Influence of magnetron sputtering spent energy on the cooper and stainless steel films thickness deposited on ceramic hollow spheres for syntactic foam composites production.

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Abstract

The two types of composite powders (CP) comprising of the ceramic hollow micro balloons with particle size 50-200 μm, (average particle size d_{50} is 175 μm) and metal - cooper and stainless steel (SS) film coating were obtained and discussed in the paper. The first one is CP, coated with Cu and second with SS were obtained by plasma vapour deposition (PVD) using a magnetron sputtering system. Investigated metal film thickness dependence of the spent sputtering energy. Magnetron sputtering conducted on vacuum machine equipped with special vibration device for continuous metal deposition on hollow particles. SEM backscattering images showed that the coating films were non-porous and uniform in thickness. By varying the sputtering rate, the coating thicknesses varied in the range of 0.4-2.5 μm for Cu films and 0.2-0.8 μm for SS films. A series of composite syntactic foam (SF) cylindrical shape samples were fabricated by spark plasma sintering (SPS), using constant pressure – 9,5 Mpa, 2,0 min of sintering time, sintering at temperature 1000°C for Cu-coated CP and 1100 °C for the stainless steel coated CP. Obtained samples of SF has apparent density 0.9-1.1 g/cm^3 range.

Introduction

The modern conditions of energy carriers market, continuous growth of constructive materials cost puts forward before engineers and material scientists new tasks to create new materials combining simultaneously many properties such as non-flammable, recyclable and lightweight. To these properties answer open-cell and closed-cell metal foams (MF), which have been used in functional and structural engineering applications [1]–[4]. The closed-cell metal foams, have been used in structural engineering applications (e.g., automotive, aerospace, industrial equipment and building construction) that require lightweight structures with high strength-to-weight and stiffness-to-weight ratios, high impact energy absorbing capacity and/or with a good damping of noise and vibration [1], [4]. The syntactic foam is a part of class of closed cell metal foams. The main distingtion from the MF is presence of an additional phase - a material separating a matrix and pore.

Usually glass or ceramic hollow micro balloons (HMB) were used for FS production. It is widely investigated in with variouse material combinations: metal matrix - glass HMB [5] , metal matrix - ceramics HMB [6], [7], polymer resin matrix – glass HMB [8]. However, during high-temperature treatment (at metal matrix sintering), where is metal melts, poor metal-ceramic interface, called by low wetting ceramic by the metal ability take a place. To prevent such effect authors proposed to coat ceramic HMB by initial metal layer. One of the low-temperature metal deposition method is plasma vapor deposition PVD. In works [9]–[11] cenospheres (CS) were successfully coated by Cu, Ag, Ni. PVD process is more beneficial in comparision with other solvents based methods (precipitation, and spray coating), the coating is more uniform and not necessary CS pretreatment [12] However in literature is not described deposited dependence metal layer thickness from magnetron sputtering energy consumption. The novelty of the present research is to proof a possibility to make minimal metal film thickness, but at the same time, enough for binding coated particles without matrix material, but using only sintering.

Experimental Details

Alumina-silica ceramic hollow microspheres – cenospheres (CS) were obtained from fly ash of coal combustion at a thermal-power plant. Now, the CS is demanded in various industry industries, and available a commercial product. Particularly this CS supplied by *Biothecha Ltd.* (Latvia), they mainly contain SiO_2 and Al_2O_3 (totally more than 80%) full composition and morphology could be find in work [].

Metallization of CS was made on semi-industrial PVD machine on Sidrabe Inc. (Latvia) Fig.1.

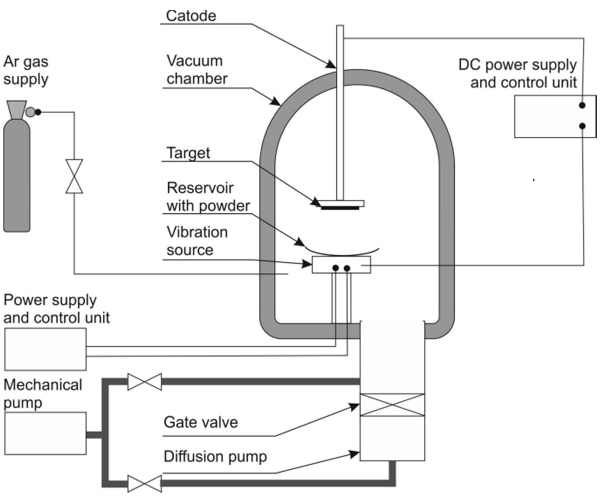

Fig. 1.Vacuum machine diagram.

