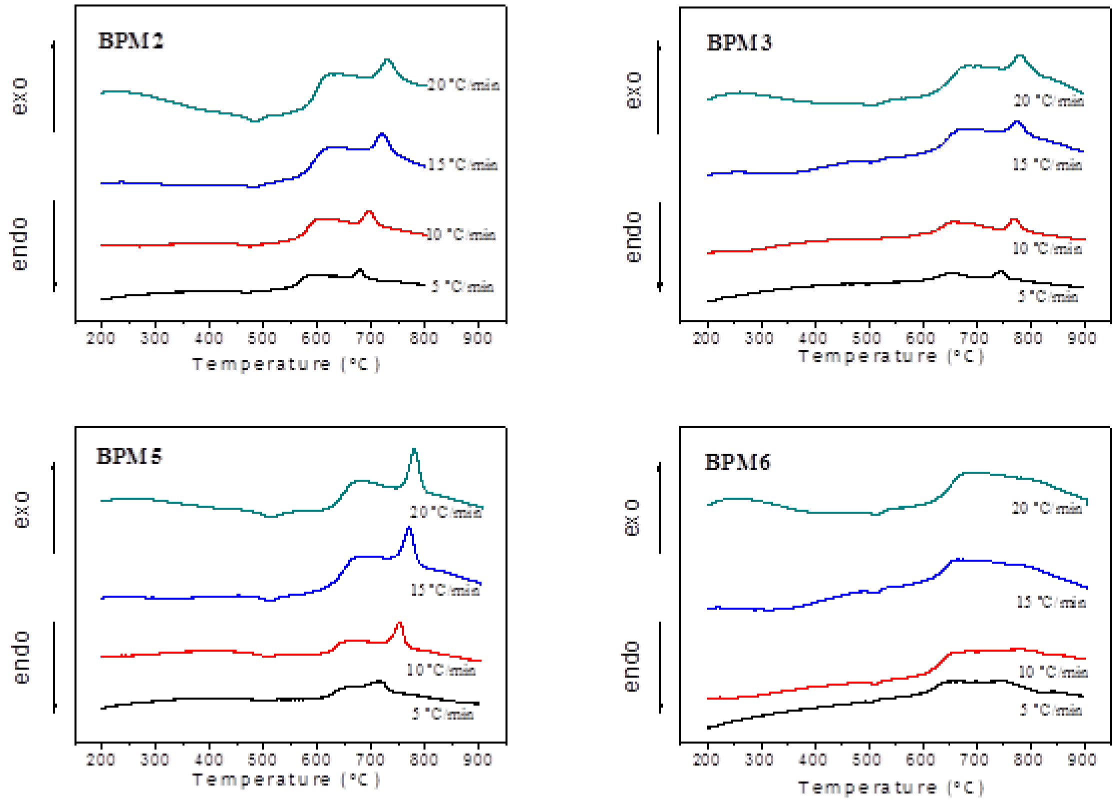


Fig. 13. DSC results at different heating rate

Structural characterization of the bulk glasses by XRD

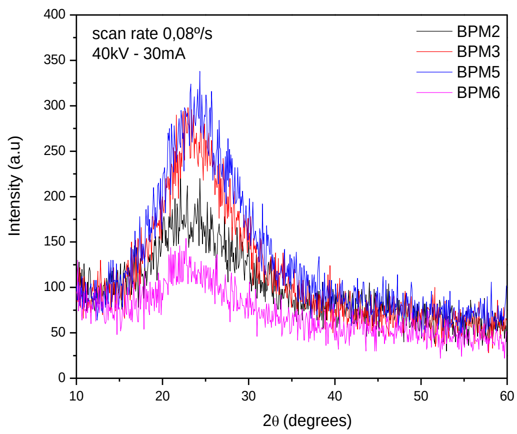


Fig. 14a) XRD patterns for BPM samples Indicate fully amorphous materials

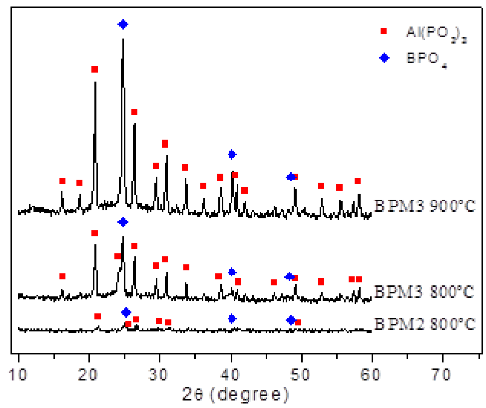


Fig 14b) Structural characterization of some heat-treated glasses
XRD patterns obtained for BPM2 and BPM3 treated at the indicated temperature (phosphate phases are identified)

