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Quality of the recovered metal matrix as a measure of the efficiency of the MMC recycling process

Katarzyna Gawdzińska[1], Dorota Nagolska[2], Agnieszka Kochmańska[3]

[1] Maritime University of Szczecin, Institute of Basic Technical Sciences
70-205 Szczecin, ul. Podgórna 51/53, e-mail: k.gawdzinska@am.szczecin.pl
[2] Poznań University of Technology, Institute of Materials Technology
61-138 Poznań, ul. Piotrowo 3, e-mail: dorota.nagolska@put.poznan.pl
[3] West Pomeranian University of Technology, Institute of Materials Science and Engineering
70-310 Szczecin, al. Piastów 19, e-mail: agnieszka.kochmanska@zut.edu.pl

Key words: casting, metal matrix composite (MMC), material recycling

Abstract

Quality of composite castings with infiltration reinforcement is dependent on many various factors. Technological parameters of the saturation process, manufacturing methods and quality of components are considered as the basic ones. During the recycling process of composite materials, the basic component that is being reclaimed is the metal matrix. The main aim of this paper is an evaluation of chemical composition and microstructure of the recovered metal matrix material.

In the first stage, influence of preparation of liquid matrix metal is analyzed, with particular attention paid to its purity, degree of degassing and overheating, as well as an influence of the reinforcing material type on the final structure of composites obtained in the process of vacuum saturation. Specific of the structure and flaws occurring in such materials are of the particular concern here, as they are decisive for the methods of quality evaluation of these materials. Manufactured composite's castings are subjected to the recycling process by a melting of the metal matrix on the second stage. The metal obtained in this way is analyzed regarding impurities occurring in its volume and, simultaneously, a comparison of evaluation methods of traditional metal alloys and metal composite castings is performed.

Introduction

There are many elements that shape the quality of manufactured materials in the process of manufacturing of the metal matrix composite castings, similarly as in case of the traditional castings (i.e. out of cast steel, cast iron or non-ferrous metals) [1]. However, a quality of castings made out of metal matrix composites is not determined solely by the behavior of crystallizing and cooling of the metal, but also by the reinforcement. Besides usual defects resulting from character and method of preparation of the metal matrix, the structural defects resulting from an interaction of the reinforcement with the solidifying casting may also appear [2]. The reinforcement stays in solid phase during composites' manufacturing by saturation, but its contact with the liquid matrix may result in a presence of precipitations of various types [3]. It can, for instance, cause a nucleation of the new phases, which traditionally would never appear in the crystallizing matrix, directly inside the metal or on reinforcement's elements (Fig. 1), being a propagator of reaction between matrix components or directly interacting with the matrix [4, 5]. The reinforcement can itself be a cause of defects such as: impurities (Fig. 2a), irregular arrangement of reinforcing elements (Fig. 2b), damage in the reinforcing phase (Fig. 2c) or insufficient saturation of the reinforcing structures (Fig. 2d) in the composite castings [2].

All the elements mentioned above obviously influence the quality of manufactured composite castings with infiltration reinforcement and can be a reason of disabling the castings already manufactured from further usage. As such, they can cause an increase in quantity of the composite scrap,

