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Aluminosilcate glass cenospheres as a component of cast magnesium matrix composites

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Abstract

The main characteristic of aluminosilicate glass cenospheres and their possible use as a component of magnesium matrix composites were presented. The results revealed that particular parts of cenospheres differ insignificantly from each other and it depends on which power plant cenospheres come from. The characteristic of cenospheres concerned their structure and phase composition and dimensions such as: wall thickness and size of pores occurring in walls, depending on cenospheres origin. Thickness of cenospheres walls and pore size were measured by using ImageJ computer software. The structure of aluminosilicate glass cenospheres and their phase composition have been investigated by scanning electron microscopy and X-ray diffraction (XRD). The possibility of cenospheres utilization in the fabrication of magnesium matrix composites was presented on the example of AZ91 magnesium alloy composite with fly ash cenospheres. Composite was fabricated by pressure infiltration method.

Key words: AZ91 magnesium alloy; Cenospheres; Metal matrix composites

1. Introduction

Cenospheres are in the size range of 1 to 500 μm in diameter, with closed-porous solid walls (the wall thickness is generally about 5-10% of cenospheres diameter), filled with nitrogen and carbon dioxide. The major chemical constituents of fly ash are SiO_2, Al_2O_3, Fe_2O_3, CaO, TiO_2. The phase-mineral composition of fly ash cenospheres commonly includes aluminosilicate glass, quartz, mullite, calcite, hematite, ferrite and alumina [1-3]. Fly ash cenospheres (FAs cenospheres) are also referred to as microspheres, hollow spheres, hollow ceramic microspheres or microballons. The main characteristic of cenospheres are: hollow spheres with spherical morphology, particles sizes ranging from sub-micron to millimeters in size, ultra low density, low thermal conductivity, resistant to acids, low water absorption, etc. It is estimated that cenospheres are present to an extent of about 1% fly ash from thermal plants [3-6]. Because of the low density and hollow nature cenospheres float in the lagoons, where fly ash is stored. The float is collected, further separation is carried by froth flotation and dried. Final selecting is carried out in a pneumatic conveying system connected to tribo electric separator. In the tribo separator the fly ash is divided into three fraction, namely cenospheres, carbon and iron particles. For many years, cenospheres have been widely used in various industries such as construction, automotive, paint industry and other industries [7-9].

In recent years, research on cenospheres as a reinforcement of metal matrix composites (MMCs) have been analyzed [10-13]. Therefore, fly ash cenospheres are potential particles used in metal matrix composites, since they are low-cost and low-density reinforcement available in large quantities as a waste by-product in thermal power plants. They can be used for the synthesis of ultra-light composite materials due to its significantly low density. The material costs of composites can be reduced by incorporating fly ash into the matrices of metallic alloys. Cenospheres as a reinforcement can also improve certain properties of obtained composites such as lower density, high specific strange, stiffness, strength to weight ratio [10-12].

Metal matrix composites with cenospheres have many advantages over monolithic alloys, for example lower density. One of these materials can be magnesium alloys. AZ91 magnesium alloy, due to its good properties, is one of the most widely used magnesium alloys [10, 11]. Composites with cenospheres can be fabricated by many fabrication methods, but the most known are die casting method and pressure infiltration method [10-14].

In the present work, the main characteristic of fly ash cenospheres were presented. Additionally, AZ91 magnesium alloy was adopted as a matrix alloy for the fabrication of light composite with cenospheres.

2. Experimental material and procedures

Two types of fly ash aluminosilicate glass cenospheres (FAs – fly ash cenospheres) depending on their origin were examined, namely FAs supplied by Eko-Export S.A. (Bielsko Biała, Poland) and Trade Industrial Company Rider (Kazakhstan) with the chemical composition listed in Table 1. Hollow aluminosilicate glass cenospheres, supplied by Eko-Export S.A., were used as a component of composite fabrication. The size of cenospheres used was in the range of 63÷125 μm. AZ91 cast magnesium alloy, with nominal composition given in Table 2, fabricated by Hydro Magnesium Ltd., was chosen as a matrix alloy. AZ91 alloy composite filled with modified hollow fly ash cenospheres was fabricated by a pressure infiltration method.

