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Microstructure of metal matrix composites reinforced by ceramic microballoons

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In Press: MAteriali in Technologije / Materials and Technology, accepted manuscript

Abstract

Metal matrix composites reinforced by ceramic hollow microspheres were produced as special porous metals, called metal matrix syntactic foams (MMSFs). In this paper the microstructure of the ceramic hollow microspheres as reinforcing element was investigated in connection with the production of MMSFs by pressure infiltration. SL150 and SL300 type ceramic microspheres from Envirospheres Ltd. (Australia) were investigated. They contained various oxide ceramics, mainly Al_2O_3 and SiO_2. The chemical composition and the microstructure of the microspheres had strong effect on their infiltration characteristics; therefore in the view of MMSF production it was very important to know microstructural details about the microspheres. Due to this energy dispersive X-ray spectroscopy maps were recorded from the cross sections of the microspheres' wall. The results showed that the Al_2O_3 and SiO_2 distribution was not equal; the Al_2O_3 phase was embedded in the surrounding mullite and SiO_2 phase in the form of needles. Line energy dispersive X-ray spectroscopy measurements were performed in order to investigate the possible reaction between the different aluminium alloy matrices and the ceramic microspheres. The results showed that, due to the uneven distribution of Al_2O_3 rich particles, the molten aluminium could reduce the SiO_2 rich parts of the microspheres and the wall of the hollow microspheres became damaged and degraded. This chemical reaction between the microspheres and the walls could make the infiltration easier, but the resulting mechanical properties will be lower due to the damaged microsphere walls.

Keywords: microsphere, microballoon, energy dispersive X-ray spectroscopy, metal matrix syntactic foam, metal matrix composite

1. Introduction

Nowadays metallic foams become more and more important and this is confirmed by the increasing number of papers published on this topic. The ‘conventional’ metallic foams, which contain metallic and gas phases only, have wide spread literature. However there are still existing problems for example in accordance to the foaming process of the foams [1, 2]. The metallic foams have a special class which satisfies the definition of particle reinforced metal matrix composites also. These are the metal matrix syntactic foams (MMSFs). The first of them was produced in the ‘90s. The MMSFs have numerous perspective applications as covers, hulls, castings, or in automotive and electromechanical industry sectors because of their high energy absorbing and damping capability. In these porous materials the porosity is ensured by incorporating ceramic hollow microspheres [3]. The microspheres are commercially available and they contain mainly various oxide ceramics [4, 5]. The quality of the microspheres has a strong effect on the mechanical and other properties of the foams. The most important properties of the foams are the compressive strength and the absorbed energy. Wu et al. [6] examined the effects of the microballoon size on the compressive strength. They found that smaller microspheres ensure higher compressive strength because they contain fewer flaws in their microstructure, than the larger ones. The damage propagation of the foams was also investigated. The fracture was initialized in the corners of the specimens by the shearing of the microspheres. Rohatgi et al. [7] also investigated the size effect of the microspheres, but not only in the view of compressive strength, but in the view of infiltration too. Their measurements showed that the larger microspheres can be infiltrated easier. Palmer et al. [8] proved that the larger microspheres contain more porosity in their wall and more flaws in their

microstructure, than the smaller ones. The results of the performed upsetting tests were compared to other works on this topic [9, 10]. The conclusions were the same. Balch et al. [11] performed a special upsetting test. The loading was applied in small steps and after each test X-ray or neutron diffraction measurements were carried out. The main aim of the work was to investigate the load transfer from the matrix to the microspheres. They found a chemical reaction between the microspheres and the matrix materials which has detrimental effect on the load transfer and through that on the mechanical properties of the foams. In their previous work Balch et al found that the microspheres have at least the same importance in the syntactic foams than the matrix material. Their fracture strength and the yield strength of the matrix determine the failure stress of the syntactic foams. Therefore the investigation of the microstructure and the quality of the microspheres is very important [9]. Besides the compressive strength other mechanical properties, such as the tensile strength, or the hardness of the syntactic foams were investigated [12, 13]. The sliding behavior of the syntactic foams was also examined because the ceramic microspheres have large hardness and therefore the composite show better wear behavior, than the pure matrix [14, 15].

