

Wear Resistance of Aluminum Matrix Composites Reinforced with Al_2O_3 Particles After Multiple Remelting

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(Submitted November 10, 2015; in revised form December 7, 2015; published online January 4, 2016)

Based on previous results, the commercial composites of A359 (AlSi9Mg) alloy reinforced with 22 vol.% Al_2O_3 particles were submitted to multiple remelting by means of gravity casting and squeeze-casting procedures. The studies were focused on tribological tests, x-ray phase analyses, and microstructural examinations. More promising results were obtained for squeeze-casting method mainly because of the reduction of the negative microstructural effects such as shrinkage porosity or other microstructural defects and discontinuities. The results showed that direct remelting may be treated as economically well-founded and alternative way compared to other recycling processes. It was underlined that the multiple remelting method must be analyzed for any material separately.

Keywords metal matrix composites, microstructure, remelting, wear resistance

1. Introduction

Nowadays for many materials dedicated to various practical applications, the production process is the one task and utilization method of these materials is the second one that has to be resolved. For many years, the efforts are oriented on the production of composites including aluminum matrix composites. This fact results from their attractive useful properties obtained for example due to the lower weight of composites compared to the steel or cast irons. In consequence, there is a research necessity of economically well-founded methods of their recycling. In general, the necessity of the usage of several or at least two technological stages results from many theoretical-practical conditionings (Ref 1, 2). This fact complicates the course of the recycling process as well as causes an increase of its cost. Previous results concerning the effects of multiple repeated remelting of A359 matrix composites reinforced with SiC particles (Ref 3, 4) showed that this way may be an alternative for other recycling methods. In that case, the following factors were analyzed in detail: the temperature of the casting process, the chemical composition

This article is an invited submission to JMEP selected from presentations at the Symposium “Metal-Matrix Composites,” belonging to the topic “Composite and Hybrid Materials” at the European Congress and Exhibition on Advanced Materials and Processes (EUROMAT 2015), held September 20-24, 2015, in Warsaw, Poland, and has been expanded from the original presentation.

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of the metal matrix, the content and the morphological properties of the reinforcement phase, and the reaction on the interfaces between the reinforcement and the metal matrix of the composite. The above-mentioned factors can have a significant influence also on the results obtained in the successive remelting performed for recycling of the products eliminated from operation.

As it has been mentioned, one of the basic threats which may have to be faced already at the first casting of such composites is the possibility of the occurrence of disadvantageous chemical reactions. They take place on the reinforcement-matrix boundaries, causing the composites' microstructural degradation affecting the decrease of mechanical characteristics.

The work (Ref 5) from 1993 can be recognized as one of the complex reports containing a detailed analysis of the conditions required for the chemical reaction resulting in the decomposition of SiC. Consequent to this reaction, Al_4C_3 precipitates are created as well as pure Si crystals appeared. They both significantly lower the mechanical properties of the composite. The author of the work (Ref 5) states that this reaction is detrimental especially for the three reasons:

1. it is the cause of the creation of the disadvantageous product of the reaction in the form of Al_4C_3 precipitates, which can be observed on the reinforcement-metal matrix boundary, weakening the strength of both the reinforcement phase and the interfaces and, consequently, of the composite as a whole;
2. Al_4C_3 , in some environments as an unstable compound, causes the corrodibility increase of the composite;
3. the creation of Al_4C_3 causes changes in the silicon content in the alloy, and in the case when this reaction is intense, the composition of the matrix's alloy can undergo a significant and negative change.

Required Si contents in the matrix of composites reinforced by SiC at different temperatures, preventing the creation of Al_4C_3 (Ref 5, 6), are shown in Fig. 1a.

As a result of the performed investigations, it was turned out that if we remelt the composite reinforced by SiC, but with the matrix of aluminum alloy with 8 wt.% Si content, the