Table 1. Chemical composition of aluminosilicate glass cenospheres

Chemical composition [wt.%]	Poland	Kazakhstan
SiO_2	53÷63	54÷65
Al_2O_3	21÷31	21÷39
Fe_2O_3	4.4÷5.6	1.6÷3.8
CaO	1.5÷2.5	0.7
MgO	0.3÷0.7	0.7÷2.3
K_2O+Na_2O	0.8÷1.6	0.3÷1.3
TiO_2	0.1÷0.7	-

Cenospheres were etched with hydrofluoric acid solution in order to show the porous and the small amount of crystalline structure in their aluminosilicate walls.

Microstructural examination of fly ash cenospheres and etched cenospheres was performed by means of JEOL JSM-6610LV scanning electron microscope (SEM).

Phase constitutions of the investigated cenospheres were analyzed by X-ray diffraction (XRD) using a Brucker D8 Advance diffractometer. $Cu_{K\alpha}$ X-ray radiation was used.

In order to measure thickness of walls and size of pores cenospheres were placed in epoxide resin, then obtained material was grinded and polished. After polishing pictures of ceonspheres walls were carried out by means of light microscopy (LM). Measurements were done by using ImageJ computer software [15]. Measurements were conducted with 95% confidence.

The specimen of fabricated composite was prepared by the standard technique of grinding and polishing followed by etching in a solution of 1% nitric acid in alcohol. The microstructure was characterized by a NEOPHOT-21 light microscope (LM).

Table 2. Chemical composition of AZ91 alloy according to ASTM B93-94

	Chemical composition [wt.%]*)						
Alloy	**Al**	**Zn**	**Mn**	**Si**	**Fe**	**Cu**	**Ni**
AZ91	8.5-9.5	0.45-0.9	0.17-0.04	max 0.05	max 0.005	max 0.003	max 0.002

*) Mg rest

3. Results and discussion

Fig. 1a shows a typical structure of used cenospheres. Hollow cenospheres size is in the range of 63÷125 μm and the mean size is 100 μm (Poland) and 92 μm (Kazakhstan). Cenospheres are characterized by a spherical shape. There is a small amount of cracked cenospheres (Fig. 1a). The fracture of cenosphere wall is shown in Fig. 1b. It was observed the occurrence of pores in the wall. Shape of pores is also close to sphere. It was caused during forming the ceramic hollow microsphere by the solidification of inorganic carbon residue around the gas molecules. The mean thickness of cenospheres wall and the mean pore size for separate groups from Poland and Kazakhstan are shown in Table 3.

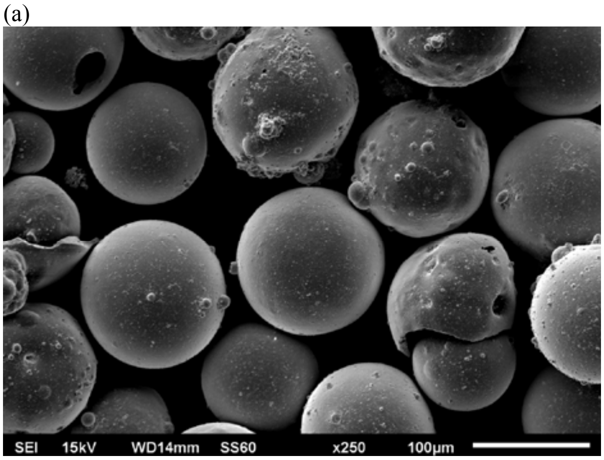
Table 3. The mean thickness of cenospheres wall and the mean pore size

	Mean thickness size [μm]	Mean pore size [μm]
Poland	6.4	4.6
Kazakhstan	5.6	2.9

Fig. 2 shows the structure of cenosphere wall with revealed pores and crystalline phases. In order to reveal the small amount of crystalline structure in cenospheres walls FAs were etched with hydrofluoric acid solution. The surface of cenospheres wall consisted mostly from aluminosilicate glass (amorphous phase) was dissolved in the solution and crystalline phases remained. This crystalline phase was identified as mullite. There are also visible some pores in the cenospheres wall.

In order to identify the existing phases in the cenospheres XRD analysis was performed. Fig. 3 shows a typical X-ray diffraction pattern of aluminosilicate glass cenospheres. The diffraction lines were indexed as arising from three different phases. It can be seen that besides the amorphous phase, there are reflexes from mullite, quartz and calcite. No ferrite, alumina or hematite phases were revealed in cenospheres.

