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THERMAL INSULATION PROPERTIES OF HOLLOW GLASS MICROSPHERES/EPOXY AND CERAMIC MICROSPHERES/EPOXY COATINGS

STEVAN STUPAR
Military Technical Institute, Belgrade, e-mail: stevan.stupar13@gmail.com

DRAGANA LAZIĆ
Military Technical Institute, Belgrade, e-mail: lazicdragana85@gmail.com

NEGOVAN IVANKOVIC
University of Defence, Military Academy, Belgrade, Serbia, e-mail: negovan.ivankovic@gmail.com

DENIS DINIĆ
CBRNE Centre, Kruševac, Serbia, e-mail: denis.dinic@yahoo.com

***Abstract**: The thermal insulation properties of the hollow glass microspheres/epoxy and ceramic microspheres/epoxy coatings were studied as possible materials for suppressing the thermal signature of the object in the real conditions. The study was separated into two parts, the particles and coatings characterization by SEM/EDS analysis and thermal insulation properties determination. The characterization of particles and coatings was depicted the microstructure, morphology, and particle size distribution. During the thermal insulation properties study, the influence of the initial concentration of the particles, and the heating temperature influence on thermal insulation properties was investigated. The thermal insulation properties were confirmed in both types of coatings related to the ceramic plate with basic military epoxy coating and ceramic plate without coating.*

***Keywords**: Coatings; thermal signature propulsion; camouflage; thermal insulators*
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INTRODUCTION

According to Planck`s law, any object with a temperature above absolute zero emits thermal radiation, which results in the existance of thermal signature of the body in infra-red range [1]. This principle is used for the modern equipment for military observation and reconnaissance aims to discover the IR signature of the objects. Based on the dependence of thermal radiation on surface emitance and temperature, the propulsion of the thermal signature of the body can be achieved by controlling these parameters.

The surface emitance, as one of two parameters for determination of the thermal radiation, can be controled by nanostructure-based surfaces (e.g., metasurfaces [2] and dielectric materials [3]) or multilayer films [4,5]. The second parameter, the temperature of the surface can be reduced using materials with low thermal conduction capacity which causes the blocking of heat transfer from engine of object to surface. This characteristic of the materials is the one of the most important parameter to develop energy-saving technology and has attracted attention in recent years, especially in civil engineering [6,7]. Nowadays, many new materials are being developed for thermal insulation: thermal insulators [7], phase-change materials [8], and transformation thermotics [9].

According to the defined range of thermal conductivity coefficient (λ), materials with λ<5 W/(m K) are good thermal insulators.The non-metalic materials (e.g. ceramics, glass, polimers, etc.) have lower thermal coefficient unlike metals, as a consequence of heat transfer by phonons [10]. The hollow glass microspheres (HGM) and ceramic microspheres (CM) are promising materials for decreasing thermal radiation and contribution on camouflage characteristics of coatings [7,11].

According to our knowledge, a comparison of thermal insulation and physical-mechanical properties of HGM/epoxy and CM/epoxy coatings has not been investigated before. In the first part of the study,caracterisation of additives and coatings was performed. Second, the influence of supplementation of the hollow glass microspheres and ceramic microspheres to increasing thermal insulation properties was investigated by comparison of the ceramic plate without coating, with epoxy and modified epoxy coating. Also, the influence of heating temperature on thermal insulation properties was investigated.

EXPERIMENTAL PART

Materials and characterisation

The hollow glass microspheres and ceramics microspheres were purchased from 3M. The commercial code for the hollow glass microsphere is K20 and for ceramics microspheres is W410. The epoxy coating and thinner used as medium for was purchased as final product for military purposes are provided by Pitura doo.

The microstructure and morphologies of the powders and coatings were examined using the scanning electron microscope, JEOL JSV-6610LV. The distribution of particle size was done using the images where individual particles were distinguishable and their diameters were measured using the image analysis software. The SDL Atlas G209A thermostatic chamber was used for sample conditioning. The surface temperature during the thermal insulation performance test was followed continuously by the thermocouple (Hanna HI Termocouple thermometer HI 93551).

Preparation of HGM/epoxy and CM/epoxy coatings

All coatings were prepared by uniform mechanical mixing of additives and epoxy coatingusing the mechanical stirrer (Witeg HS–100D) for 30 minutes. Before the HGM and CM particles addition, thinner was directly added to all initial epoxy coatings in mass ratio 30:1 and mixed to adjust the density. After initial coating preparation for application, the coatings were supplemented by 16.6, 33.2, 49.8, and 66.4 g/L of HGM and CM particles and homogenized by mixing. After preparation, the application of coatings with the desired thichkness on different materials was performedusing a film applicator (Model 352, Erichsen CO.).

