Application of sol-gel process for preparation of functional materials

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Abstract:

A development of high-performance materials for electronics, optics, ceramics fabrication is restrained by traditional modes of their production. Basic technologies for materials include high-temperature physical and chemical processes and require special conditions to attain desired properties of final products. Advanced preparation methods for materials with new features are feasible on the basis of colloid-chemical processes and nanochemistry. In this respect the sol-to-gel transformation followed by solidification and chemical modification are of great interest to attain a variety of different functional properties of materials.

Keywords: *sol-gel, colored silica film, rust-proofing inorganic covers, colloidal nanosized silica.*

1. Introduction

Sol-gel process in comparison with traditional methods of deriving of vitreous structures has unique advantage: it allows preparing for the same composition, such as silicon oxide, as considerably distinguished shapes - fibrils, films, monoliths, xerogels, aerogels by changing only some experimental conditions. Parameter of processing which needs to be driven, viscosity, pH of sol and concentration of oxides which should be in fixed limits.

Distinctivenesses sol-gel of production engineering in comparison with traditional methods of forming of composite materials are:

- High chemical homogeneity and purity component, containing in a final material on a molecular scale;
- Pliability and controllability of process;
- Forming of silicate and ceramic matrixes at lower temperatures;
- The high reactive capacity of porous xerogels and subsequent them solid; phase transferring in glass or an aerogel, without of a stage of a melting, allows to gain the broad audience of chemical combinations.

Sol-gel technology is very simply, cheep, ecologically. For example, this methods yields high-purity and activated silica glasses for optics, optoelectronics and laser optics at lower temperatures eliminating the fusion stage. The sol-gel transition occurs due to polycondensation, hydrolysis, gel formation followed by heat treatment producing dense gels. Unlike fused glasses, gel ones contain less impurities that results from the quality of initial materials. They also have a lower synthesis temperature. The sol-gel technology is believed as energy and resource saving process. Is one more advantage is the simplicity of necessary equipment [1].

2. Results and Discussions

2.1. Colored silica sol-gel films on soda-lime glasses and plastic substrates

Glass tiles are generally used for decoration purposes. There are two types of sol-gel coatings: inorganic and hybrid inorganic-organic ones. Especially, when the safety regulation matters, the inorganic sol-gel coatings are useful. The inorganic sol-gel coatings are based mostly on metal oxides result in minimum harmful fumes under fire. Inorganic sol-gel coatings in building industry, like glass tiles, are developed in many countries [2].

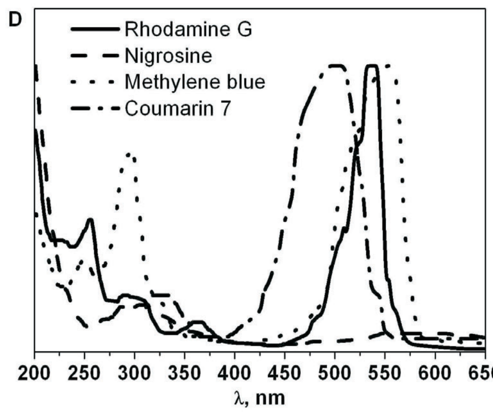

Fig. 1. Absorption spectra of silica sol-gel films doped with organic dyes.

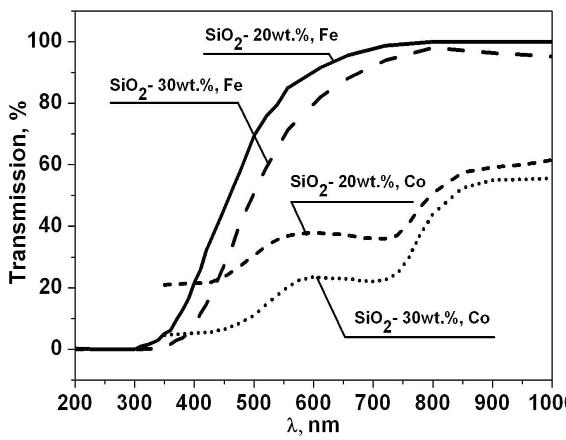

Fig. 2. Transmission spectra of silica sol-gel films doped with metal oxides.