As it can be seen in the previous paragraph the quality and chemical composition of the microspheres influence many properties of the syntactic foams. And they have also strong influence during the production of the syntactic foams. The foams are usually produced by mixing technique and gravitational casting or by pressure infiltration. In all cases the contact angles between the ceramic microspheres and the metal matrix have a detrimental effect on the infiltration characteristics and on the threshold pressure (in the case of pressure infiltration) [16, 17, 18]. The contact angle is influenced by many parameters and among them the chemical composition and the possible reaction

between the reinforcement and the matrix material. Therefore the microspheres should be precisely investigated on the microstructure's scale.

The ultimate method for this purpose are scanning electron microscopy and energy dispersive X-ray spectroscopy (EDS). EDS is sensitive to the chemical composition and able to investigate a point, a line or even an area. These possibilities give extremely good opportunities to get detailed information about the microstructure of the microspheres and about the distribution of their constituents. According to the published works mentioned above the main aims of this work were to investigate the microstructure and the distribution of the constituents in ceramic hollow microspheres and to investigate the interface between the different aluminium alloy matrices and the ceramic microspheres in order to provide information for MMSF production and on their expectable mechanical behavior.

2. Materials and methods

The investigated materials were SL150 and SL300 type microspheres provided by Envirospheres Ltd. (Australia) [4]. Their main parameters are listed in Table 1. The phase composition was determined by X-ray diffraction measurements. For this purpose a Phillips X-Pert type diffractometer with 35 mA cathode heating current and copper anode (CuKα, λ=0.154186 nm) with 40 kV voltage was used. The rotating speed of goniometer was 0.04 degree/s. The Scanning Electron Microscope (SEM) tests were performed by a Phillips XL-30 type electron microscope equipped with an EDAX Genesis EDS analyzer. The ceramic microspheres were coated with carbon in order to get a conductive layer on them, but basically they were investigated in as received condition. The excitation was 15 kV and EDS maps were recorded from the surface of

the microspheres. Five typical microspheres were investigated from each SL type microsphere group.

Later the microspheres were incorporated in pure aluminium (Al99.5), aluminium-silicon (AlSi12), aluminium-magnesium-silicon (AlMgSi) or aluminium-copper (AlCu5) alloys to create MMSFs. The foams were designated according to their matrix and reinforcement. For example Al99.5-SL150 denotes pure aluminium matrix syntactic foam with SL150 microsphere reinforcement. The volume fraction of the microspheres was maintained at relatively high (~60 vol%) level and the production method is described in details elsewhere [3]. The density and porosity values of the MMSFs were listed in Table 2, while the chemical compositions of the MMSFs are shown in Table 3. In Table 2 the theoretical density and particle porosity was calculated from the geometrical parameters of the microspheres. The matrix porosities were calculated as the difference between theoretical and measured density divided by the theoretical density. The negative matrix porosity refers to infiltrated microspheres (the particle porosity should be decreased). However, the values of matrix porosity are always remained below 8%, so the infiltration can be qualified as good enough. The values of Table 3 were determined by XRD measurements described above. Line EDS measurements were carried out on the foams to characterize the elemental distribution at the interfaces, where the hollow microspheres are in contact with the matrix. All EDS line analysis were performed on metallographically polished surfaces. The polishing was performed on cross sections by standard metallographic methods. The excitation voltage for the EDS analysis was 20 kV. The line EDS measurements started from the matrix materials and crossed the wall of the hollow sphere. One hundred points were

measured along each line; each point was excited for 20 s with 35 μs detector acquisition rate.

3. Results and discussion

3.1. Investigation of the microspheres' walls (map EDS measurements)

In Fig. 1 a typical site from the outer surface of an SL150 type microsphere is shown, while in Fig. 2 the SEM micrograph from the cross section of a microsphere in Al99.5 matrix foam sample is presented. In both images needle-like structures can be clearly observed. They are densely situated and they do not have any distinguished direction. The different gray scale of the needles on the back-scattered electron (BSE) images indicates somewhat different chemical composition. The needles are very small, their length is between 5 and 10 μm, while their diameter is lower than 0.5 μm. EDS maps give much more useful information than single EDS spot measurements, because single spots can be largely effected by surrounding matrix. This effect can be decreased or avoided by EDS maps, in which case the results can show the distribution of the elements.