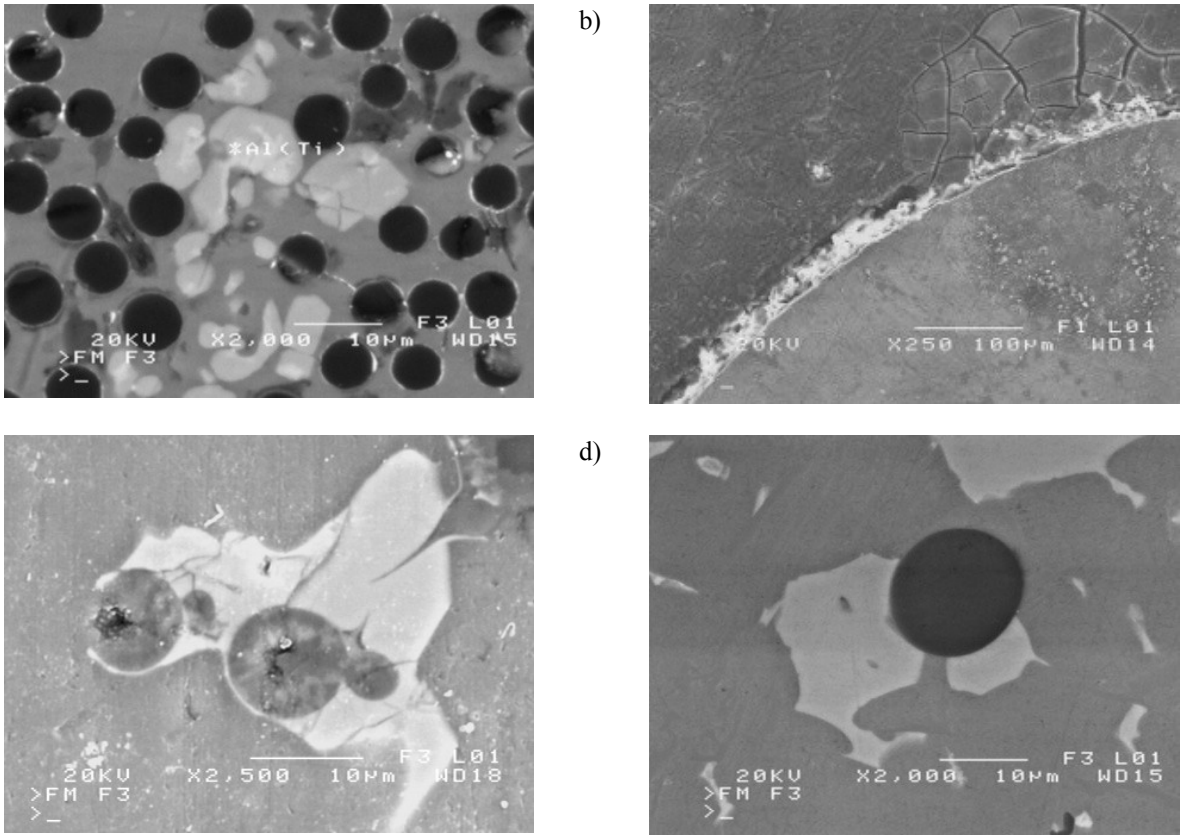

Fig. 1. Impurities of the metal matrix: a) inclusions formed in the matrix, b) brittle phases on the interfacial surface, formed as a result of reaction between reinforcement and matrix, c) and d) precipitations of the matrix phases on the reinforcement material [2]