Vacuum chamber was equipped with vibrotransporting device and magnetron sputtering system. Pumping system and system of gas-feeding provide receiving and maintenance of pressure in a vacuum chamber in the range of $2\cdot10^{-5} \div 1\cdot10^{-2}$ Torr.

The sputtering system includes the water-cooled magnetron placed on the top of vacuum chamber over a vibrohopper and the magnetron power supply "AE Pinnacle". Magnetron had planar Cu and SS target with 163 mm in diameter and initial thickness of 16 mm. The vibrotransporting system includes the water-cooled vibrohopper of a bowl-shaped look leaning on leaf spring springs, and the water-cooled electromagnet powered from sound frequency generator through the current amplifier. When giving alternating current on an electromagnet winding the vibrohopper makes the compelled quasitortional fluctuations. The parts of powder which are in a vibrohopper bowl receive mechanical influence as from a surface of constructive elements of a vibrohopper, and the contacting parts, next to them, and when imposing gravity move on very difficult trajectories that provides a forward rotary motion and intensive hashing of powder.

Deposition of copper layer CS was made as follows:

1. Portion of CS are loaded into a vibrohopper;
2. Vacuum chamber was pumped out up to the pressure of $2*10^{-5}$ Torr;
3. After receiving base pressure is switched gas system and by the flow of argon in chamber pressure in chamber increase up to value $1,2*10^{-3}$ Torr. This value of pressure kept constant during metal deposition;
4. The vibrosystem and the magnetron were switched on. Copper deposition was made at a power of 0,5 kW;
5. In 30 minutes the magnetron was switched off, but the vibrosystem remained worked;
6. In 30 minutes the magnetron for 30 minutes turned on again. The sequence of such actions repeated before "consumption" of the electric power on magnetron reached value of 6.25 kWh;
7. Process was interrupted, vacuum chamber was opened and small portion of powder captured for the first test;
8. Deposition process was continued before "consumption" by the electric power on magnetron had value of 12.5 kWh.

Three types of metal coated CS were obtained – Cu coated at 6.25 kWh, 12.5 spent energy and 12.5 kWh for SS deposition, designated as Cu@CS-6.25, Cu@CS-12.5 and SS@CS-12.5 respectively.

The CP bulk density was measured using Scott Volumeter. According to the following standard ISO 3923-2:1981; Metallic powders - Determination of apparent density (Part 2: Scott volumeter method). Measure was performed in 10 parallel measurements for each powder type.

Results and Discussion

120 g of CS was treated in a magnetron sputtering machine at 6.25 kWh and 12.5 kWh for coating by Cu samples (Cu@CS-6.25, Cu@CS-12.5) and 12.5 kWh by stainless steel (SS@CS-12.5).

Coating thickness.

Although density of CS wall (about 2.3±0.15 g/cm^3) and density of Cu and SS coating density (assuming no pores) are known, theoretical calculation of metal coating thickness is hardly predictable due to high deviation of sizes and irregular shapes of cenospheres. That's why microscopy investigation was done using a SEM. According to numerous measurements, the thickness of Cu layer in Cu@CS-6.25 sample vary from 0.4 µm to 1.2 µm, in Cu@CS-12.5 sample from 1.0 µm to 2.5 µm and SS film thicness 0.2-0.8 µm. CS wall thickness vary between 5µm and 15µm, the Cu coating adds from 3% to 30% to the wall thickness, 10% in average for sample Cu@CS-6.25 and 20% in average for the Cu@CS-12.5. The SS@CS-12.5 has 0.1-0.25 µm range thick coating is only, it is 3% from wall tickness. With aim to determinate real amount of deposited metals, powder bulk density were measured. Obtained composite powder Cu@CS-6.25 has bulk density 0.442±0.003 g/cm^3, Cu@CS-12.5 has bulk density 0.514±0.002 g/cm^3 , SS@CS-12.5 has 0.423±0.002 g/cm^3. And raw material (uncoated. CS) has bulk density 0.390±0.003 g/cm^3.,

Calculating weight increase it is +15,2%, +30,8% and +8,5% for Cu@CS-6.25, Cu@CS-12.5 and SS@CS-12.5 powders respectively.

For the Cu coating that data has good correlation with for sputtering spent energy 6.25 kWh and 12.50 kWh.

Coating morphology.

The SEM images fig. 2 show that Cu coating is uniform and non-porous. At Fig 2f, as darker point are visible CuO grains called by natural oxidation in air atmosphere. On the fig 3 is shown crossections of Cu@CS-6.25 and Cu@CS-12.5 samples. 3a and 3b images demonstrates complete coating of CS by Cu. Close look (fig 3c, 3d) shows thickness variation at the one particle from 0.7 µm to 1.4 µm for the Cu@CS-6.25 and from 1.0 µm to 2.6 µm for the Cu@CS-12.5 powders. Another situation take place with SS coating: it is less rough and less thick (fig 3f) and less thickness variation.