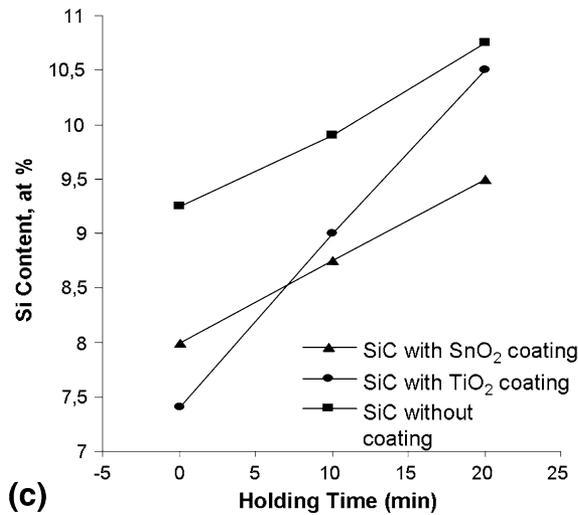
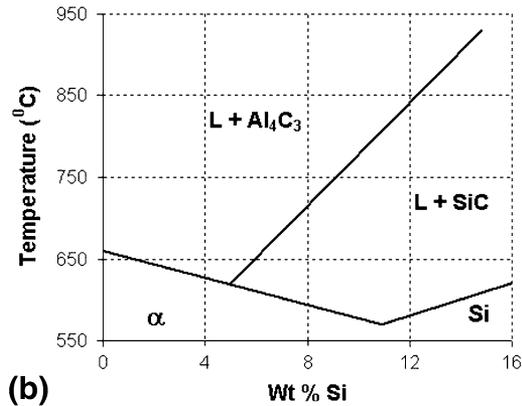
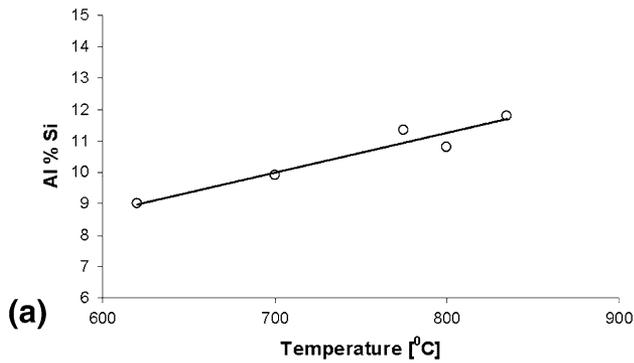


Fig. 1 Conditions of Al_4C_3 creation: (a) required Si contents in the matrix of composites reinforced by SiC at different temperatures, preventing the creation of Al_4C_3 (Ref 5, 6); (b) stability area of SiC, depending on the Si content and casting temperature (Ref 7); and (c) barrier oxide coatings on SiC inhibiting Al_4C_3 creation (Ref 8)

reaction of the Al_4C_3 creation does not take place (Ref 5, 6). Moreover, in (Ref 7) the stability area of SiC depending on the Si content and casting temperature is shown (Fig. 1b). The paper (Ref 8) shows the examples of introduced barrier oxide coatings on SiC inhibiting Al_4C_3 creation (Fig. 1c) and consequently improving the useful properties of produced composite materials.

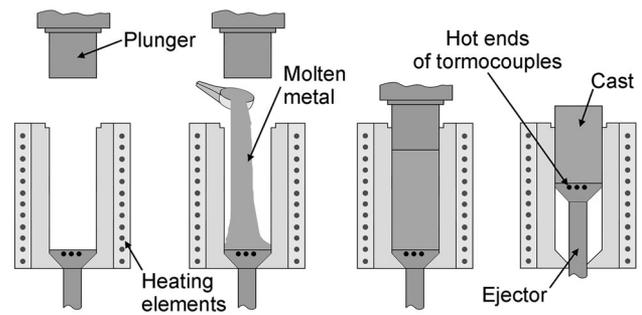


Fig. 2 Scheme of squeeze casting process

These reasons, among others, made the authors think that only the foundry alloys are appropriate for direct remelting, and pure aluminum as well as the aluminum wrought alloys assigned for plastic treatment (containing significantly lower silicon) are not suitable for recycling by way of direct remelting. In order to assure a better stability of SiC, different techniques of producing protective barriers are used directly on the reinforcement particles, and their task is to prevent the occurrence of the undesirable reaction. It is implemented, for example, by producing coatings on SiC in the form of oxides such as TiO_2 or SnO_2 , which inhibit the creation of Al_4C_3 (Fig. 1c). However, it should be emphasized that those coatings are effective with short remelting times at 850 °C. In the case when the time of the process is longer (Fig. 1c), the positive effect of the coatings is not observed (Ref 8). Taking into account these conditions in the previous studies (Ref 3, 4), multiple remelting treated as simulating the recycling process and related to A359 alloy reinforced with SiC particles was carried out. The simple gravity casting was employed and based on that it was possible to prove that up to forth remelting the fundamental mechanical properties were the same as for the composite in the as-received state. Next, it was ascertained that up to the tenth remelting the mentioned properties were progressively worsening and at last they were changed for the worse of about 27%. Instead, the wear intensity for the first and the tenth recasts was approximately the same. The observed differences in wear intensity values were affected by shrinkage porosity which is characteristic of the gravitational mold cast (Ref 3, 4).