Thermal insulation performance tests

The thermal insulation properties of coatings were based on temperature reduction through the applicated coating. The thermal insulation tests were performed under air atmosphere, the temperature of the ambience was maintined at 21 ± 0.1 ˚C. The air velocity and relative humidity were adjusted as 1 ± 0.1 m/s and 65 ± 4%, respectively. The thermal insulation was examined onto the ceramic plates (150x120 mm) without any coating as refferent sample for thermal insulation parameters of ceramics, plate applicated with base coat, HGM/Epoxy and CM/Epoxy coatings. Before setting measurement, all samples were conditioned at 21± 0.1 ˚C in a thermostatic chamber for 4 hours, and the oil bath was heated on required temperature for measurement. The ceramic plates were positioned onto the aluminum carrier in order to ensure direct contact with the tempered oil from the oil bath and accumulate the temperature uniformly over the entire plate surface. The coating thichness of all samples were in the range 130-140 µm, and time of heating was 60 minutes. At least three measurements were done for each determination of thermal insulation parameters.

RESULTS AND DISCUSSION

Structure and morphology

In the first part of the research, an examination of the morphology and structure of the additives and coatings was conducted. The morphology of particles (HGM and CM) and (HGM/epoxy and CM/epoxy coatings). Figure 1 and Figure 2 show the SEM photographs, Energy-dispersive X-ray spectroscopy and distribution of diameter of the hollow glass and ceramic microspheres, respectively. The SEM photographs of coatings with addition of microspheres are presented in Figure 3.

Energy-dispersive X-ray spectroscopy of the marked area of SEM photograph confirms the presence of silicon and oxygen for HGM particles and silicon, aluminum, and oxygen for CM particles which confirms the expected chemical composition and purity of the powders. In HGM particles the presence of silicon and oxygen was approximately 49 and 39 wt. %, respectively. In CM particles presence of oxygen, silicon, and aluminum was approximately 44, 27, and 23 wt. %, respectively. The presence of sodium and calcium in HMG particles and magnesium, potassium, titanium, and ferrum in CM particles in small amounts is caused by impurities remaining after the synthesis process originated from precursors. By analyzing the results of the particle size distribution of HGM and CM particles, the particle size of HGM and CM particles has a normal distribution with the most common range of diameter is 50-60 and 60–70 µm, respectively. The average diameter of HGM particles was 51.23 µm, and for CM particles was 63.42 µm. According the Figure 3, the applied coatings adhere well to the surface and cover and protect the microspheres from physical damage.

Thermal insulation properties

The thermal insulation properties are studied in two parts, the effect of the initial concentration od supplemented microspheres and the effect of heating temperature.

The effect of the initial concentration of HGM and CM particles in epoxy coatings was studied at identical thicknesses of coatings and heating temperature (60 ˚C). In this part of the experiment, the initial concentration of particles was in the range 16.6 – 66.4 g/L. Figure 4 shows the relative ratio of surface and oil bath temperature versus time for the initial concentration of particles.

According to results presented in Figure 4 the addition of HGM and CM particles into epoxy coatings improve the thermal insulation characteristics. By comparison of two types of additives, the HGM particles have better insulation properties than CM particles at the same initial concentrations. The temperature after 60 minutes of heat exposure, at the surface of the ceramic plate with the addition of 66.4 g/L HGM particles in the epoxy coating was 20.26 % lower than the temperature of the oil bath. The ceramic plate with applied CM/epoxy coating with the same initial concentration of particles, the surface temperature was 18.81 % lower than the T_{max}.

The second part includes testing the effect of temperature on thermal insulation properties. Figures 5 and 6 shows the relative ratio of the coated ceramic surface and the oil bath temperature versus time for the different heating temperatures (60, 80, 100 ˚C).