Because of this reason EDS map analysis were performed on the cross sections of the microspheres to get additional information about the element distribution in the microsphere's wall. For this analysis the EDS maps of a SL300 type microsphere are presented as example (Fig. 3). It is important to emphasize that all of the other microsphere types (SL150 and SL300) showed the same features. Fig. 3a shows the SEM image of the investigated surface. The needle-like structure can be again well observed. Fig 3b shows the distribution of aluminium. It is evident that the needles

contain more Al than the surrounding environment, but Al can be found everywhere, not only in the needles. From the XRD measurements it is also known that Al_2O_3 and SiO_2 forms mullite ($3Al_2O_3 \cdot 2SiO_2$), therefore – in accordance to the EDS map and Table 1 – the wall of the microspheres is built up from the mixture of mullite and amorphous SiO_2. This means that the distribution of Al_2O_3 is uneven, it can be found as the part of mullite in the wall and as Al_2O_3 needles embedded in this wall matrix. Fig 3d definitely confirms this conclusion by showing the distribution of the silicon. Si can be found everywhere on the surface (mainly amorphous SiO_2 mixed with mullite) except in the needles (the needles appear black in this picture). This indicates that, the needles do not contain Si and therefore they are really Al_2O_3 needles. Finally, as it is expected the oxygen distribution (in Fig. 3c) is totally balanced, it is built in the Al_2O_3 and SiO_2 also. The analysis above show the formation of Al_2O_3 rich zones and it indicates the presence of amorphous SiO_2 rich zones too. The amorphous SiO_2 is undesirable, because – opposite to Al_2O_3 and mullite – the chemical stability of SiO_2 at elevated temperature is not good enough. During the production of MMSFs the molten aluminium can reduce the SiO_2 according to the following chemical reaction:

$$4Al_{(liq)} + 3SiO_{2(sol)} \rightarrow 2Al_2O_{3(sol)} + 3Si_{(sol)} \quad (1)$$

At first sight this reaction is advantageous, because it forms Al_2O_3 (with better properties) from amorphous SiO_2. But this is a diffusion controlled reaction and leads to the degradation of the microspheres' wall as it is shown in Fig. 4a. This indicates drastic drop in the compressive strength and other mechanical properties as it is shown in previous papers [3, 20, 21]. As the XRD results of Table 3 shows it, the reaction produces mainly γ-Al_2O_3 and it took place only in the case of pure aluminium matrix reinforced with SL150 and SL300 type microspheres. In the case of SL300 type

microspheres the reaction was suppressed by the lower infiltration temperature. The infiltration temperature had a strong influence on the kinetics of the change reaction. That is why there was no reaction in the case of the Al99.5-SL300 type syntactic foams infiltrated at lower (690 °C) temperature [3]. This indicates that there is a limit relating to the temperature. Above this temperature the reaction is intensive, but below the temperature limit, the reaction will not occur (for details see [3]). The reaction did not take place in the case of AlSi12 matrix material; because the driving force of the diffusion controlled chemical reaction is the Si difference between the molten matrix and the solid microspheres. In the case of AlSi12 matrix the large Si content decreased the driving force; the reaction became suppressed and did not take place. The walls of the microspheres remained unharmed as it can be observed in Fig. 4b. The most γ-Al_2O_3 was found in the MMSFs with AlMgSi1 matrix and the above mentioned reaction also took place in the samples with AlCu5 matrix. In the case of the MMSFs with AlCu5 matrix the thermodynamic conditions enabled to form $CuAl_2$ phase also.

In summary the microspheres built up from Al_2O_3 needles embedded in the mixture of amorphous SiO_2 and mullite. The chemically reactive molten aluminium can damage the microspheres by reducing their amorphous SiO_2 rich parts.

3.2. Investigation of the interface layer between microspheres and matrix

After the detailed examination of the microspheres' surfaces, the investigations of the MMSFs were carried out.

First, overview map EDS measurements were done in the microsphere-matrix region of the MMSFs with different matrices. For example in the case of Al99.5 matrix with SL300 microspheres the map EDS results are shown in Fig. 5. The area of the SEM image (Fig. 5a) was investigated for the distribution of the alloying elements (Fig. 5b -

Fig. 5d). The microstructure of the matrices grain boundaries after solidification can be seen clearly in the concentration differences between the aluminium (Fig. 5b) and silicon (Fig. 5d). Oxygen in higher concentration was only detected in the walls of the microspheres (Fig. 5c) for all of the samples respectively. In the case of the AlSi12 matrix larger areas of primer silicon were detected as expected. In the samples with AlCu5 matrix copper rich precipitations can be observed (~1 μm × 20 μm) according to XRD measurements they are $CuAl_2$ particles. In the samples with AlMgSi1 matrix magnesium showed uniform distribution in the aluminium areas, and no magnesium was detected in the primer silicon precipitations. Magnesium enrichments were detected alongside the microspheres outer walls which indicate the solution of magnesium into the microspheres wall. To confirm the facts described above and to get more detailed information about the microsphere interface-matrix region line EDS measurements were done at higher resolution.