Microstructural characterization of the bulk glasses by SEM

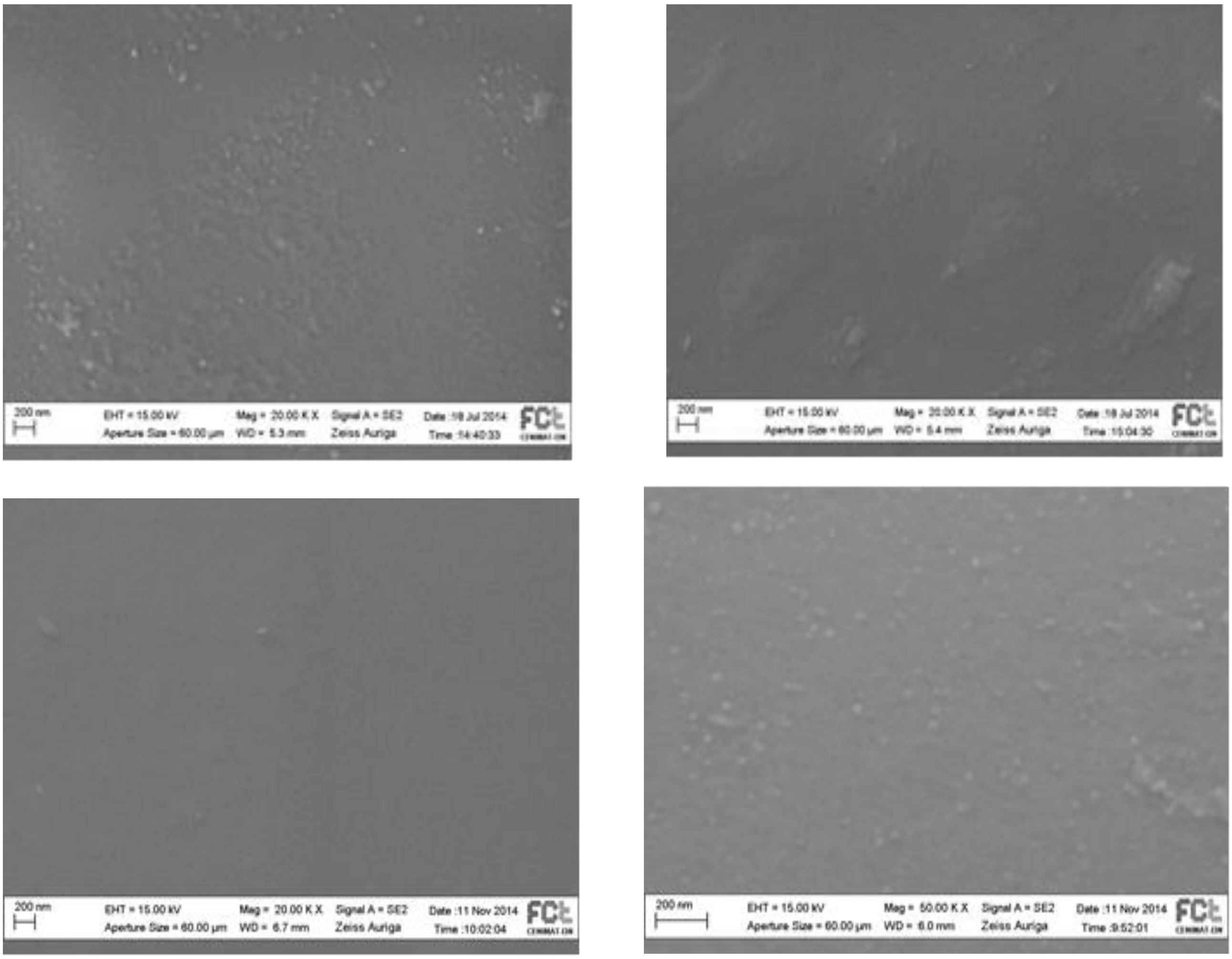


Fig. 15. SEM photos for BPM 2, 3, 5 and 6 glasses

EDS spectra of the glasses

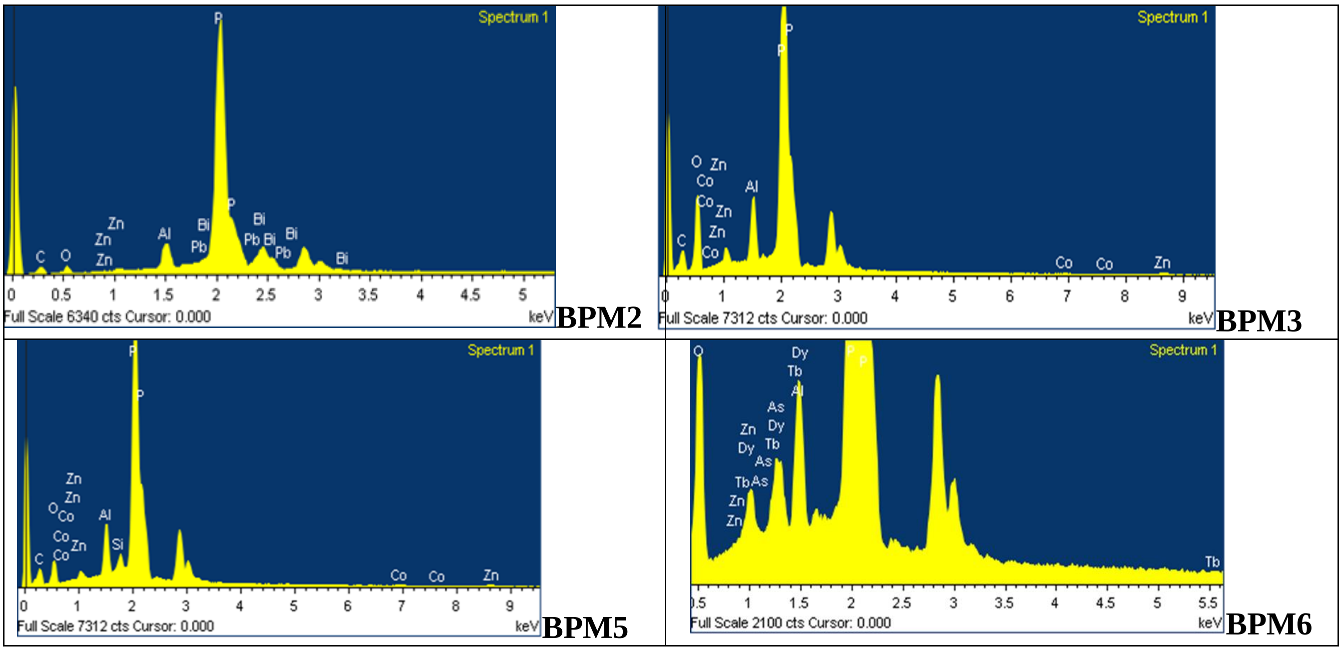


Fig. 16. EDS of the BPM glasses

Microstructural characterization SEM-EDS: i) Bulk glass with more uniform microstructure is BPM6; ii) At very high magnification, heterogeneities are observed in BPM2, BPM3 and BPM5 bulk glasses; iii) EDS spectra showed elemental composition according to XRF results, the presence of SiO2 being confirmed in BPM5 glass.