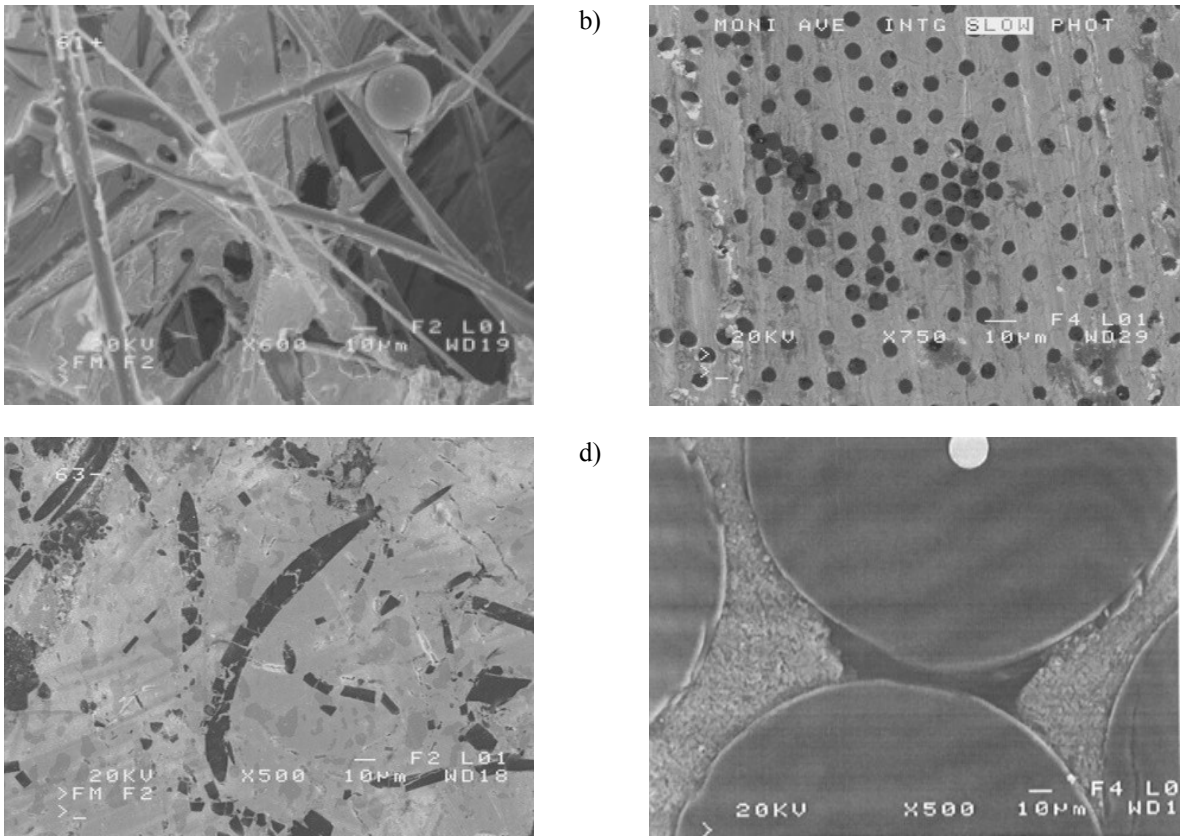

Fig. 2. The defects of the composite castings: a) foreign matter in the reinforcement, b) heterogeneity of arrangement of the reinforcement elements, c) fragmentation of the reinforcement elements, d) unfilled reinforcement space [2]

which, according to the compulsory law, should be subjected to a recycling in as wide range as possible. They can also cause a decrease in quality of this scrap and be a reason of the problems related to recycling of such materials.

The only beneficial way of recycling of the discussed materials is the process of separation of the composite components [6, 7]. Because of this, quality and quantity of matrix metal obtained during this process is a measure of its efficiency – if this metal meets specific requirements, it can be used again, e.g. for a repeated saturation of the composite reinforcements [7]. There upon it was decided to test if the defects appearing in the composite castings have relevant impact on a quality of the metal matrix, reclaimed during the recycling process. An examination of the chemical composition and metallographic, as well as the strength tests can be counted among the basic methods of determining the quality of materials, including foundry metal alloys. However, research of the composite castings requires more sophisticated testing methods (or, sometimes, a mix of different methods), widely described in literature [1, 2]. Application of these methods is often necessary to evaluate a quality of the composite castings, but is not required for a description of the metal matrix quality reclaimed during the recycling process. Hence, this work focuses only on an analysis of the chemical constitution and the metallographic quality of the examined materials, putting aside the analysis of mechanical properties. It changes not only with the chemical constitution of the material, but also with its physical form resulting for instance from a speed of the crystallizing and cooling process.

Research methodology

The following research plan has been conducted to determine an influence of the composite castings quality on a quality of metal reclaimed during the recycling process:

1) First, a quality of the metal further used for composite manufacturing was determined through:
   - analysis of the metal chemical constitution and its comparison with the standard requirements;
   - structure observation using the optical microscope and the scanning electron microscope;
   - identification of chemical components in the structure using X-ray microanalysis probe.
2) Then, the selected and examined alloy has been used to saturate the composite reinforcement and the metallographic evaluation of the obtained material has been performed to detect the defects (Figs 1 and 2).
3) Finally, the defective composites have been subjected to recycling and the obtained metal has been subjected to analysis, analogous to the analysis performed on the input alloy itself.

As a matrix for composite casting manufacturing, an AlSi12(b) alloy (former designation AK11) was used. An examination of the chemical constitution for the analyzed alloy has been carried out in laboratory of the Volkswagen company in Poznan, using emission spectroscope of ARL company, type MA 178 in atmosphere of the argon. A result of the examination has been treated as the average of minimum number of 3 measurements performed on each sample separately.

a) b)