In connection with the presented results previously, it was assumed that in the case of non-reactive system it is also possible to use the described recycling procedure. Thus, it was decided to carry out the similar multiple remelting method also for the commercial A359 matrix composites but reinforced with 22 vol.% Al_2O_3 particles. The research was focused on the methodology of remelting, microstructural effects, and wear behavior of this non-reactive system.

2. Experimental Procedure

Examined composites were subjected to 10-time remelting of the original material proposed as the simulation of the recycling process. Both the gravity cast and squeeze-cast methods were performed in the experiments. The scheme of squeeze casting process is presented in Fig. 2. Based on these procedures, the ready test specimens (pins) have been prepared for next investigations.

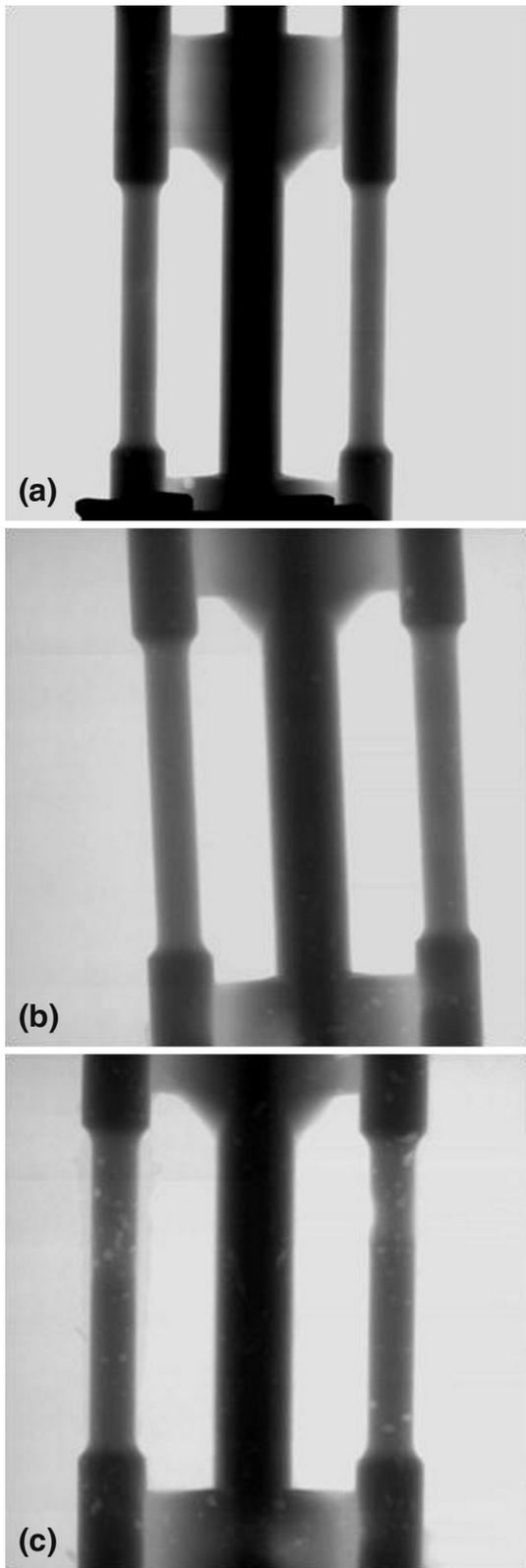


Fig. 3 Exemplary radiograms of gravity cast samples: (a) first remelting, (b) fifth remelting, and (c) tenth remelting. Higher porosity level of casting after tenth remelting (c) can be clearly seen



Fig. 4 Tribological test stand: (a, b) tester T-11 and (c) working principle; 1-sample (pin), 2-counter-sample (disk), P-pressing force, G-heating elements, and n-disk rotation