B. Characterization of thin films

Table 8. Thin films deposited by PLD

Sample code	Observation
1	BPM2-8cm-400ºC-10-4mbarO2-12500-cuart
2	BPM2-8cm-400ºC-10-4mbarO2-25000-cuart
3	BPM2-8cm-600ºC-10-4mbarO2-12500
4	BPM2-8cm-600ºC-10-4mbarO2-25000
1A	BPM1-8cm-600ºC-0.1mbarO2-12500
2A	BPM1-8cm-800ºC-0.1mbarO2-25000
3A	BPM1-8cm-800ºC-0.1mbarO2-12500

Structural characterization of the sol-gel films by XRD

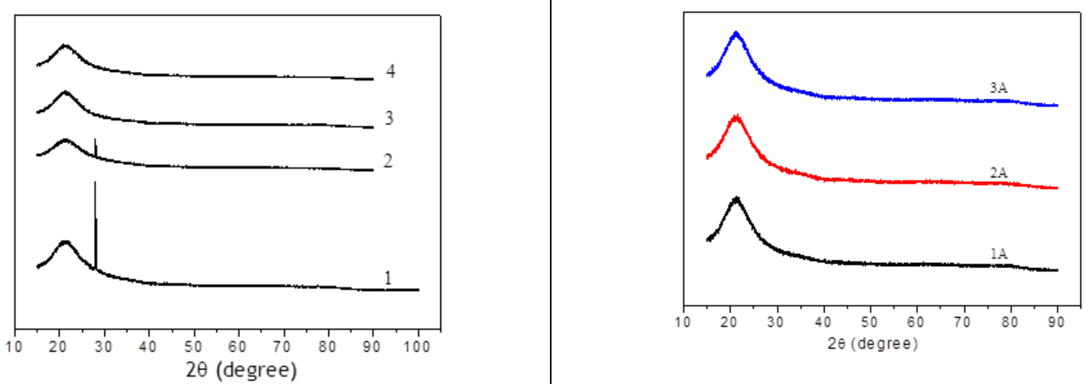


Fig. 17. XRD patterns for PLD thin films

Indicate amorphous films, except for films 1 and 2, where a peak of aluminium phosphate, hexagonal- AlPO4, corresponding to the plane (012) is identified

Microstructural characterization of sol-gel films by SEM-EDS

Film 1 was more uniform than the others. Films 1A, 2A, 3A and 4A showed irregular surface and it was difficult to measure film thickness. EDS spectra showed the presence of some elements (ex. Si) that come from the substrate.

Table 9. Thin films from BPM6 (sol-gel)

Code	Series	Thermal treatment	Substrate	Obs.	Technique
BPM6 2d1	I1	600ºC	BS	pH =1	sol-gel
BPM6 2d1	I2	800ºC	Si	pH= 1	sol-gel
BPM6 2d2	I3	600ºC	BS	pH=2	sol-gel
BPM6 2d2	I4	800ºC	Si	pH=2	sol-gel

Table 10. Thin films from BPM6 (PLD)

Code	Series	Substrate	Obs.	Average* thickness (d) (µm)	d min* (µm)	d máx* (µm)
BPM6_50f	III1	Si	25k vid			
		BS		3.7769	0.9339	4.7242
BPM6_400f	III2	Si	25k vid			
		BS		3.3423	1.1806	5.5665
BPM6_600f	III3	Si	25k vid			
		BS		1.8257	0.6401	4.5336
BPM6_602f	III4	Si	25k Ar			
		BS		3.3468	0.8807	4.6372

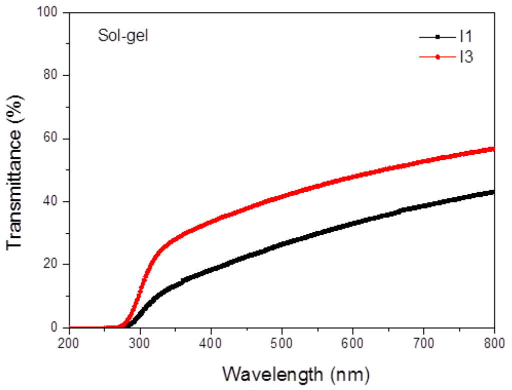


Fig. 18a) UV-VIS Transmittance of films BPM6-2d1 (I1) and film BPM6-2d2 (I3), obtained by sol-gel , deposited on borosilicate glass

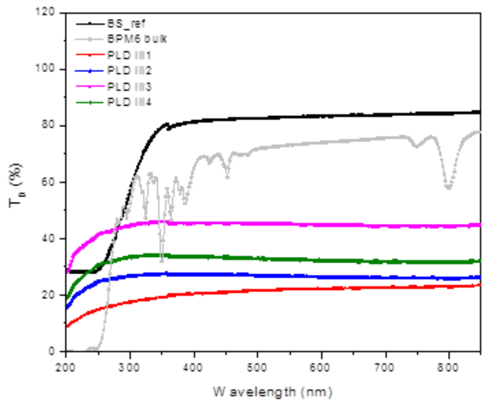


Fig. 18b) UV-VIS Transmittance of the films BPM6_III1 to III4 obtained by PLD and deposited on BS glass substrate. Comparison of values obtained for BPM6 bulk and BS substrate is shown

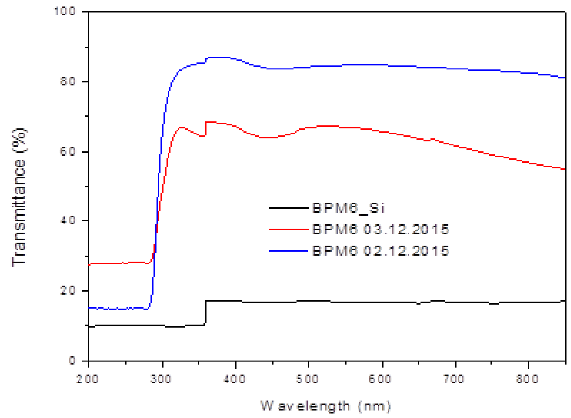


Fig. 18c) UV-VIS Transmittance of films:
BPM6 02.12.205 (MS deposition on BPM2)
BPM6 03.12.205 (MS deposition on BPM2)
BPM6/Si (BPM6 deposition by PLD at 193 nm laser wavelength on silicon)

Table 11. BPMG2d sol-gel powder samples

Code	Observation
Sample 1	dried at 150oC for 2 hours, containing P2O5-B2O3 plus Dy2O3 and Tb2O3 (68, 17, 7.5 and 7.5 molar% respectively), gelified at pH 2;
Sample 2	dried at 150oC for 2 hours, containing P2O5-B2O3 plus Dy2O3 and Tb2O3 (68, 17, 7.5 and 7.5 molar% respectively), gelified at pH 1 (HCl added);
Sample 3	dried at 150oC for 2 hours, containing P2O5-B2O3 plus Dy2O3 and Tb2O3 (68, 17, 7.5 and 7.5 molar% respectively), gelified at pH 3 (NH4OH added);
Sample 4	dried at 150oC for 2 hours, containing P2O5-B2O3 plus Dy2O3 and Tb2O3 (68, 17, 7.5 and 7.5 molar% respectively), gelified at pH 2.5 (less NH4OH added);