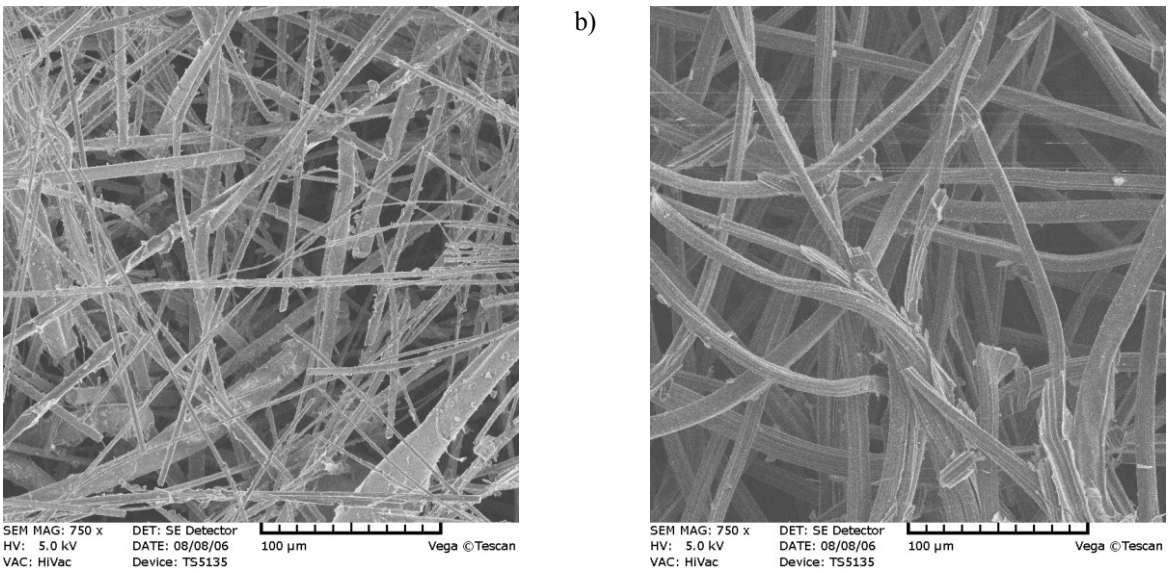

Fig. 3. The microstructure of the reinforcement preforms manufactured using the pressed short disordered fibers a) alumino-silicate (SIBRAL, produced by KERAUNION SA), b) graphite (SFA, produced by SGL Group the Carbon Company) [7]

The material specimen in form of short unordered alumino-silicate and graphite fibers was used (Fig. 3, Table 1) similarly to the reinforcement preforms. It was subjected to saturation with the matrix metal using a hydraulic press under various pressure (5–50 MPa). For research purposes, metal which was used to saturate the reinforcing preforms was heated to the temperature above 800°C.

A recycling of the manufactured composite materials has been conducted by putting the examined samples into a medium of the fused salts heated to the 740°C temperature for 30 minutes, according to the guidelines prepared before [7, 8].

Some samples cut out of the examined materials are subjected to the structural analysis on electron scanning microscope of TESCAN company, type VEGA TS 5135. The electron scanning microscope analysis has been performed using BSE (Backscattered Electrons) detector. An analysis of chemical components of the structure elements was conducted using X-ray microanalysis probe and microanalysis system AWALON 4000, which form an attachment to the microscope TESCAN VEGA TS 5135. This analysis allows to identify the metal microstructure and a character of present impurities. Both qualitative and quantitative evaluation of impurities in selected samples has been performed using the available computer software for image analysis.

Table 1. Properties of the reinforcement preforms manufactured using the pressed short fibers (manufacturer data)

	Short unordered alumino-silicate fiber	Short disordered graphite fiber
Chemical constitution	Al_2O_3 45–48%, SiO_2 51–54%	C (Graphite) 100%
Specific density	2770 kg/m^3	2100–2300 kg/m^3
Apparent density	370 kg/m^3	1500 kg/m^3
Porosity	86.66%	95%

Research results

Results of the chemical constitution analysis of the input material – a matrix for the composites with infiltration reinforcement and the material obtained as a result of recycling of these composites are shown in the table 2.

Table 2. Chemical constitution of the aluminum alloy forming a matrix for the composites with an infiltration reinforcement and metal obtained as a result of recycling of these composites

Component \ Sample	PN-EN 1706 Standard for alloy EN AC-AlSi12(b) (EN AC-44100)	Input matrix alloy AlSi12(b)	Metal melted during the composite recycling process		
			Reinforcement: short unordered fibers		
			Alumino-silicate	graphite	graphite
			Medium		
			NaCl+KCl	NaCl+KCl	POKAL
Al	rest	rest	Rest	rest	rest
Si	10.5–13.5	11.47	11.82	10.86	11.18
B		0.0010	0.0005	0.0004	0.0005
Be		0	0	0	0
Bi		0.0005	0.0005	0	0
Ca		0.0021	0.0001	0.0010	0.0034
Cd		0	0	0.0008	0.0008
Co		0.0002	0.0002	0.0002	0.0002
Cr		0.005	0.005	0.040	0.040
Cu	0.15	0.37*	0.35*	0.04	0.04
Fe	0.65	0.594	0.595	0.700*	0.683*
Hg		0	0	0.0001	0.0005
Li		0.00001	0.00001	0	0.00002
Mg	0.10	0.058	0.002	0.002	0.003
Mn	0.55	0.045	0.045	0.036	0.035
Na		0.0001	0	0.0006	0.0069
Ni	0.10	0.008	0.009	0.055	0.055
P		0.00028	0.00010	0.00044	0.00015
Pb	0.10	0.006	0.006	0.032	0.006
Sb		0.0003	0.0006	0.0468	0
Sn		0.010	0.010	0.089*	0.094*
Sr		0.0008	0.0008	0.0003	0.0004
Ti	0.20	0.0183	0.0172	0.0219	0.0230
Zn	0.15	0.142	0.136	0.041	0.065
Σ of the other elements (0.05 each)	0.15	0.04149	0.03281	0.17964*	0.14687