A series of composite SF cylindrical samples (diameter 19.5 mm, height 19.5 mm) were fabricated from CP by spark plasma sintering using constant pressure – 9,5 Mpa, 2,0 min of sintering time, sintering at temperature 1000°C for Cu@CS and 1100 °C for the SS@CS. Obtained samples has apparent density 0.9-1.1 g/cm^3 range, porosity 60-70% determinated by Archimedes method and compression strength 15-30 Mpa.

Cu@CS6.25 Cu@CS12.5

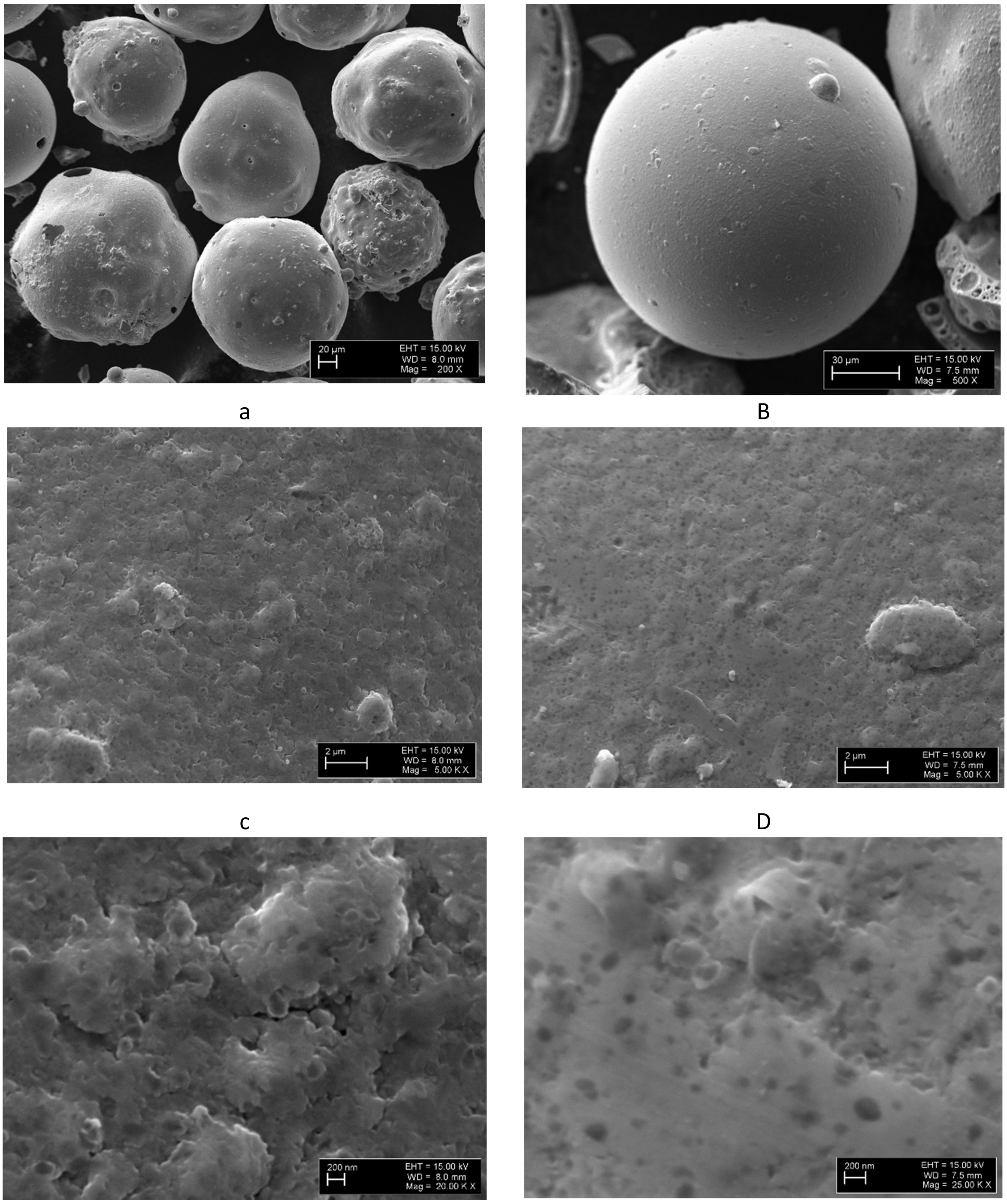

Fig 2. SEM backscattered electron analysis (BE) images of Cu@CS6.25 (a,c,e) and Cu@CS12.5 (b,d,f) surface at magnification: a)X200 b) X500 c)X 5000 d)X5000 e)20000 f)X25000 times.