Structural characterization of sol-gel powders by XRD

Sol-gel powders were not amorphous. XRD patterns for all samples look very similar as the peaks seem to appear at the same positions (2 theta value), and only some small variation in the peak intensities (as arbitrary units). Most likely the peaks can be identified as : Hydrogen phosphate (H_3PO_4) ; Phosphorous (P); Boric oxide $B(OH)_3$ and Boron phosphide (B12 P2)

III. Design and manufacturing of prototype for Faraday rotator using vitreous dopped boro-phosphatic materials - SITEX

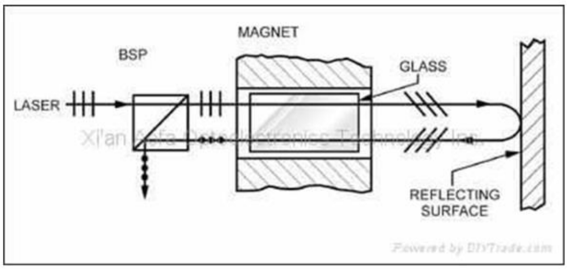
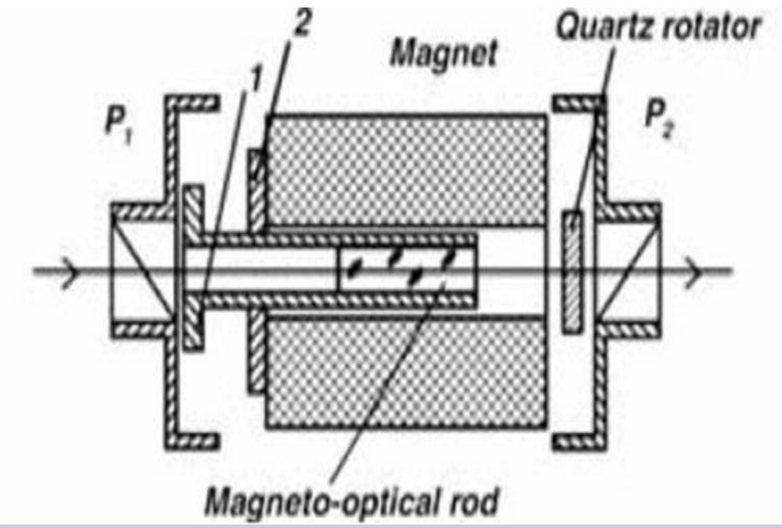
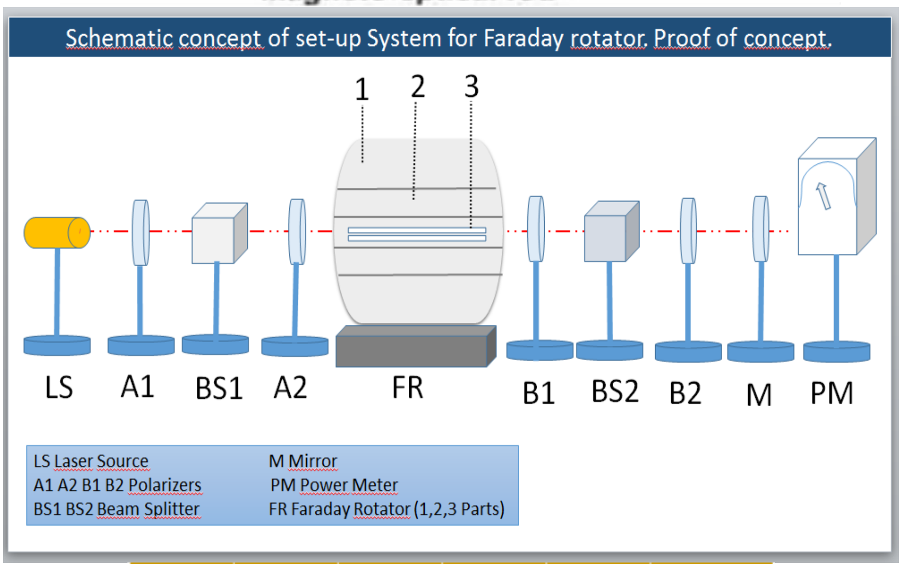


Fig. 19. Operation principle of Faraday isolator





Glass Type	Length mm	A1 mm	B1 mm	A2 mm	B2 mm
BPM6	9,87/9,86	5,04	5,08	4,97	5,07
BPM2a	9,22/9,20	4,26	4,18	4,12	4,10
BPM2b	8,88/8,85	4,16	4,33	4,20	4,32

Fig. 20. External dimensions of optical glass rods –r**ectangular version**

Table 12. External dimensions of optical glass rods –**cylindrical version**

Reper	Ext diameter mm	Length mm
Cylindrical rod of optical glass 1 Semi-finished	5	25
Cylindrical rod of optical glass 2 Semifinished	6	25

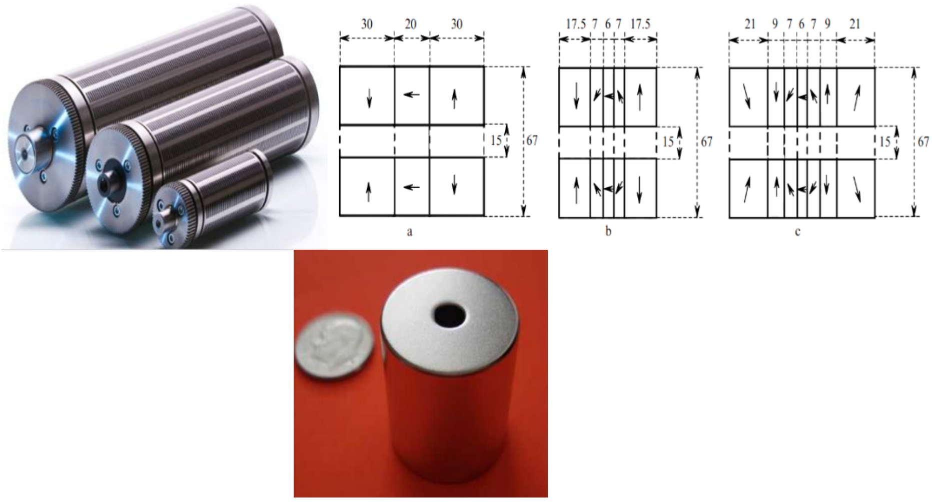


Fig. 21. Optimal dimensions of cylindrical magnet for an optical isolator with radial opening of 12mm with rings of magnetic field a) radial and axial. B) Imagnetization rings, axial and sidelong All dimesnions are in mm