* the lack of conformity with the standard.

The results show insignificant differences only between a chemical composition of the metal melted during the recycling process and the metal originally used for composite manufacturing. Comparing results of analysis of chemical composition of the input AlSi12 (b) alloy and the alloy obtained during the recycling process, the following effects are the most apparent: a variation in copper (Cu) content, increase in ferrum (Fe) and tin (Sn) content above the level allowed by both standard's fluctuations in the metal originating from recycling of the composites reinforced with the graphite fiber and decrease in intensively oxidizing magnesium (Mg) content in all samples obtained during the recycling. In metal samples originating from the composite reinforced with the graphite fiber and subjected to recycling in NaCl+KCl medium, a content of antimony (Sb) also increased significantly, but not above the value acceptable by the standard, but it caused an exceed in total impurities content above the level acceptable for this metal. A cause of this phenomenon may be the fact, that the furnace used for the recycling process was previously used for melting other alloys containing significant amounts of intensively vaporizing antimony. Anyway, all these fluctuations should be treated as insignificant and be easy to eliminate in the process of preparation of the metal for the second use.

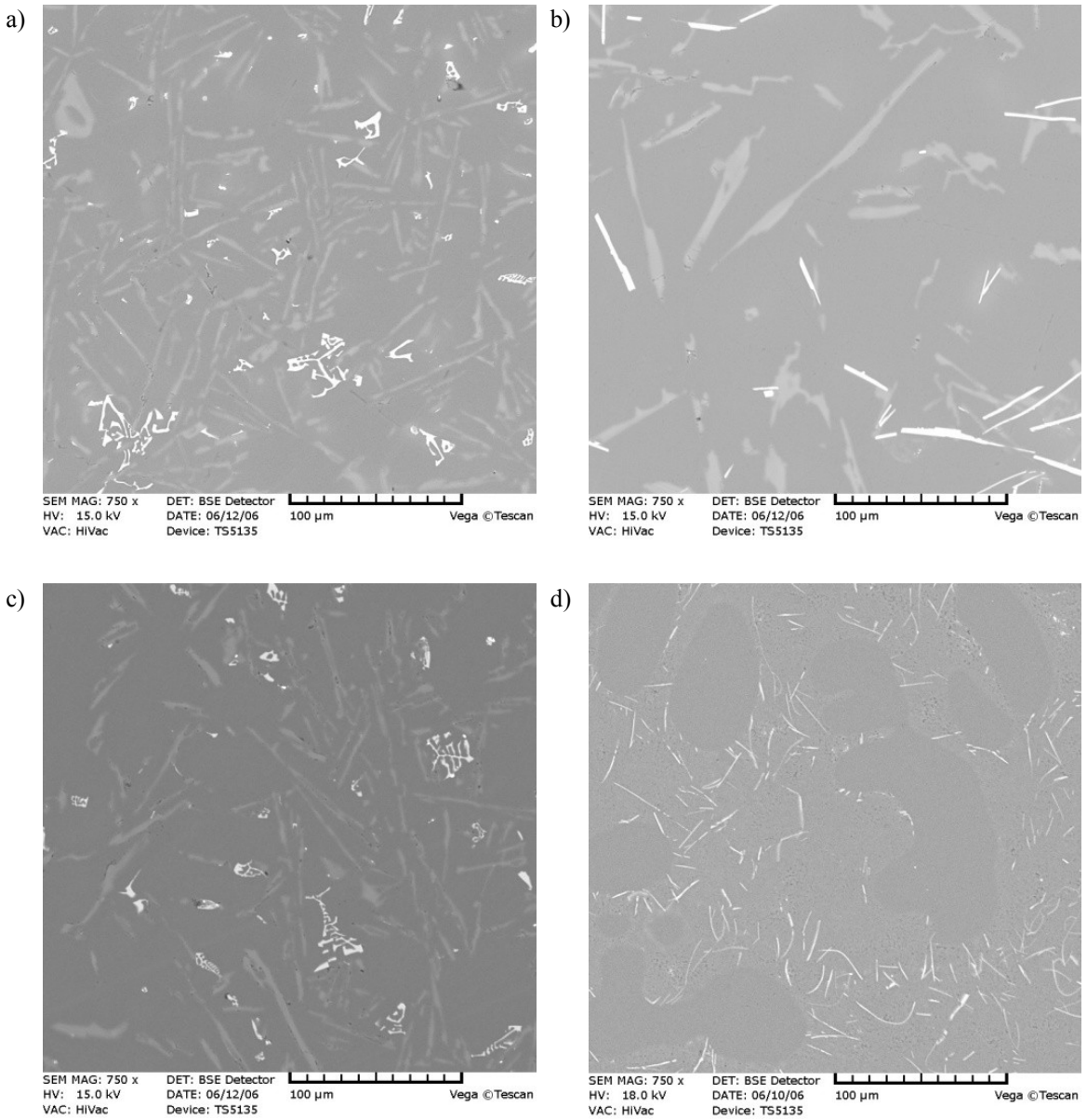

Fig. 4. Metallographic structure of AlSi12(b) aluminum alloy: a) input; b) melted out of the composite reinforced with short disordered alumino-silicate fiber during the recycling process in a medium of the fused mixture of NaCl+KCl; c) melted out of the composite reinforced with short disordered graphite fiber during the recycling process in a medium of fused mixture of NaCl+KCl and d) in a medium of fused POKAL

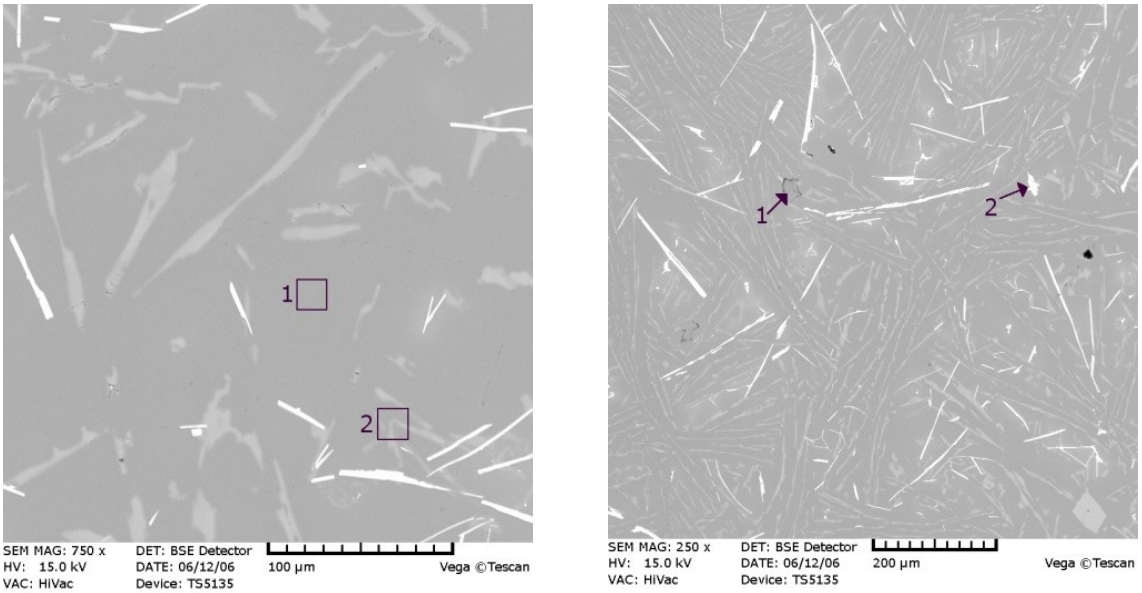

Fig. 5. The microstructure of an alloy obtained as a result of separation of components of the composite AlSi12(b) – pressed short disordered alumino-silicate fiber SIBRAL in a medium of fused mixture NaCl+KCl

Figure 4 shown below presents the structure of the input metal and the structure of the metal melted during the recycling process of the composites with infiltration reinforcement manufactured out of this metal.