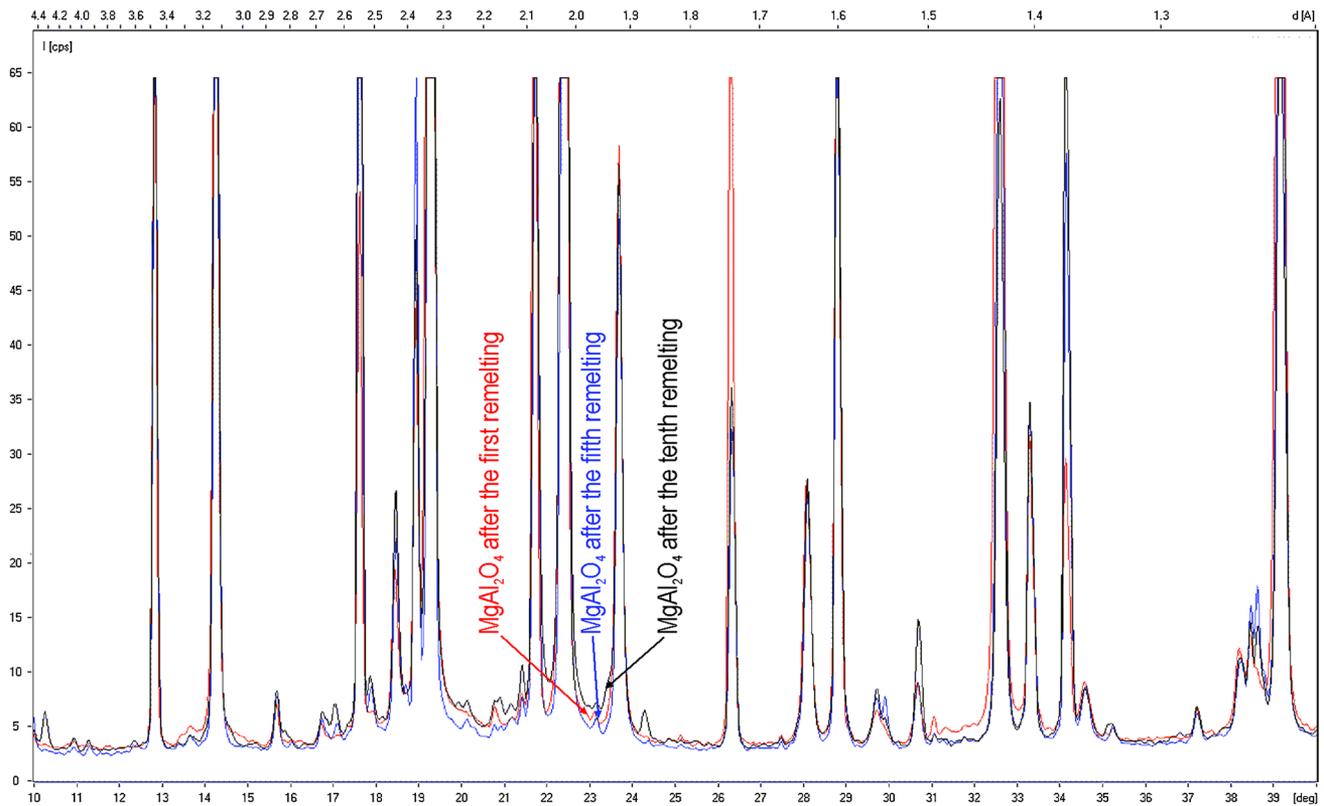


Fig. 5 Summarized results of x-ray phase analyses identified MgAl₂O₃ in alloy matrix; intensities of peaks are visible after the first (red line), fifth (blue line), and tenth remelting (black line) (Color figure online)

For squeeze castings, almost all structural discontinuities were eliminated by external pressure applied during solidification.

Each gravity casting was initially examined using x-ray methodology (Fig. 3).

Next, the composites received with both gravity casting and squeeze-casting methods were subjected to the tribological tests and microstructural examinations.

Phase analysis was carried out by means of the x-ray diffraction method. The phase composition analysis was performed using an x-ray diffractometer D8 Advance, with an energy-dispersive detector Sol-X. Studies were carried out to confirm or prevent the creation of the spinel MgAl₂O₄. Creation of the MgAl₂O₄ in the analyzed system may occur for example due to the following reaction $Mg(Al) + Al_2O_3 \rightarrow MgAl_2O_4$.