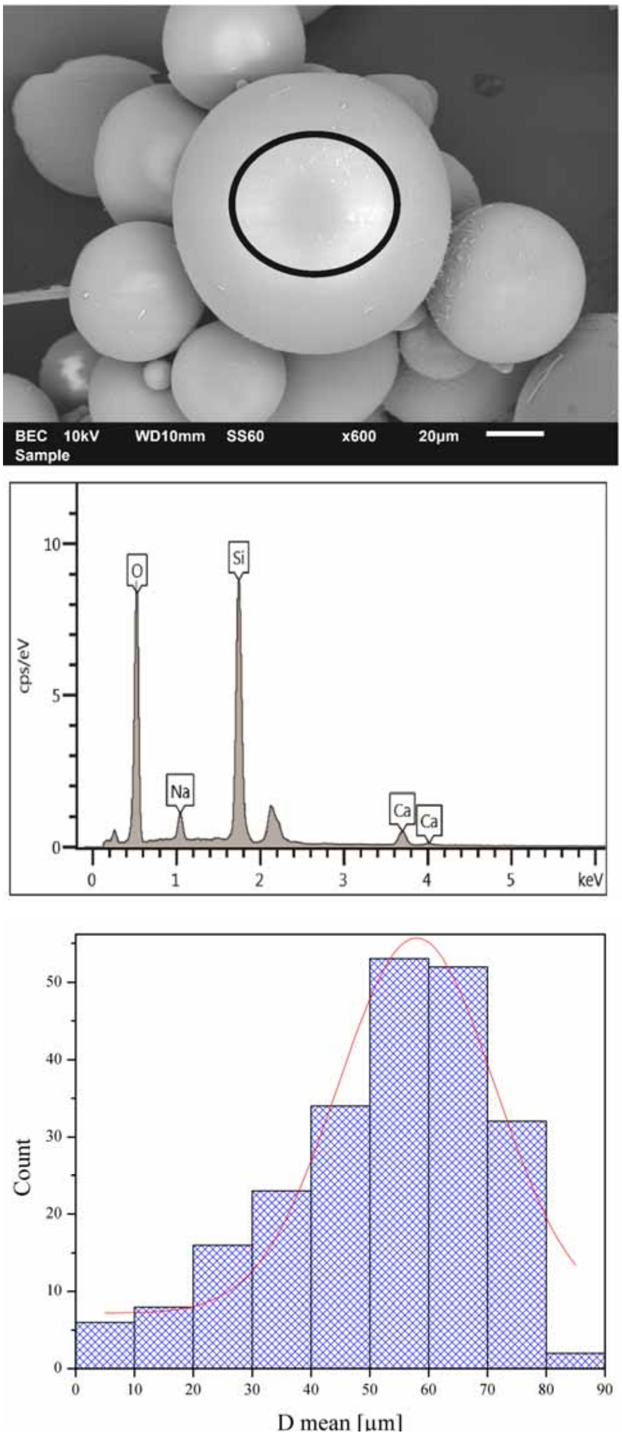


Figure 1. SEM photograph, Energy-dispersive X-ray spectroscopy and distribution of diameter of hollow glass microspheres.