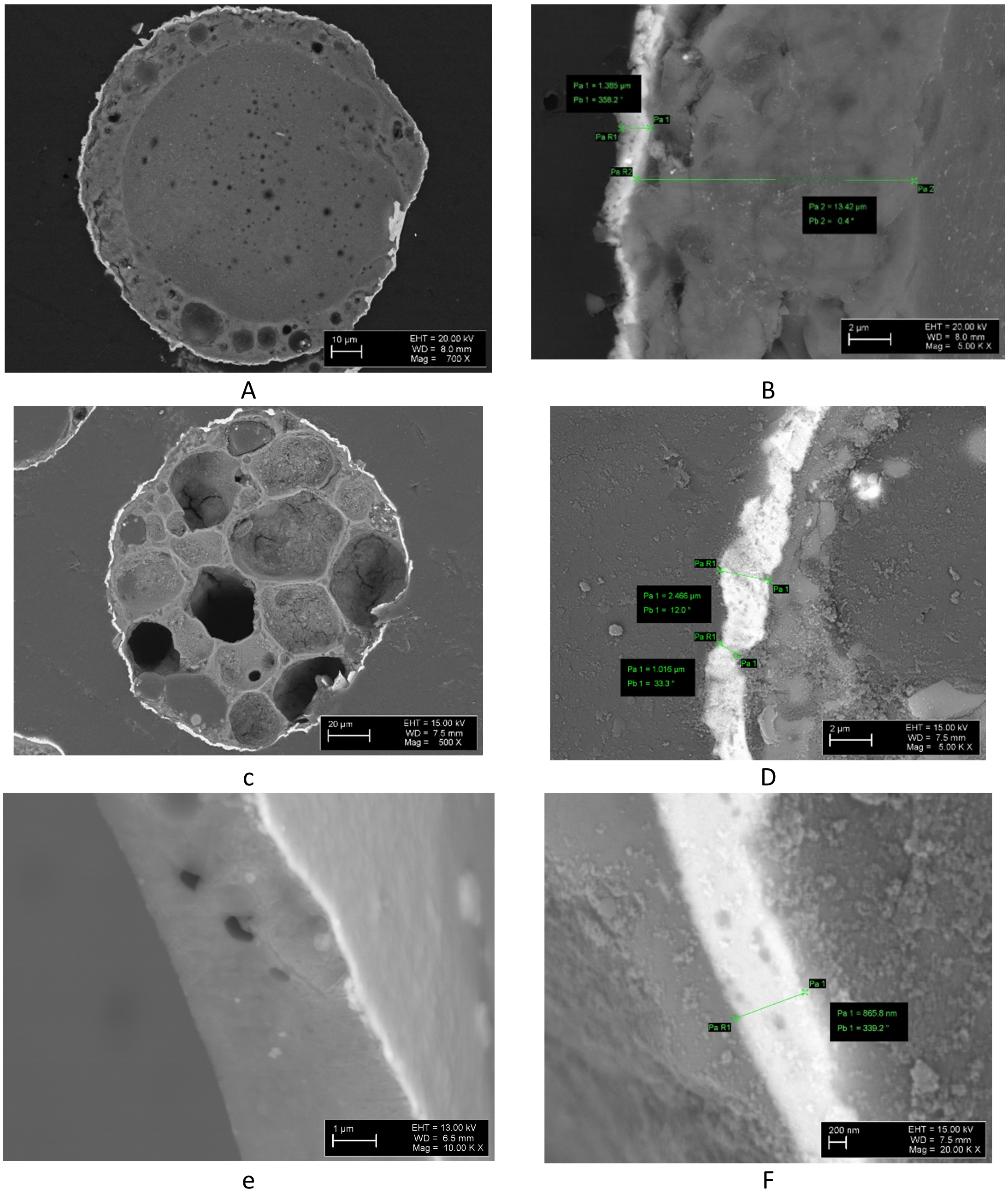

Fig 3. SEM BE images of cross-section of Cu@CS6.25 (a,b), Cu@CS12.5 (c,d) and SS@CS-12.5 (e,f) at magnification a)x700; b) x5000; c) x500; d) x5000; e) x10000; f) x20000.

Conclusions

Cu and stainless steel coated CS were fabricated by magnetron sputtering device equipped with vibrohooper. The deposited film thickness is proportional to the sputtering spent energy. However applying another metal using the same (12.5 kWh) sputtering energy was obtained different film thickness, particularly SS in comparison with Cu film thickness less almost threefold. SS coating has more smooth surface. Cu and SS coated CS is suitable for the SF composites producing. SS film coating with density 150-250 nm is enough to sinter (by SPS) mechanically strong (up to 30 MPa) composites.

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