Hybrid coatings would have both the advantages from inorganic-organic materials. In this work, we present the films doped with organic dyes (rhodamine, coumarine, nigrosine, methylene blue) and metal (Fe, Co, Fe-Co) oxides [3]. Thickness of these films synthesized on plastic substrates at temperatures 100-1500 °C and soda-lime glasses at 5000 °C was 0.2-5 µm. The films possess good adhesive properties and high stability with respect to mechanical attrition those fit the industry standards. The refraction index and factor of dispersion are the same as for conventional silica films. Absorption bands in the visible can be varied depending on type and concentration of organic dyes (Fig. 1) or inorganic dopants (Fig. 2).

2.2. Rust-proofing inorganic covers

By the sol-gel technique we have developed the technology to produce protective and rust-proofing inorganic silica covers on the metal surface. Precursor solutions for film-forming solution will be prepared by hydrolyse of silicon organic compound. That is conventional procedure used for silica sol-gel films. films are produced by spin- or dip-coating on a substrate at room temperature. Then films are heated at 400 °C in air. Advantages of protective and rust-proofing covers on the metal surfaces:

1. Resistant to against mechanical attrition the coat is formed at temperature 400 °C.
2. Inductivity quantity – 10.5-8.4.
3. Mechanical stability to a temperature cycling up to T = 700 °C.
4. Humidity and corrosion resistance in aggressive lead-acid and an alkaline condition.

At temperature of aggressive medium up to 50 °C.

2.3. Silica gel glasses, doped trivalent rare-earth ions for fiber optics applications

Sol-gel silica glasses doped with rare-earth ions are an important class of optical materials with applications including solid-state lasers, optical waveguides, fiber amplifiers and fiber optic sensors. The silica gel glasses can be obtained by the sol-gel process. The samples were prepared from tetraethoxysilane, water, ethanol, fumed silica and soluble in water or ethanol the salts of the RE^{3+} (Er^{3+}, Nd^{3+}, Sm^{3+} *et al.*) elements.

Sol-gel glasses usually contain a considerable amount of OH^- groups, which reduce the efficiency of luminescent emission. A significant reduction in hydroxyl ion concentration is possible by thermal treatment of xerogel in the fluore-containing atmosphere. The samples were then vitrified in air. Then rods were formed from small samples of the doped glass, which were subsequently used in the drawing of PCS-type fibers with the core diameter of 200 µm.

The properties of these fibers are more interesting: their ability of being fiber sensors and pumped amplifiers.

The glasses synthesized by the sol-gel method have almost all properties of silica glass (Table 1), possess high optical quality and can be made large enough to their practical application. The technology may contain additional stages to reduce the concentration of OH^- groups if necessary. The above-described sol-gel method can be used to fabricate silica glass blanks of various shapes including blanks of the glass activated by rare-earth elements which can be applied as light filters and blanks for optical fiber drawing.

Table 1. Comparative characteristics of fused and sol-gel glasses.

Characteristic	Sol-gel glasses	Fused silica glasses
Density, g/cm^3	2,201	2,203
Chemical resistance	Class A	Class B
Refraction index	1,458	1,458
Concentration of OH^- groups, wt.%	0,0005-0,1	0,03-0,05
Linear thermal expansion coefficient, K^{-1}	$0{,}55\times10^{-6}$	$0{,}55\text{-}0{,}58\times10^{-6}$
Microhardness, MPa	7000-8500	6860-8850
Young's modulus, GPa	7,0±0,1	7,3

All matrices synthesized by the sol-gel method show the effect of the sensibilization of the luminescence of rare-earth ions by silver ions because of the above-described features of the matrices. For example, the amplification of luminescence reaches 2-5 times in Sm-Ag- and Eu-Ag-containing glasses and 2-3 times in Sm-Cu- and Eu-Cu-containing systems compared to single-activated glasses.