The microstructure of the metal obtained as a result of separation of components of the composite AlSi12(b) – pressed short unordered alumino-silicate fiber SIBRAL in medium of a fused mixture NaCl+KCl is shown in the figure 5. Results of X-ray microanalysis of structural components indicated in this figure are presented in the table 3.

The microstructure of the metal obtained as a result of separation of components of the composite AlSi12(b) – pressed short disordered graphite fiber in medium of a fused mixture NaCl+KCl is shown in the figure 6. Results of X-ray microanalysis of structural components indicated in this figure are presented in the table 4.

Table 3. The results of microanalysis structural components indicated in the figure 5

Structure element	Content [% of mass]						Identification
	Al	Si	O	Cu	Fe	Mn	
□ 1	98.84	0.98	0.18				Al-Si solution
□ 2	69.59	30.23	0.18				Al-Si eutectic
← 1	98.19	0.80	0.41	0.61			impurity
← 2	58.55	16.12			24.17	1.16	impurity

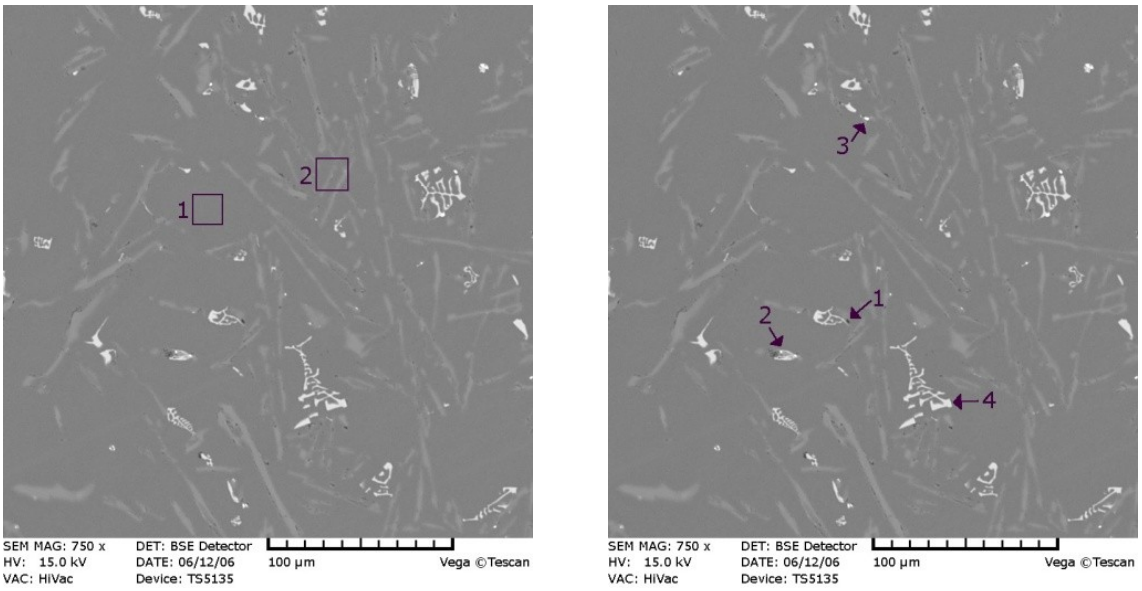

Fig. 6. The microstructure of an alloy obtained as a result of separation of components of the composite AlSi12(b) – pressed short disordered graphite fiber in a medium of fused salt mixture NaCl+KCl

Table 4. The results of microanalysis structural components indicated in the figure 6

Structure element	Content [% of mass]											Identification
	Al	Si	O	C	Cu	Fe	Pb	Ni	Sn	Mn	Cr	
□ 1	98.75	1.16	0.09									Al-Si solution
□ 2	84.99	14.89	0.12									Al-Si eutectic
← 1	38.15	25.02	1.25	35.59								impurity
← 2	69.18	4.46		13.07	0.42	11.49	1.11	0.27				impurity
← 3	14.10	56.98	0.57	10.57			0.78		17.00			impurity
← 4	57.52	7.92		8.98		24.06				1.38	0.13	impurity