EDS line measurements were performed on polished specimens perpendicular to the interface layer between the microspheres and the matrix material. The interface layer is very important, because that is responsible for the load transfer from the matrix to the microspheres. The line-scan profiles showed the alternation of chemical elements along the measurement lines. The most important benefit of the EDS line method is that this offers a very good opportunity to examine the interface layer and the changes in the hollow microsphere wall in the matrix. Examples for the AlMgSi1 and the AlCu5 matrices are shown in Figs. 6 and 7, respectively.

In Fig. 6 the wall of an SL150 type hollow microsphere can be observed in high magnification BSE image. It can be seen that the outer edge of the wall is not very well defined. This means that the surface of the microsphere is degraded. As mentioned

above, the exchange reaction produces Al_2O_3, which is advantageous, but not at a price of degrading the hollow microsphere's wall. The diffusion process made a relatively wide interface layer on the outer surface of the hollow microspheres (i.e. from point B to point C). The width of the interface layer was relatively wide ~2-4 μm (in previous works 6 μm was also obsereved [21]) and it was measured between the significant changes of the derivation of the fitted curves showing the changing of the Al. Along the interface the Si and Al contents increased and decreased, respectively, with a moderate slope. After point C the Al content was alternated according to the actual composition of the wall. From point D the measurement is not reliable because of the curvature of the inner surface of the hollow microsphere.

In all of the aluminium and Al-alloy matrices the alternation of the Al and Si content were observed, because of the presence of primer Si precipitations in the alloy. In the case of AlMgSi1 matrix (Fig. 6) such primer Si precipitation can be seen between points A and B. Points B and C were close to each other. This indicates that there is a very narrow (i.e. less than 3 μm) observable interface layer. In the concentration-sensitive BSE image, the lighter needle-like phases can be observed again in the wall of SL150 spheres. The increase of the Mg content after point B confirms magnesium solution into the microspheres wall in less than 4 μm depth (this was also indicated by the map EDS measurements). In the case of AlCu5 matrix (Fig. 7) between points A and B a precipitation ($CuAl_2$ phase) can be observed, the interface layer was about 3 μm wide in both SL150 and SL300 cases respectively. Parts of the microspheres' wall were covered in Cu precipitations, which can be seen clearly on the BSE image, but according to the line EDS measurement (between point B and C) this is not a solution in the microspheres' wall, just a precipitation on it. In the case of AlSi12 matrix the outer

surface of the microspheres were seemingly unharmed as the exchange reaction was suppressed by the large Si content of the matrix. The interface layer was less than 1.5 μm for both SL type microspheres respectively.

According to this, a well-defined Al and Si alternation occurred in the line of EDS analysis diagram. In the lighter areas, the Si content decreased while the Al content increased. There was no large fluctuation in the oxygen content within the matrix and within the microspheres walls. This implies that again lighter phases in the wall of hollow microspheres are Al_2O_3 particles embedded in SiO_2 and mullite matrix. At point D, on the inner side of the hollow microsphere wall, a very narrow Fe, Mg and K rich zone can be observed (originated from the various oxides of the hollow microspheres). Considering the possibility of chemical reactions between the microspheres and the matrix materials, it is worth mentioning that, the ceramic microspheres can be used as a source of alloying elements in MMSF systems. The appropriate choice and concentration of alloying element in the wall material or in a surface coating [8] of the microspheres can result in precipitation, grain refinement, and microstructure as per the designed scheme. These alloying elements can enhance the mechanical and/or other properties of the MMSFs. However, there is an effective range of the alloying due to the limited time after infiltration to cooling. If the effective distance between the microspheres is small enough, it is possible to guarantee a homogenous microstructure. If the distance is larger, then the alloying has effects only in the vicinity of the microspheres.

In summary EDS line measurements are applicable to investigate the interface layer between the microspheres and the matrix material. The investigations proved the presence of Al_2O_3 needles in the wall and the exchange reaction between the SiO_2

content of the microsphere and the molten aluminium during the production. The suppressing effect of high Si content was also confirmed. Alloying or coating of the microspheres offers a great opportunity to influence the microstructure and the properties of the MMSFs.