(a)



(b)

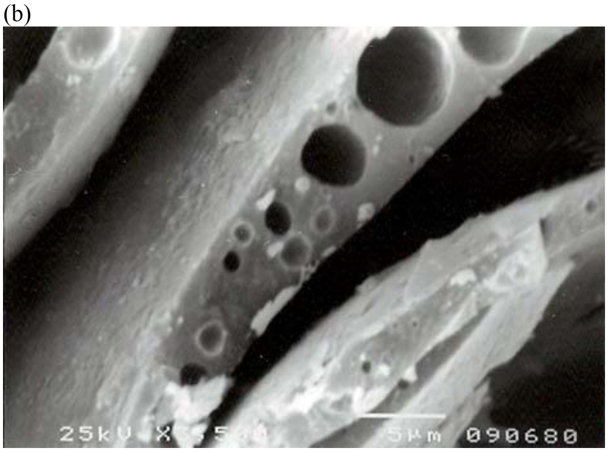


Fig. 1. Structure of cenospheres a) typical cenospheres, b) the fracture of cenospheres wall ; SEM

The microstructure of AZ91 magnesium alloy composite with 60% vol. fly ash cenospheres is shown in Fig. 4. The microstructure of obtained composite is characterized by homogeneous structure with uncracked particles in whole volume of obtained material. The microstructure of matrix consists mainly of solid solution of aluminium in magnesium (α phase) and α + γ eutectic (where γ is the intermetallic compound $Mg_{17}Al_{12}$).

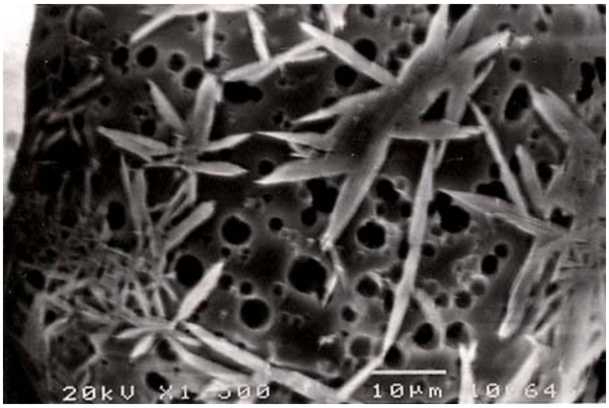


Fig. 2. Structure of cenosphere with revealed pores and crystalline phase; SEM

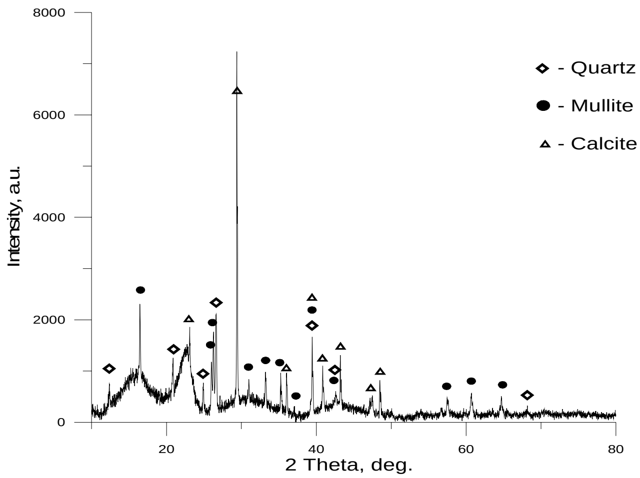


Fig. 3. X-ray diffraction pattern of the aluminosilcate glass cenospheres

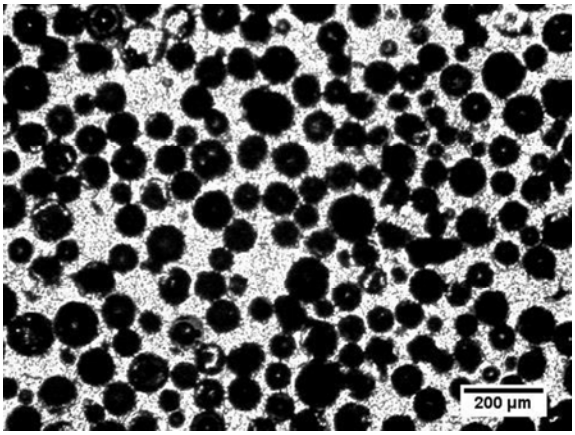


Fig. 4. Microstructure of fabricated AZ91 magnesium alloy composite with aluminosilicate glass cenospheres; LM

4. Summary

The aluminosilicate glass cenospheres characteristics and the possibility of use cenospheres as a component of magnesium matrix composites were presented. The results showed that particular parts of cenospheres differ insignificantly from each other and it depends on which power plant cenospheres come from. The investigation using hydrofluoric solution and XRD analysis revealed that cenospheres have crystalline phases in their walls structure besides amorphous aluminosilicate glass. The uniform and homogeneous microstructure of AZ91 composite filled with cenospheres testified that it is a feasibility of fabrication magnesium matrix composites with cenospheres as a component.