Table 13. Magnetic characteristics of cylindrical magnets of Neodymium Iron Boron

Neodymium Iron Boron / Magnetic Properties									
Grade	Press[1]	Br (Gauss)	Hc (Oersteds)	Hci (Oersteds)	BHmax (MGOe)	Temperature Coefficients (%/°C)		Maximum Operating Temp @ Pc=2 (2)	
		Range	Typical	Minimum	Range	of BR	of Hci	(°C)	(°F)
N5311	D	14,200 ~ 14,700	10,300	11,000	48 ~ 53	-0.11	-0.65	~ 80	~ 170
N5014	I	13,900 ~ 14,400	13,100	14,000	46 ~ 51	-0.11	-0.61	~ 130	~ 260
N5011	D	13,900 ~ 14,400	10,300	11,000	46 ~ 51	-0.11	-0.61	~ 110	~ 230
N4916	D	13,500 ~ 14,100	12,700	16,000	44 ~ 49	-0.11	-0.61	~ 140	~ 280
N4914	D	13,600 ~ 14,100	12,800	14,000	45 ~ 49	-0.11	-0.61	~ 130	~ 260

Table 14. The selection range characteristics for magnetical cylinders

OD mm	**15**	**15**	**20**	**20**	**25**	**25**	**25**	**30**	**30**
ID mm	5	6	5	8	5	8	10	8	10
Lungime mm	20	30	30	50	30	40	50	50	60
Cantitate(buc) ***magnetizare longitudinala***	1	1	1	1	1	1	1	1	1
Valoare energie maxima (BH) MGO	35	35	40	40	42	42	42	45	45

a)

Fig. 22. Images of Faraday isolator a) Design version SOLIDWORKS as OEM Isolator Faraday integrated system c) Design versiune AUTOCAD as OEM Isolator Faraday SITEX concept with wave tunning

Table 15. Main technical characteristics for Faraday rotator

Valoarea centrala a lungimii de unda	633 nm
Domeniul de funcționare	603 - 663 nm
Transmitanța	71 - 75%
Izolație	35 - 40 dB
Diagrama performantelor	Diagramele de jos ştanga dreapta
Diametrul maxim fascicul	1.8 mm
Puterea maxima admisa	0.3 W
Densitatea maxima de putere	Nivel de blocare 25 W/cm^2 Transmitanța 100 W/cm^2

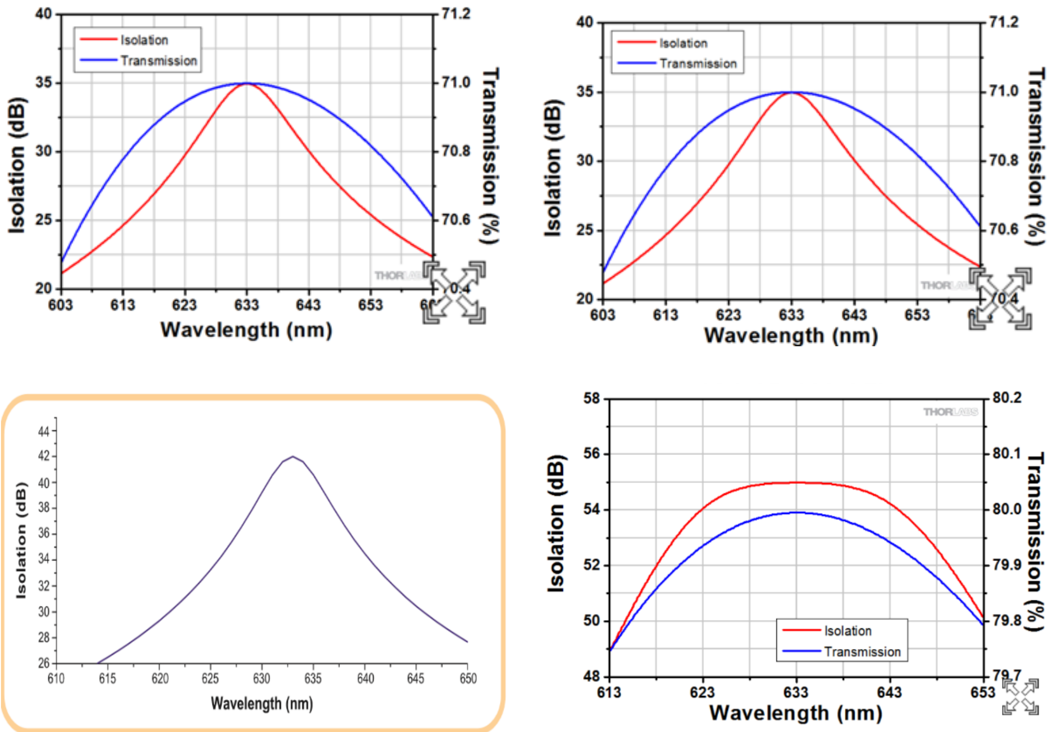
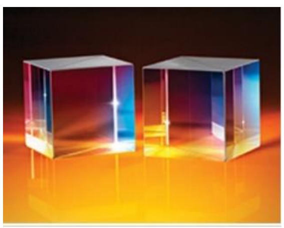
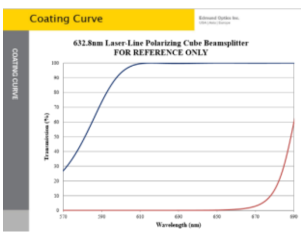


Fig. 23. Main technical features of Faraday rotator for 633 nm, wavelength depending of polarization.