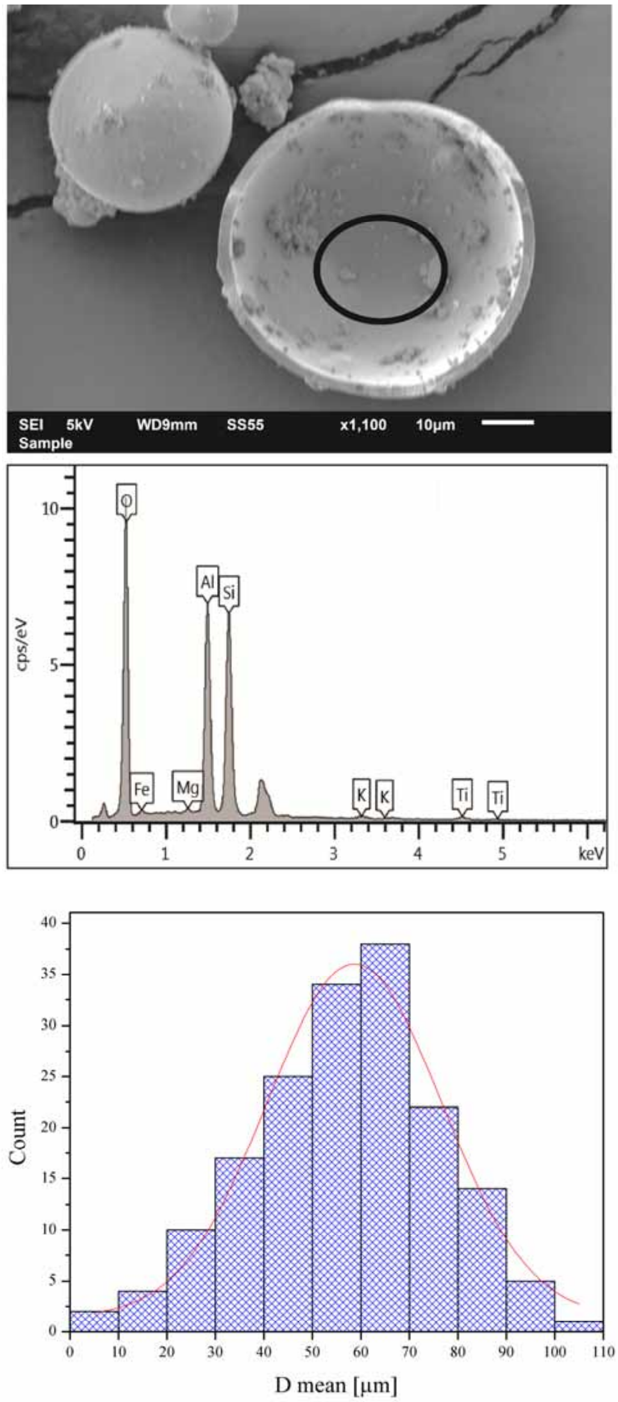
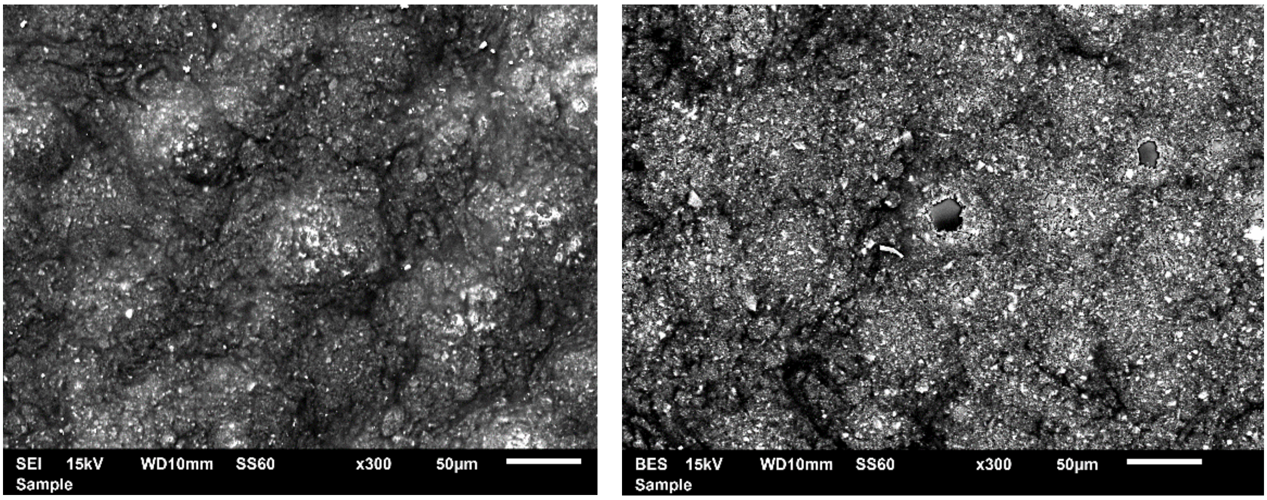


Figure 2. SEM photograph, Energy-dispersive X-ray spectroscopy and distribution of diameter of ceramic microspheres.



a) b)

Figure 3. The SEM photographs of: (a) HGM/epoxy coating; (b) CM/epoxy coating.

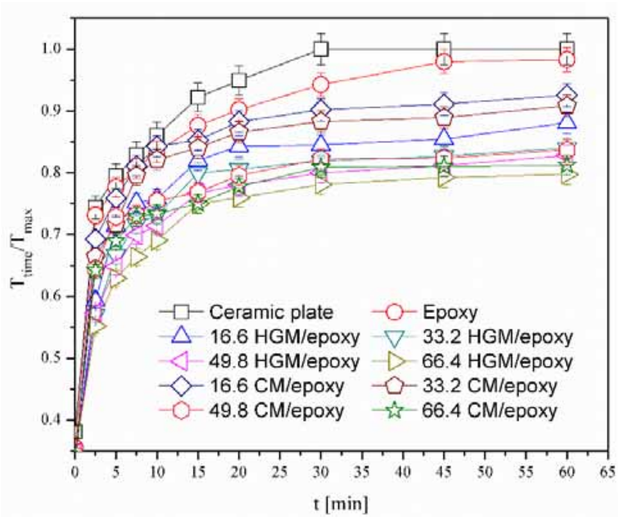


Figure 4. The relative ratio of surface and oil bath temperature versus time for the initial concentration of HGM and CM particles in epoxy coating.