For the examination of the tribological properties, a tribological tester with the connection pin-disk (the *pin-on-disk* method) was used. A scheme of the research stand and the principle of operation is shown in Fig. 4. An immobile sample 1 in the form of a pin is pressed down with force P to disk 2 (counter-sample), rotating with speed n. The friction pair is placed in an isolated chamber equipped with a heating element G. In the tests, a sample made of steel 4140, 50 HRC in hardness, was used.

The tribological tests were performed at the conditions of technically dry friction, with the constant rubbing speed equaling 0.5 m/s, the constant friction path of 2500 m, and the constant load of the friction pair equaling 10 N. All the tests were conducted at the same environment conditions: temperature of 21 °C and relative humidity of 55% (measured with a hythergraph type LB-700, Production No. 334). Next, the

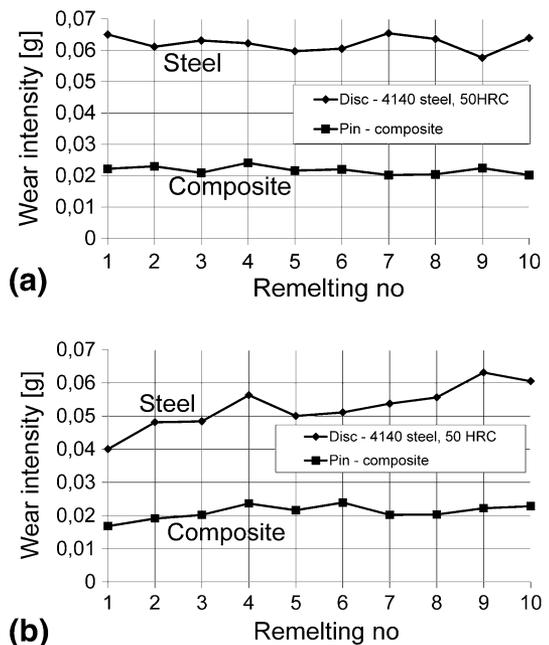


Fig. 6 Wear resistance of disk (counter-probe) made of 4140 steel, 50 HRC in hardness and A359/22 vol.% Al₂O₃ (pin), vs. remelting number for (a) gravity casting and (b) squeeze casting

composites after the first, fifth, and tenth remelting were submitted to the microstructural examination in the light microscopy range.