The authors assume that the microspheres also have an effect on the orientation and size of the matrix materials grains. Therefore a following paper will deal with the electron backscatter diffraction (EBSD) investigation of MMSFs.

4. Conclusions

From the results of the above mentioned and discussed EDS and XRD measurements the following conclusions can be drawn:

- The microstructures of the microspheres, their volume fraction and properties have strong effect on the properties of the MMSFs. SEM and EDS investigations showed that in the wall of the hollow ceramic microspheres Al_2O_3 needles can be found. These needles are packed densely and they are embedded in the mixture of mullite and SiO_2.
- Due to the uneven distribution of Al_2O_3, SiO_2 rich zones were formed at the surface of the microspheres. During the MMSF production the molten aluminium chemically attacked these zones and this reductive chemical reaction resulted in severe damage of the microspheres' wall.
- In the case of Al99.5, AlCu5 and AlMgSi1 matrices the reaction was intensive. The driving force of the diffusion controlled reaction was the Si concentration gradient between the microspheres and the matrix. In the case of AlSi12 matrix

syntactic foams, the reaction was suppressed by the considerable amount of the Si in the matrix alloy.

- In the case of AlCu5 matrix local copper precipitations were found on the microspheres walls while in the case of AlMgSi1 matrix magnesium solved into the outer region of the microspheres' walls.

Besides the concrete conclusions above it is worth to mention here that the ceramic microspheres can be used as a source of alloying elements in MMSF systems. The appropriate choice and concentration of alloying element in the wall material or in a surface coating of the microspheres can enhance the properties of the MMSFs.

Acknowledgements

Special thanks to Professor J.T. Blucher for his kind support and to I. Sajó for XRD measurements. This paper was supported by the János Bolyai Research Scholarship of the Hungarian Academy of Sciences. The investigations were supported by The Hungarian Research Fund, NKTH-OTKA PD 83687. This work is connected to the scientific program of the " Development of quality-oriented and harmonized R+D+I strategy and functional model at BME" project. This project is supported by the New Széchenyi Plan (Project ID: TÁMOP-4.2.1/B-09/1/KMR-2010-0002).

Tables

Table 1. Morphological properties and phase constitution of the applied hollow ceramic spheres

Type	Average diameter (μm)	Size range (μm)	Specific surface (μm^{-1})	Al_2O_3	Amorphous SiO_2	Mullite	Quartz
				wt%			
SL150	100	56-183	0.060	30-35	45-50	19	1
SL300	150	101-330	0.040				

Table 2. Calculated and measured density and porosity values of the MMSFs [3]

Specimen	Density (gcm^{-3})		Porosity (%)		
	Theoretical	Measured	Particle	Matrix	Total
Al99.5-SL150	1.34	1.43	50.9	-6.2	44.7
Al99.5-SL300	1.42	1.52	48.2	-7.2	41.0
AlSi12-SL150	1.32	1.31	50.9	1.1	52.0
AlSi12-SL300	1.40	1.37	48.2	1.9	50.1
AlMgSi1-SL150	1.34	1.52	50.9	-13.4	37.5
AlMgSi1-SL300	1.42	1.57	48.2	-10.5	37.7
AlCu5-SL150	1.37	1.53	50.9	-11.6	39.3
AlCu5-SL300	1.44	1.62	48.2	-12.2	36.0

Table 3. Phase constitution of aluminium matrix syntactic foams according to XRD measurements (wt%)

Specimen	Al	Si	Mullite	α-Al_2O_3	γ-Al_2O_3	Amorphous	$CuAl_2$
Al99.5-SL150	67	8	11	3	11	0	-
Al99.5-SL300	78	0	11	0	0	11	-
AlSi12-SL150	72	7	13	0	0	8	-
AlSi12-SL300	72	7	12	0	0	8	-
AlMgSi1-SL150	60	7	8	0	25	0	-
AlMgSi1-SL300	60	6	6	0	28	0	-
AlCu5-SL150	60	6	8	8	12	0	6
AlCu5-SL300	60	5	10	7	12	0	6

Figures

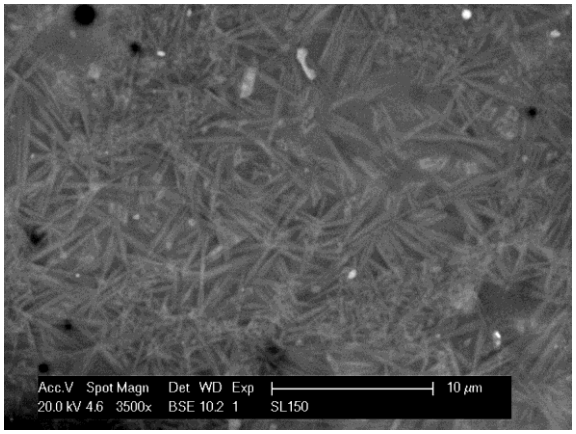

Fig. 1 SEM image from the surface of a SL150 type ceramic hollow microsphere.