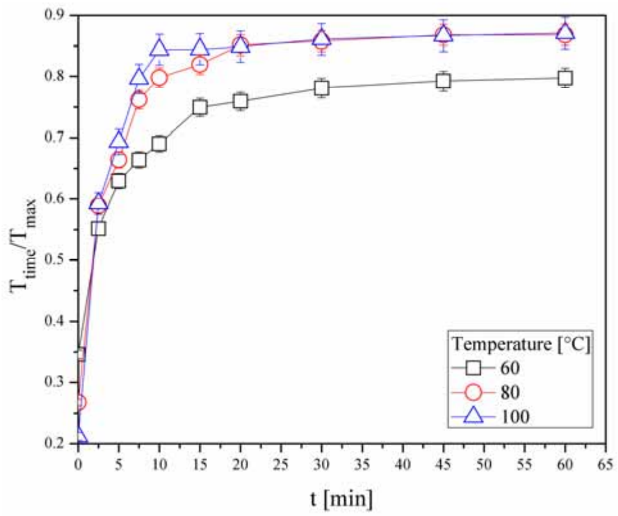


Figure 5. The relative ratio of surface and oil bath temperature versus time for the heating temperature of HGM/epoxy coating. Initial particles concentration: 66.4 g/l.

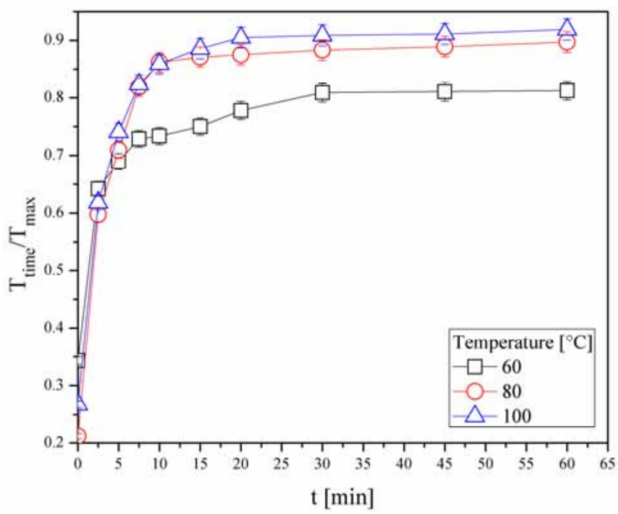


Figure 6. The relative ratio of surface and oil bath temperature versus time for the heating temperature of CM/epoxy coating. Initial particles concentration: 66.4 g/L.

Figures 5 and 6 confirms the good thermal insulation properties of coatings applied on ceramic plate. The temperature on the coating surface after 60 minutes of heating at 60, 80, and 100 ˚C was was lower for 20.26, 13.21, 12.91 %, respectively for HGM/epoxy coating. At the same heating temperatures, the lower thermal insulation efficiency was obtained using the CM/epoxy coating, the temperature on the surface was lower for 18.81, 10.29, and 8.11 %, respectively. The good thermal insulation properties of both materials can be attributed to the air "scavenged" inside the microspheres. The higher thermal insulation efficiency of HGM particles can be attributed to the better thermal insulation properties of glass compared to ceramics.

CONCLUSION

Here, epoxy coatings supplemented with HGM and CM particles were applied as coatings with thermal insulation properties in order to improve the camouflage characteristics in the infra-red range of electromagnetic spectra and physic and mechanical properties.

The SEM/EDS analysis was confirmed the expected particle morphology and distribution of diameters. The HGM/epoxy and CM/epoxy coatings adhere well to the ceramic surface and the microspheres are mostly immersed and protected by basic epoxy coating from mechanical damage.
The studies of thermal insulation properties of HGM/epoxy and CM/epoxy coatings shows that the addition of both types of particles improves the thermal insulation properties compared to non-coated and epoxy coated ceramic plates. Obtained results of thermal insulation properties at 60 ˚C, show that the HGM/epoxy coating has better properties than epoxy coating with some amount of CM particles. Also, the increasing heating temperature has a negative effect on thermal insulation properties.
Therefore, the application of HGM/epoxy and CM/epoxy coatings contributes to decreasing thermal radiation of the surface and the suppression of the thermal signature of the body.

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