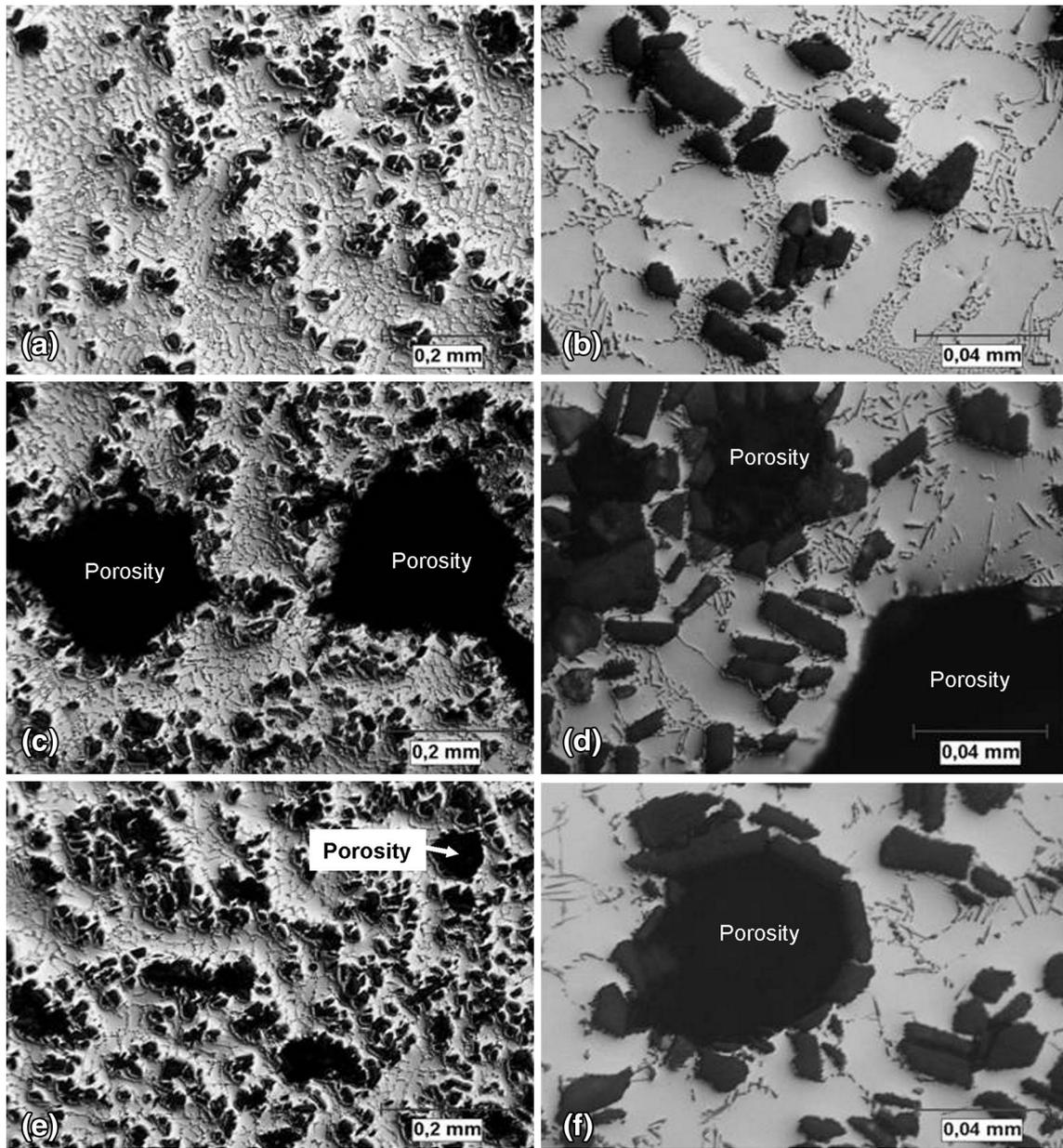


Fig. 7 Microstructural effects of gravity casts vs. remelting number: (a, b) the first, (c, d) fifth, and (e, f) tenth remelting

3. Results and Discussion

3.1 X-ray Analysis

During x-ray analysis, it was taken into account that the exposition surface of the composite sample covered only about 74% of the exposition surface of the reference material. Therefore, it was necessary to introduce the suitable correction of measured diffraction intensities. It should be also considered that the absorptivity of the spinel MgAl_2O_4 ($31.36 \text{ cm}^2/\text{g}$) slightly differed from the coefficient of the absorption of the corundum ($32.32 \text{ cm}^2/\text{g}$). Thus, from this reason and with good approximation, the occurrence absorption in the corundum and in the spinel can be taken into account together. Finally, it should be also underlined that all diffractograms were assem-

bled in identical time intervals. The summarized effect of performed analyses is presented in Fig. 5.

In general, MgAl_2O_4 concentrations (in weight) in examined composites are as follows: after the first recast—3.7%, after the fifth recast—5.2%, and after the tenth recast—5.4%, therefore the concentration values are practically on the same level. Bases on those, it is possible to ascertain that multiple remelting does not affect disadvantageous reactions in the alloy matrix especially at the interface of alloy matrix/reinforcing particles.

3.2 Assessment of Wear Resistance

The wear resistance determined by means of the tribological tests and described by weight loss versus the remelting number is presented in Fig. 6. The wear resistance was described by

Table 1 Porosity of the composite reinforced with 22 vol.% Al₂O₃ particles after multiple remelting

Remelting no.	\bar{V}_v , %	\bar{N}_A , 1/mm ²	\bar{F} , μm
1	2.74	62	28.4
2	3.93	96	30.5
3	4.68	105	32.1
4	4.01	105	30.2
5	4.78	103	32.2
6	4.36	103	31.2
7	4.50	119	27.9
8	4.52	115	27.8
9	4.06	98	27.6
10	7.59	151	31.6

weight—by means of weighing the samples before and after the test. The results of the samples after gravity casting and squeeze casting are presented in Fig. 6(a) and (b), respectively. The values of determined friction coefficients were constant and 0.5.

Determined wear resistance confirms the efficiency of the proposed recycling method by means of direct multiple remelts of the investigated composite. It is ascertained that after multiple gravity remelting the slight differences between the values of wear intensity versus remelting number were observed (Fig. 6a). Simultaneously, in the case of squeeze-casting the wear resistance after all remelting times is practically the same as in the as-received state (Fig. 6b).