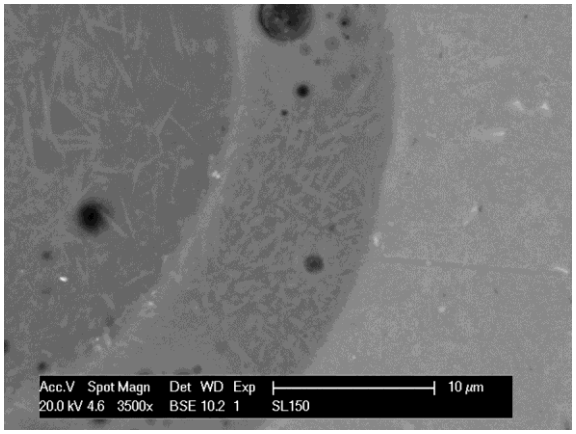

Fig. 2 SEM image from the cross section of a SL150 type ceramic hollow microsphere's wall.

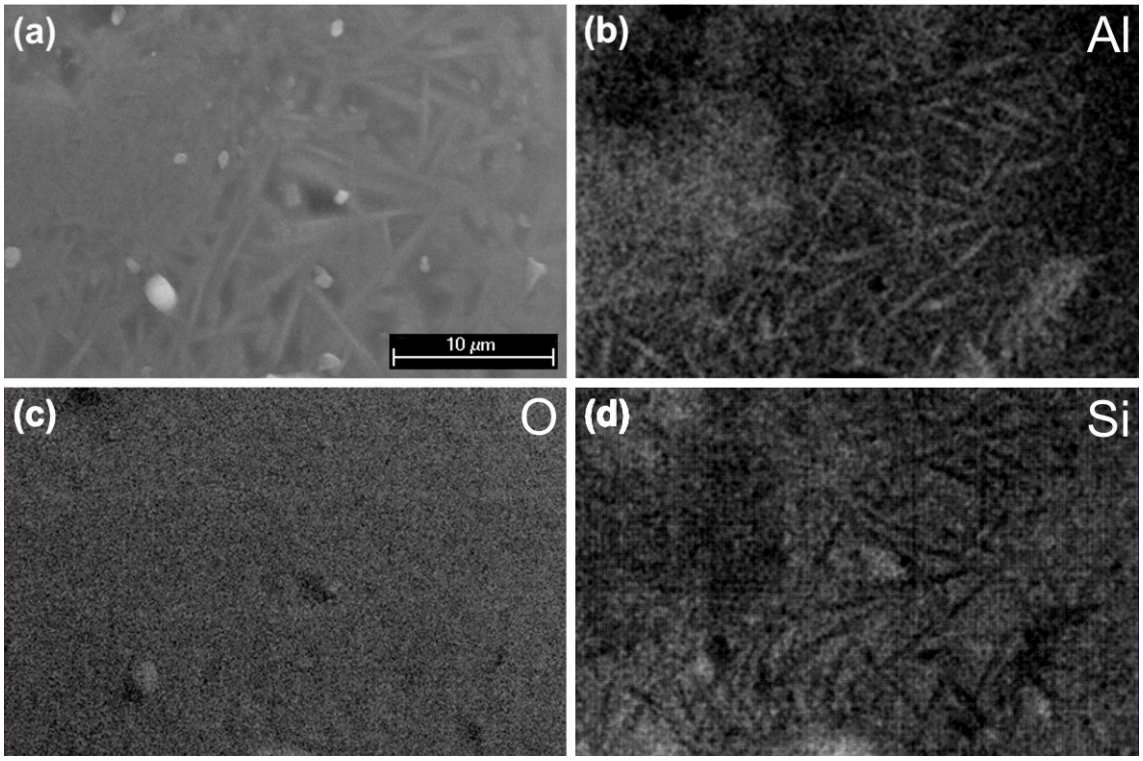

Fig. 3 SEM image from the surface of a SL300 type microsphere (a) and EDS maps of this area for the element distributions of: Al (b), O (c) and Si (d).