Literature:

[1] Vassilev V. S., Menendez R., Diaz-Somoano M., Martinez-Tarazona R. M.: Phase-mineral and chemical composition of coal fly ashes as a basis for their multicomponent utilization. 2. Characterization of ceramic cenosphere ans salt concentrates, Fuel, 2004, Vol. 83, p. 585-603.

[2] Anshits N. N., Vereshchagina T. A., Bayukov O. A., Salanov A. N., Anshits A. G.: The nature of nanoparticles of crystalline phases in cenospheres and morphology of their shells, Glass Physics and Chemistry, 2005, Vol. 31, No. 3, p. 306-315.

[3] Wang A., Zhang Ch., Sun W.: Fly ash effects: I. The morphological effect of fly ash, Cement and Concrete Research, 2003, Vol. 33, p. 2023–2029.

[4] Kutchko B. G., Kim A. K.: Fly ash characterization by SEM–EDS, Fuel, 2006, Vol. 85, p. 2537–2544.

[5] Sobczak J., Wojciechowski A., Boyko L., Drenchev P., Darłak P., Dudek P.: Materiały wysokoporowate, Wydawnictwo Instytutu Odlewnictwa, Kraków 2005.

[6] Pichór W.: Kierunki wykorzystania w budownictwie mikrosfer powstających jako uboczny produkt spalania węgla kamiennego, Materiały Ceramiczne, 2005, Vol. 4, p. 160-165.

[7] Bhattacharyya S., Donahoe R. J., Patel D.: Experimental study of chemical treatment of coal fly ash to reduce the mobility of priority trace elements, Fuel, 2009, Vol. 88, p. 1173–1184.

[8] Vassilev V. S., Menendez R., Alvarez D., Diaz-Somoano M., Martinez-Tarazona R. M.: Phase-mineral and chemical composition of coal fly ashes as a basis for their multicomponent utilization. 1. Characterization of feed coals and fly ashes, Fuel, 2003, Vol. 82, p. 1793-1811.

[9] Matsunaga T., Kim J.K., Hardcastle S., Rohatgi P.K.: Crystallinity and selected properties of fly ash particles, Materials Science Engineering A, 2002, Vol. 325, p. 333–343.

[10] Rohatgi P. K., Daoud A., Schultz B. F., Puri T.: Microstructure and mechanical behavior of die casting AZ91D-Fly ash cenosphere composites, Composites Part A, 2009, Vol. 40, p. 883–896.

[11] Daoud A., Abou El-khair M. T., Abdel-Aziz M., Rohatgi P. K.: Fabrication, microstructure and compressive behavior of ZC63 Mg–microballoon foam composites, Composites Science and Technology, 2007, Vol. 67, p. 1842–1853.

[12] Huang Z., Yu S., Li M.: Microstructures and compressive properties of AZ91D/fly-ash cenospheres composites, Trans. Nonferrous Metals Society China, 2010, Vol. 20, p.458-462.

[13] Rohatgi P. K., Guo R. Q., Iksan H., Borchelt E. J., Asthana R.: Pressure infiltration technique for synthesis of aluminum-fly ash particulate composite, Materials Science and Engineering A, 1998, Vol. 244, p. 22-30.

[14] Kamieniak J., Żydek A., Braszczyńska-Malik K. N.: Możliwości wytwarzania kompozytów na osnowie stopu magnezu z mikrosferami glinokrzemianowymi, X Międzynarodowa Konferencja Naukowa „Nowe Technologie i Osiągnięcia w Metalurgii i Inżynierii Materiałowej", Częstochowa 2009, s. 194-197.

[15] Abramoff M.D., Magelhaes P.J., Ram S.J.: Image Processing with ImageJ, Biophotonics International, 2004, Vol. 11, 36-42.