Table 16. Technical characteristics Beamsplitters Edmund Optics USA





Dimensions (mm)	5.0 x 5.0 x 5.0

Dimensional Tolerance (mm)	±0.1
Clear Aperture (%)	90
Surface Flatness	λ/4
Surface Quality	40-20
Beam Deviation (arcmin)	±2
Bevel	Protective bevel as needed
Design Wavelength DWL (nm)	632.8
Extinction Ratio	T_p/T_s >1000:1 @ 632.8 nm
Substrate	N-BK7
P-Polarization Transmission (%)	>95
S-Polarization Reflection (%)	>99.5
Coating Specification	R_{abs} <0.25% @ 632.8 nm
Construction	Cube
Type	Linear Polarizer
RoHS	Compliant

Table 17. Technical sheet for Laser source TOPAG Germany

TOPAG Lasertechnik GmbH
Nieder-Ramstädter Straße 247
D-64285 Darmstadt
Fon +49-6151-425978, Fax -88
E-Mail info@topag.de
Internet www.topag.de

TOPAG Lasertechnik

Laser Diode Module
LDH – Series - Red

PRODUCT FEATURES

- Collimated or Adjustable focus
- High Stability and low noise
- Reverse Polarity Protection

APPLICATIONS

- Measurement
- Bioanalytical
- Automation
- Alignment

SPECIFICATIONS

OPTICAL

Wavelength	635 nm – 690 nm
Optical Output Power	7 - 100 mW Depending on wavelength
Power Stability	< 1%
Laser RMS Noise	< 0.5%
Wavelength Drift	0.2 nm/°C
Beam Size (1/e2)	Adjustable or Collimated (5mm)
Divergence at Collimation	< 0.5 mrad
Laser Structure	Single Mode Laser
Pointing Stability	< ± 25 µrad
Laser Operation	CW

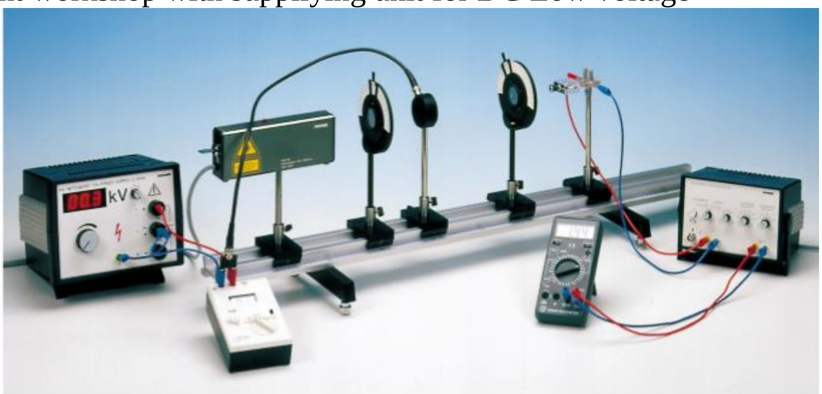
ELECTRICAL

Operating Voltage	3.3 to 5 VDC
Operating Current	< 110 mA
Control Circuit	Auto Power Control
Electrical Connections	+Red, -Black

MECHANICAL

Dimensions	12 mm (D) x 51 mm (L)
Cable	200 mm
Operating Temperature	-10°C to + 50°C *
Storage Temperature	-40°C to + 80°C
Heat Sink Requirements	Recommended for extended use

Tabel 18. Measurement workshop with suppliying unit for DC Low voltage



Compound	*Quantity*
Source, 0-12VDC 13670.93	*1*
Diode Laser, He-Ne 3.0 mw, 5VDC 08181.93	*1*
Polarizor 08610.00	*2*
Support l = 60 cm 08283.00	*1*
Positioner h-30 mm 08286.01	*1*

Photoelement 08734.00	*1*
Faraday rotator	*1*
Amplifier 13626.93	*1*
Digital Multimetter 07134.00	*2*
Cable BNC, L=750 mm 07542.11	*1*
Adapter, conector-BNC/4 mm 07542.27 1	*1*
Cable, 750 mm, blue 07362.04	*2*
Cable, 750 mm, red 07362.04	*2*
Generator 1MHz for electro-optical modulator 13650.93 /Potentiometter	*1*
Acoustic module, 8 Ohm/5 kOhm 13765.00	*1*

Fig. 24. Measurement workshop with suppliying unit for AC high voltage

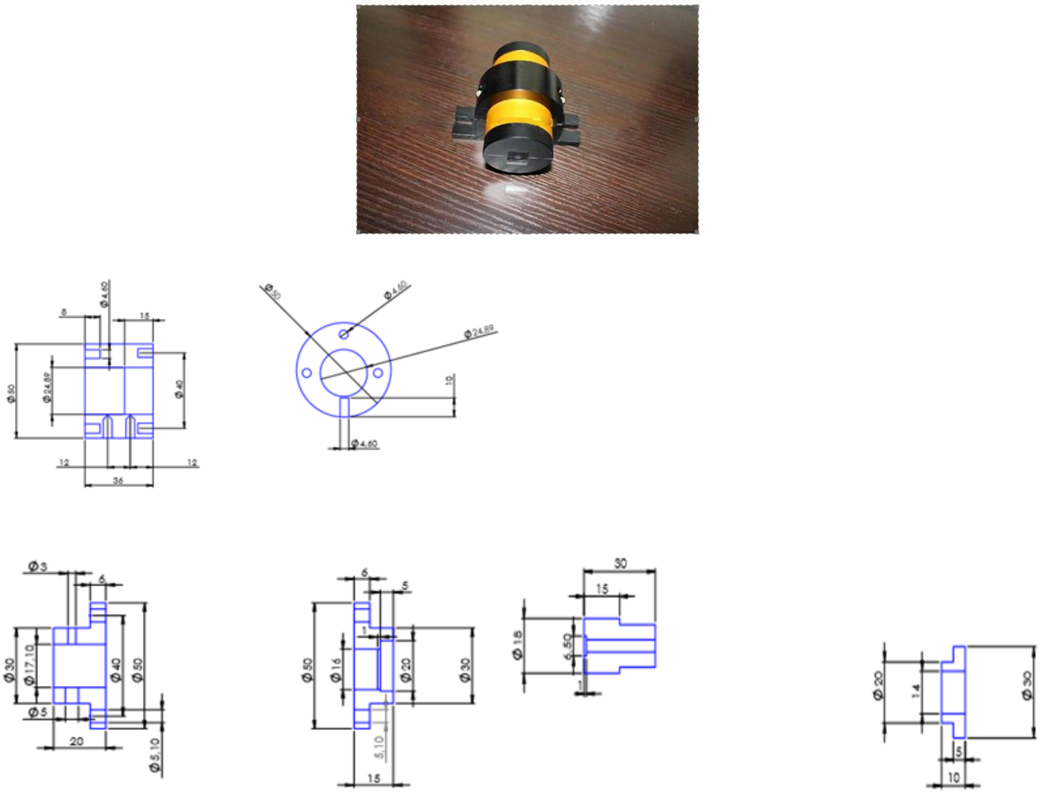
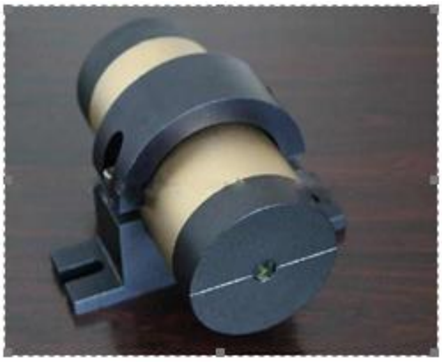