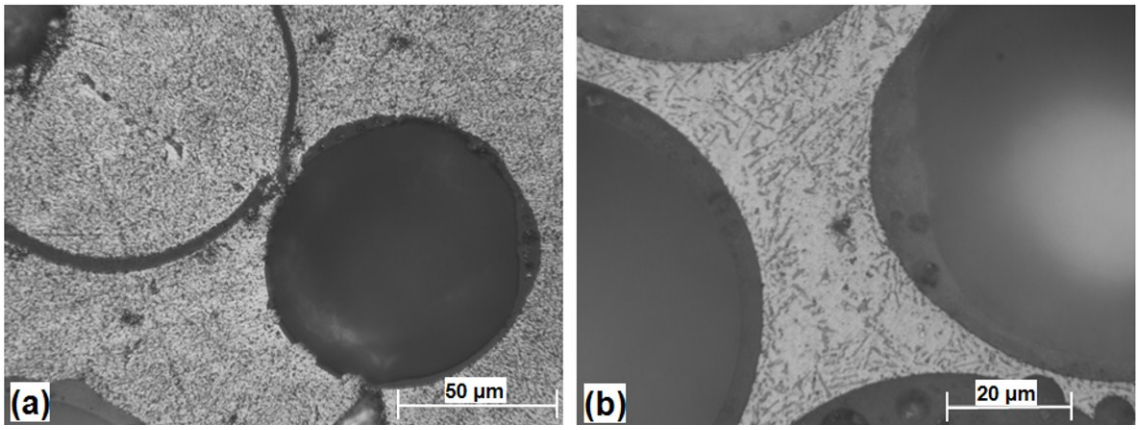

Fig. 4 Micrographs from a damaged (a) and an undamaged (b) microsphere in Al99.5 (a) and AlSi12 (b) matrix.

