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Radio-frequency Induction plasma modification effects of disperse systems based on mineral glass

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Abstract. In the article the possibility of use of high-frequency induction plasma discharge of low pressure as a tool for pre-processing the glass optical elements. Established effects of polishing, improve the adhesion of the glass elements to the polymer binder and the increase of the reflection coefficient.

1. The introduction

Low-temperature nonequilibrium plasma can serve as way for achieving the surface effects of materials modification. During materials treatment surface is bombarded by active particles, such as ions, electrons, plasma-forming gas atoms. As a result, near-surface materials area becomes modified. Given method is ecologically clean due to the absence of active chemical and biological substances which resulting in the absence of effluents and emissions of harmful gases.

Other advantages include short time of treatment the possibility to set accurate parameters for obtaining desirable effects, and wide variety of treated materials [1]. Plasma technologies can be used for obtaining various properties to non-organic materials, including ones consist of mineral glass.

Glas is an amorphous solid that does not have ordered crystal structure, and is obtained by overcooling of a melt [2]. Technical and household glasses are needed to be adequate to increased requirements for its hardness, wear resistance, optical properties and soundproofing. Needed glass properties can be obtained by adding different components during the producing of materials at the silicate formation process [3].

Traditional methods of the surface treatment of end glass product are used for improving decorative and operational properties, and consist in chemical etching with sulfuric and hydrofluoric acids [4-5]. Disadvantages of traditional methods are: low ecological purity of the processes, lengthy duration and necessity of preliminary treatment.

Plasma modification does not change internal structure of the material and provides a wide variety of its effects. In the investigation [6] functional microrelief on the surface of the diffractional optical elements has been created by using radio-frequency induction discharge. Formed elements are a diffractional lattices of phase type. In the investigation [7], cold argon plasma was used for changes in the glass surface. The changes in the chemical structure (silicate system regrouping), surface energy and morphology (microrelief surface formation) under plasma treatment was also discovered. It should be noted that there is a possibility of the large micro-meters roughness removal from production fiber glass surface with negligible weakening [8]. The glass surface adhesion increasing without microrelief...
formation can occur due to the chemically active centers forming on the surface, and because of silicate system regrouping during ion dispersion of the near-surface layer.

2. Materials, methods and equipment

Objects of this investigation are the glass microspheres (GMS) with 50-100 µm dispersion, which are used for road markings as a retroreflective material approached to BS 6088:1981.

We have considered the possibility of the microspheres surface smoothing under the influence of radiofrequency induction low-pressure discharge with inert gas – argon (Ar).

Technical parameters of the treatment were the following: plasma forming gas – argon; gas rate \( G_{\text{Ar}} = 0.04 \text{ g/s} \); current intensity on the a generating lamp anode \( I_A = 2.5 \text{ A} \); pressure in the working chamber \( P = 40–90 \text{ Pa} \). GMS were injected into the discharge and with plasma forming gas flow were passed through discharge “core”. Behind a plasma generator cut, treated particles were caught by filter-trap. Plasma treatment was made with experimental RF Induction plasma set which has been described in the source [9].

3. Results

Investigation of the surface before and after RF Induction plasma treatment was done by Scanning Electron Microscope (SEM) method and Atomic-Force Microscope (AFM) method. Established plasma effect of surface polishing and microcracks healing is shown with SEM-images (Figure 1).

AFM-images before and after RF Induction plasma treatment are shown at the Figure 2-3. Calculated values of roughness are shown in the Table 1.

| Roughness parameter | Starting sample | Sample after RF Induction plasma polishing |
|---------------------|----------------|------------------------------------------|
| \( Z_{\text{max}} \), nm | 338,732        | 187,857                                  |
| \( R_z \), nm        | 170,145        | 92,8459                                  |
| \( R_a \), nm        | 17,2589        | 7,48692                                  |

\* \( Z_{\text{max}} \) – maximal relief height; \( R_z \) – sum of the mean absolute values of the five largest profile ledges and five largest profile valleys within basic length, \( R_a \) – arithmetic average of the absolute values of the profile deviation.

At the SEM-images it is shown that surface of the starting microspheres is covered with apprets, pollutes and defects (figure 1 a). Surface of the experimental sample shows less amount of pollutes, mechanical defects are smoothed (figure 1 b), which also is confirmed with AFM data of roughness and with higher magnification.
Thus, microscope results demonstrate cleaning effect and polish effect of the retroreflective microspheres surface, and, also, microcracks and microroughnesses healing. It is concluded that such treatment can elevate optical properties of the glass microspheres and its adhesion to polymer binding agent.

Resulting from the specific application of the GMS, the increase of its adhesion to polymer binder under plasma treatment, is a necessity which allows to extend service life of the covering, based on GMS.

In the study, the possibility of RF plasma discharge application has been investigated as an instrument for surface modifying and increasing the adhesion force.

For the wear resistance examination of the retroreflective materials based on GSM we made test samples. For the substrate we used plates of steel with measurements 150×60 mm. Beforehand, plates were degreased and coated with layer of the polymer bonding agent (acryl copolymer) with 0,2-0,3 mm thickness and with layer of the spheres with 0,1-0,15 g/sm² consumption. After 48 hours the obtained composite was tested with the use of laboratory test bench for determination surfaces service life under mechanical load. Results are shown in the Table 2.

| Sample   | Mass lost after mechanical impact, g | Mass lost relatively to starting sample % |
|----------|--------------------------------------|------------------------------------------|
| Treated  | 1,69                                 | 18,76                                    |
| Untreated| 2,5                                  | 27,79                                    |
Results show that the mechanical resistance of the coating based on plasma modified microspheres has increased 9%.

The destruction of the coating was evaluated by confocal laser scanning microscopy (CLSM) method (Figure 4).

Figure 4 CLSM-images of the retroreflective coatings on the steel plates with acryl copolymer binder a) GSM without plasma treatment; b) GMS modified under plasma treatment.

Modified microspheres after physical and mechanical tests maintain their even distribution.

Microscopy results demonstrate cohesive destruction of the material and of the spheres themselves (Figure 4 b), which provides evidence for adhesion force increasing.

In addition to service life of the coating, important factor for obtained material is reflection coefficient. Measurements were made with spectrophotometer X-RiteColor DTP22 Swatchbook. Control samples and samples with modified under plasma treatment GMS were investigated and compared. Results are shown at the Figure 5.

Diagram at the Figure 5 shows that after modification in RF gas discharge, reflective coefficient for silicate spherical materials with 10-60 μm dimensions shifts in the more high areas, which provides the evidence for improvement in optical properties of the GMS after plasma treatment. In terms of obtained data, positive influence of plasma treatment at optical properties of the glass microspheres surface has been established.

4. Conclusions
Thus, it is concluded that RF Induction low-pressure plasma treatment for GMS modification potentially is a highly effective method. During the investigation, positive effect for the roughness
the surface has been confirmed. Parameters of the roughness were reduced more than 80%, and the exploitation properties, such as adhesion interaction, was increased 66% in comparison with the starting sample, and reflective coefficient was also increased. Therefore, RF Induction plasma treatment can be recommended as a method of GMS preprocessing for retroreflective coatings.

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