Fig. 25. Technical drawings for Faraday isolator Model 1



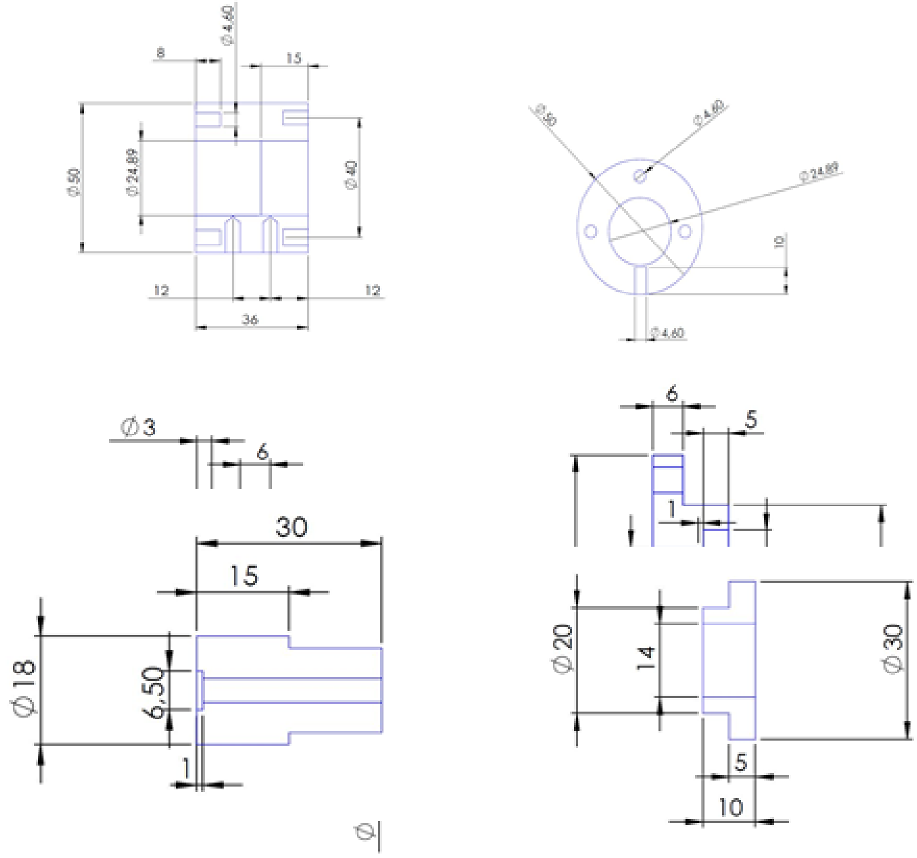


Fig. 26. Technical drawings for Faraday isolator Model 2

Dissemination

ISI Published articles

1. C. Nico, R. Fernandes, M. P. F. Graça, M. Elisa, B. A. Sava, R. C. C. Monteiro, L. Rino, T. Monteiro, "Eu^{3+} luminescence în aluminophosphate glasses", J. Lumin., 145, pp. 582- 587, 2014
2. R. S. Soares, R.C.C. Monteiro, M.R.A. Lima, B.A Sava, M. Elisa, "Phase transformation and microstructural evolution after heat treatment of a terbium-doped aluminium phosphate glass", J. Mater. Sci., 49 (13), pp. 4601-4611, 2014
3. C. R. Iordanescu, I. D. Feraru, M. Elisa, I. C. Vasiliu, A. Volceanov, S. Stoleriu, M. Filipescu, "Structural and morphological studies of Nd-doped phosphate thin films deposited by PLD on silicon wafers", J. Optoelectron. Adv. Mater., vol. 16 (3-4), 2014, p. 288 – 294
4. Roque S. Soares, Regina C. C. Monteiro, Andreia A. S. Lopes, Maria M. R. A. Lima, Bogdan A. Sava, Mihail Elisa, "Crystallization and microstructure of Eu^{3+}-doped lithium aluminophosphate glass", J. Non-Cryst. Solids, 403, pp. 9–17, 2014