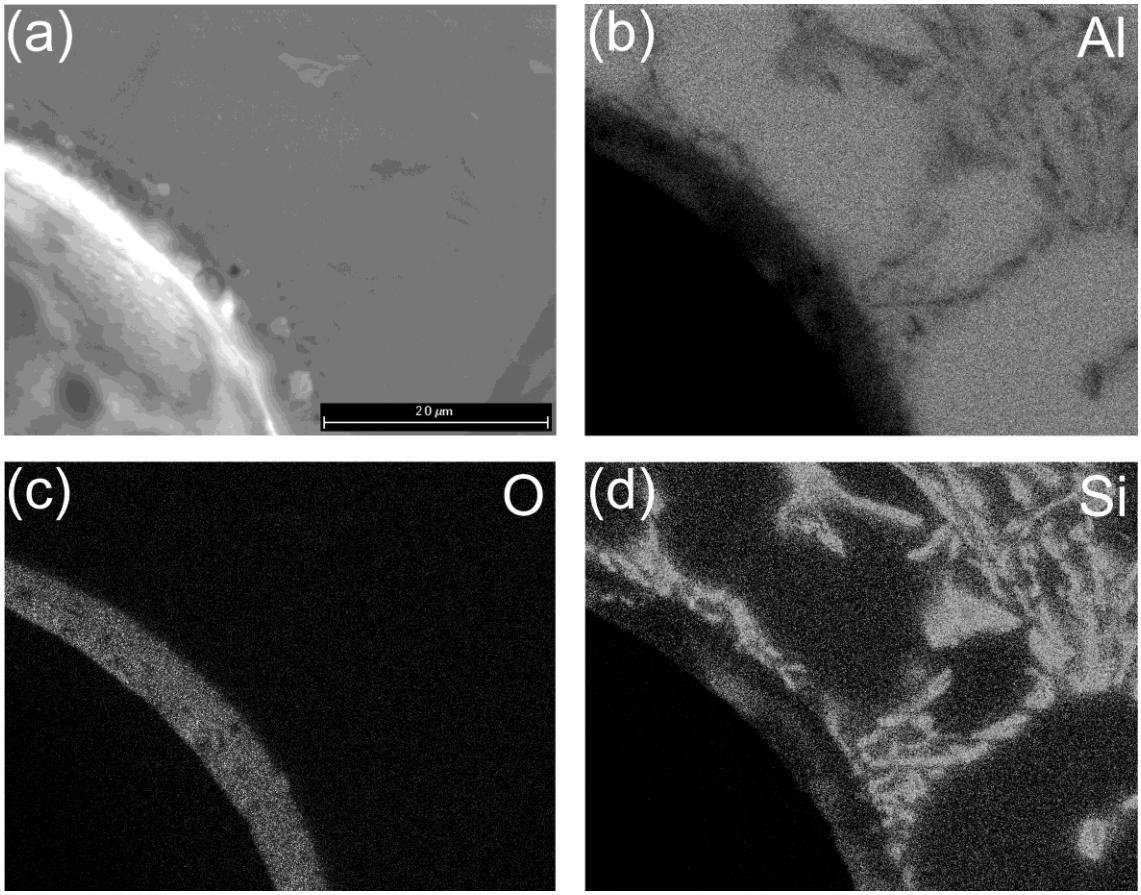

Fig. 5 SEM image from an Al99.5 matrix MMSF with SL300 type microsphere (a) and EDS maps of this area for the element distributions of: Al (b), O (c) and Si (d).

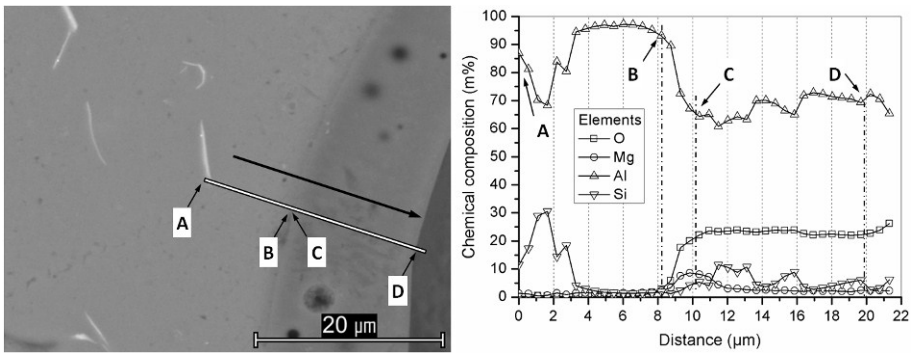

Fig. 6 BSE image and EDS line-scan profiles of the AlMgSi-SL150 syntactic foam

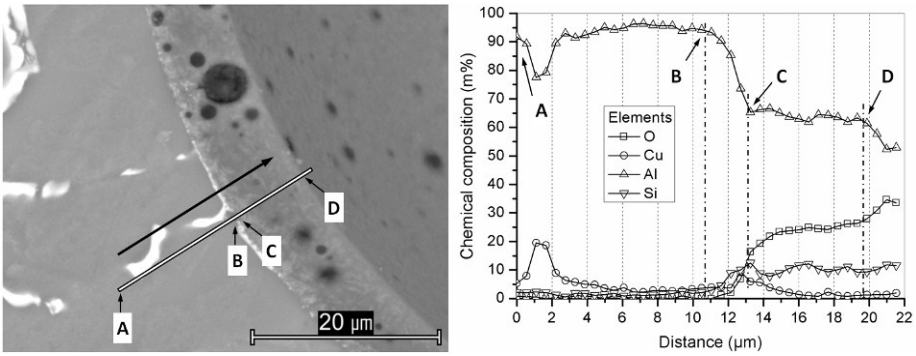

Fig. 7 BSE image and EDS line-scan profiles of the AlCu5-SL300 syntactic foams

Figure captions

Fig. 1 SEM image from the surface of a SL150 type ceramic hollow microsphere.

Fig. 2 SEM image from the cross section of a SL150 type ceramic hollow microsphere's wall.

Fig. 3 SEM image from the surface of a SL300 type microsphere (a) and EDS maps of this area for the element distributions of: Al (b), O (c) and Si (d).

Fig. 4 Micrographs from a damaged (a) and an undamaged (b) microsphere in Al99.5 (a) and AlSi12 (b) matrix.

Fig. 5 SEM image from an Al99.5 matrix MMSF with SL300 type microsphere (a) and EDS maps of this area for the element distributions of: Al (b), O (c) and Si (d).

Fig. 6 BSE image and EDS line-scan profiles of the AlMgSi-SL150 syntactic foam

Fig. 7 BSE image and EDS line-scan profiles of the AlCu5-SL300 syntactic foams

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