Table 2. Dependence of intensity of peak luminescence of Sm-Ag-containing glass on its composition.

Glass composition			Amplification of peak luminescence
SiO_2	Sm_2O_3	Ag_2O	I_{lum} (λ_{exc} = 280 nm; λ_{reg} = 280 nm)
96,9	2,0	1,1	1,1
96,0	2,0	2,0	2,0
99,3	0,2	0,5	0,5
99,0	1,0	-	-

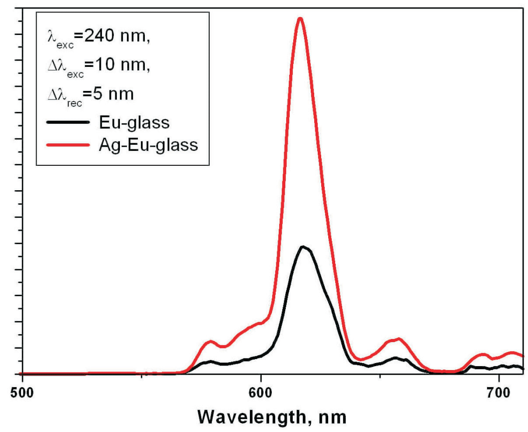

Fig. 3. Luminescence spectra of Eu- and Eu-Ag-containing glasses.

Complex centers with a high efficiency of the luminescence of Eu^{2+} in the luminescence band with the maximum at 450 and 590 nm appear in Eu-Al-containing glasses because of the above-described reasons. These centers transform effectively UV radiation into the yellow-blue spectral band.

2.4. Colloidal nanosized silica for mechanochemical polishing of single-crystalline silicon

A colloidal dispersion, referred to as a sol, contains very fine particles (1-100 nm in diameter) that remain in suspension for very long times. In colloidal silica, the particles are amorphous and they have a negative electrical charge. Owing to electrostatic repulsion of particle from each other the stability of dispersion is increased. They are nearly insoluble in the dispersing medium.

Colloidal nanosized silica is meant for a finishing polish of optic and electronic products. In particular, it is used at the stage of finishing polish of single-crystalline silicon wafers. It is an ultra-disperse colloid system based on silicon dioxide. It is a liquid of the color of the milk without any visible mechanical inclusions. The obtained product has following characteristics:

• Density of suspension	1.09-1.10 g/sm^3
• pH at 20 0C	5.5-7.0
• Contents SiO2	13-17 wt%
• Size of particles	30-80 nm
Viscosity	1.5-1.7 MPa·s

Colloidal nanosized silica possesses a polishing capability for optic and electronic products at a normal speed of a material lift. The time of processing of single-crystalline silicon wafers is 30 minutes at the primary stage of mechanochemical polishing and the material removal has compounded 30-35 microns. The time of processing is 15 minutes at the secondary stage of mechanochemical polishing and the material removal has compounded 5-7 microns. These suspensions on the basis nanosized particles of fumed silica have high stability, small of an arising static electricity on polish, high efficiency, good selectivity, ease of usage, the minimum impurity by ions of metals and are usable at planar stage of metallical layers by production of integrated circuits.

3. Conclusion

Sol-gel method offers the advantage of a relatively simple production procedure of the vitreous material. This method is known to produce materials from solutions either in bulk, coating, films, fibers or powders. Silica sol-gel materials are characterized by the lowered maintenance of the impurity, the caused cleanliness of initial materials, and low temperature of synthesis; besides a sol-gel method gives more ample opportunities of influence on physical and chemical parameters of final products.

Sol-gel technology is resource- and energy-saving, allows receiving ready products or perform with form and sizes to finished articles (rational preparations) with the big percent of an output suitable and small quantity of waste products. Its advantage is also simplicity of the used equipment.

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