5. Bogdan Sava, Mihai Elisa, Cristina Bartha, Raluca Iordanescu, Ionut Feraru, Carmen Plapcianu, Roxana Patrascu, "Non-isothermal free-models kinetic analysis on crystallization of europium-doped phosphate glasses", Ceramics International, 40, 8A, 2014, pp 12387-12395
6.M. Sofronie, M. Elisa, **B. A. Sava**, **L. Boroica**, M. Valeanu, V. Kuncser, "Rapid determination of Faraday rotation in optical glasses by means of secondary Faraday modulator", Review of Scientific Instruments 86, 053905 (2015); DOI: 10.1063/1.4920920
7. B. A. Sava, C. Tardei, C. M. Simonescu, L. Boroica, A. Melinescu, " Hydroxyapatite nanopowders obtained by sol-gel method, synthesis and properties", Optoelectronic and Advanced Materials-Rapid Communications, Vol. 9, No. 11-12, Nov. – Dec. 2015, p. 1415 – 1424
8.Olga Shikimaka, Daria Grabco, **Bogdan Alexandru Sava**[*], Mihail Elisa, **Lucica Boroica**, Evghenii Harea, Constantin Pyrtsac, Andrian Prisacaru, Zinaida Barbos, "Densification contribution as a function of strain rate under indentation of terbium-doped aluminophosphate glass", Journal of Materials Science, DOI 10.1007/s10853-015-9460-8, 2015, 51(3), 1409-1417 (2016). Springer
9. Valeanu, Mihaela; Sofronie, Mihaela; Galca, Aurelian Catalin; Tolea, Felicia; Elisa, Mihai; Sava, Bogdan; Boroica, Lucica; Kuncser, Victor; "Relationship between magnetism and magneto-optical effects in Rare Earth doped aluminophosphate glasses", Journal of Physics D: Applied Physics, 49 (2016) 075001
10. Christu Tardei, C.M. Simonescu, Cristian Onose, B.A Sava, L. Boroica, B.G. Sbârcea, "Evaluation of lab scale nano-hydroxyapatites for lead ions removal from aqueous solutions", Romanian Journal of Materials, 46 (3) (2016) 289-295
11. B. A. Sava, M. Elisa, L. Boroica, V. Kuncser, M. Valeanu, I. C. Vasiliu, I. Feraru, R. Iordanescu, "Sol-gel preparation and structural investigations of silico-phosphate glasses doped with Fe ions", Journal of Sol-Gel Science and Technology, DOI 10.1007/s10971-016-4192-z, 2016
12. Mihail Elisa, Raluca Iordanescu Cristina Vasiliu, Bogdan Alexandru Sava, Lucica Boroica, Mihaela Valeanu, Victor Kuncser, Adrian Volceanov, Mihai Eftimie, Alina Melinescu, Anca Beldiceanu, "Magnetic and magneto-optical properties of Bi and Pb-doped aluminophosphate glass", J. Non-Cryst. Solids (2016) approved for publishing

Patent Request SITEX OSIM no 02655/a/j1st june,2015
Patent Request INFLPR OSIM under submission

Papers presented :

1. B. A. Sava, O. Shikimaka, D. Grabco, M. Elisa, L. Boroica, E. Harea, C. Pyrtsac, "Thermally induced indentation recovery în terbium dopped aluminophosphate glasses", The 3rd International Conference on Competitive Materials and Technology Processes, IC-CMTP3, Miskolc Lillafured, Hungary, 2014, Book of Abstracts, Session 5, Glasses, Coatings and Related Materials, p. 96, ISBN 978-963-12-0334-9
2. B. A. Sava, M. Elisa, Lucica Boroica, Olga Shikimaka, Daria Grabco, V. Kuncser, and R. Medianu, "Rare earth doped phosphate glasses for magneto-optical devices", The 3rd International Conference on Competitive Materials and Technology Processes, IC-CMTP3, Miskolc Lillafured, Hungary, 2014, Book of Abstracts, Session 5, Glasses, Coatings and Related Materials, p. 97, ISBN 978-963-12-0334-9
3. B. A. Sava, "Phosphate and borate-phosphate glasses. Obtaining, properties and some applications", The Sixth Balkan Conference on Glass Science & Technology, The 18th Conference on glass and ceramics, 01. – 04.10.2014, Nessebar, Bulgaria, Abstract Book, p 22-23, Invited paper.
4. L. Boroica, B. A. Sava, M. Elisa, V. Kuncser, R. Iordanescu, I. Feraru, M. Eftimie, "Structure and properties of doped-borophosphate glasses for magneto-optical devices" Sixth Balkan Conference on Glass Science & Technology, 18th Conference on Glass and Ceramics, Nessebar, Bulgaria, 2014, Abstract Book, pp. 25-26.

5. Sava B., Iordanescu R., Feraru I., Elisa M., Vasiliu I., Boroica L., Bartha C., Plapcianu C., Palade. P., Valeanu M., Kuncser V., Volceanov A., Stoleriu S., "Optical, thermal and structural properties of iron-doped phosphate glasses', 12th European Society of Glass Technology-ESG 2014, Parma, Italy, Book of Abstracts, Special Glass Poster Session -13 SGP, pag.205.
6. Eftimie M., Volceanov A., Dima V., Elisa M., "Physical and morphological characterization of some phosphate-based glasses for magneto-optical application", 12th European Society of Glass Technology-ESG 2014, Parma, Italy, Book of Abstracts, Special Glass Poster Session -03 PMP, p.192.
7. L. Boroica, B. A. Sava, M. Elisa, M. Valeanu, V. Kuncser, M. Sofronie, O. Shikimaka, D. Grabco, "Magneto-optical and mechanical properties of rare-earth doped phosphate glasses", 7th International Conference on Materials Science and Condensed Matter Physics-MSCMP 2014, Vadu lui Voda, , Rep. of Moldova, 2014, Abstract Book, p. 88.
8. O. Shikimaka, D. Grabco, B. A. Sava, M. Elisa, L. Boroica, E. Harea, C. Pyrtsac, A. Prisacaru, D. Spoiala, "Micro- and macro-mechanical properties of aluminophosphate glasses depending on their compozition and loading conditions", 7th International Conference on Materials |Science and Condensed Matter Physics-MSCMP 2014, Vadu lui Voda, Rep. of Moldova, 2014, Abstract Book, p. 131.
9. B. A. Sava, L. Boroica, M. Elisa, G. Socol, A. Andrei, A. M. Niculescu, O. Shikimaka, D. Grabco, "Doped boro-phosphate vitreous materials as PLD thin films, obtaining and properties", 7th International Conference on Materials Science and Condensed Matter Physics-MSCMP 2014, Vadu lui Voda, Rep. of Moldova, 2014, Abstract Book, p. 135.
10. Iordanescu R., Feraru I., Elisa M., Sava B., Boroica L., Valeanu M., Kuncser V., Sofronie M, ".Magnetic and magneto-optical properties of La, Y and Eu-doped phosphate glasses", The 8th International Conference on Borate Glasses, Crystals and Melts "Borate Phosphate 2014", Pardubice, Czech Republic, Published by ICARIS Ltd., p. 206.
11. Sava B. A., Boroica L., Elisa M**.**, Medianu R. V., Monteiro R. C. C., **"**Boro-phosphate glasses doped with d and f oxides for photonics, preparation and characterization", The 8th International Conference on Borate Glasses, Crystals and Melts "Borate Phosphate 2014", Pardubice, Czech Republic, Published by ICARIS Ltd., p. 219.
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