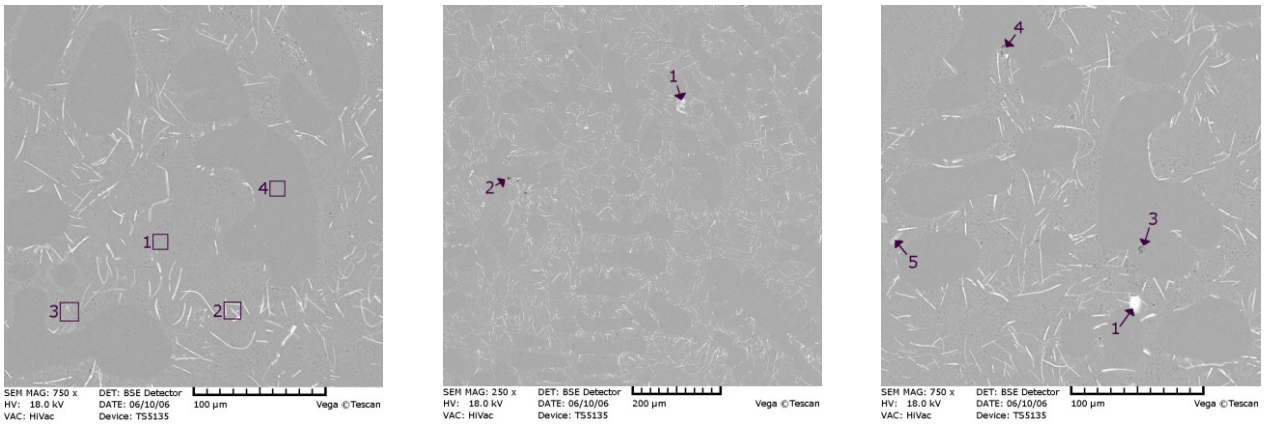

Fig. 7. A microstructure of an alloy as a result of separation of components of the composite AlSi12(b) – pressed short disordered graphite fiber in a medium of fused POKAL

Table 5. The results of structural components microanalysis indicated in the figure 7

Structure element	Content [% of mass]								Identification
	Al	Si	O	Fe	C	Mn	S	Ca	
□ 1	85.63	14.07	0.31						Al-Si eutectic
□ 2	86.01	12.46	0.17	1.37					Al-Si eutectic
□ 3	85.30	14.08	0.45	0.16					Al-Si eutectic
□ 4	98.87	0.96	0.17						Al-Si solution
← 1	26.59	3.61	0.04	3.64	65.96	0.16			impurity
← 2	72.41	1.33	15.35	1.30	8.23		1.08	0.30	impurity
← 3	78.63	1.09	3.90		10.38		2.88	3.14	impurity
← 4	79.81	1.09	0.33		18.77				impurity
← 5	64.80	7.76	0.14	3.74	23.55				impurity

A microstructure of the metal obtained as a result of separation of components of the composite AlSi12(b) – pressed short unordered graphite fiber in medium of a fused POKAL is shown in the figure 7 and the results of X-ray microanalysis of the structural components indicated in this figure are presented in the table 5.

The conducted metallographic studies have confirmed low content of impurities in the metal. On the examined polished sections, no particles that could be unequivocally identified as fragments of the composite reinforcement have been found.

In the metal originating from the recycling of the composites with AlSi12(b) matrix, in all observed samples subjected to x-ray microanalysis, some content of ferrum (Fe), aluminum (Al), silicon (Si) and manganese (Mn), forming the so-called Chinese script [9] has been found (Figs 5, 6, 7 and tables 3, 4, 5), which is compatible with the results of the chemical analysis that confirmed relatively high content of this element. However, an increase of the iron content does not exceed 0.7%, so it should not influence a strength of the metal obtained [9]. The other metallic impurities are observed also on the polished sections: chrome (Cr), copper (Cu), manganese (Mn), nickel (Ni), lead (Pb), in case of the composite subjected to recycling in the POKAL medium – also calcium (Ca)

and the non-metallic sulfur (S). All the examined precipitations occurred in the presence of carbon (C) in the metal originating from composites reinforced with the graphite fiber, while a presence of the carbonic reinforcement may favor the formation of precipitations [2, 4]. Systematic observation of surfaces of examined samples has indicated only single, fine precipitations. Hence, an influence of these precipitations on a quality of the metal obtained should be considered trifling.

Conclusions

Both, chemical and metallographic, analysis did not reveal significant increase in impurities in metal obtained during recycling attempts, in a comparison with the metal used for composite manufacturing. These deviations from the guidelines contained in the standard result mostly from a quality of the metal used for composite casting manufacturing and not from the recycling process itself. The reinforcement particles have not been found in the metal and occurring impurities can be considered as typical for used alloys, which shows the high quality of the recovered metal matrix. Therefore, the defects of structure of the composite castings have no influence on quality of the metal obtained in the recycling process because of the method and a character of the recycling process.

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