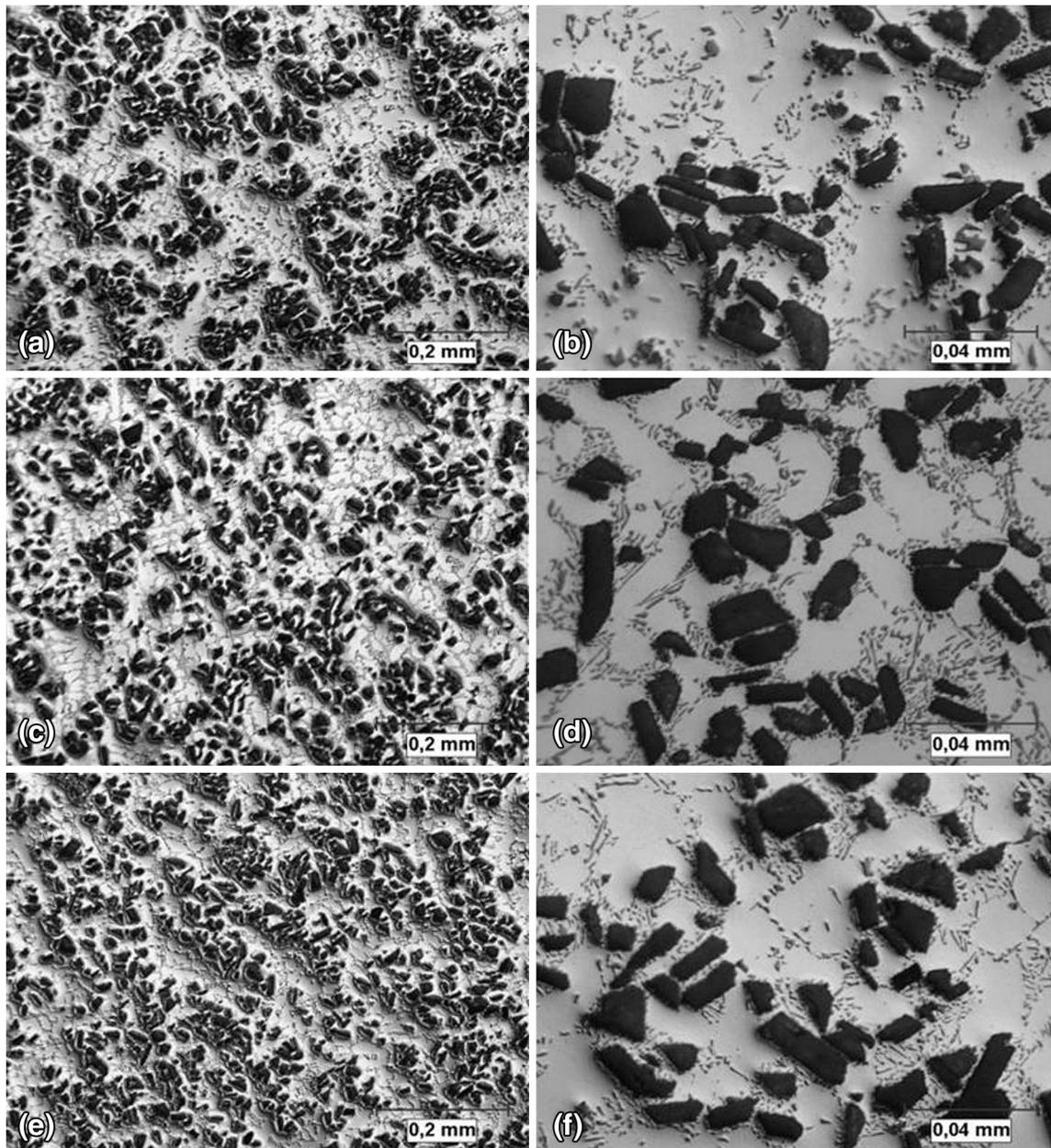


Fig. 8 Microstructural effects of squeeze casts vs. remelting number: (a, b) the first, (c, d) fifth, and (e, f) tenth remelting

3.3 Microstructural Examination

Microstructures of composites after multiple remelting performed by means of gravity casting and squeeze casting are presented in Fig. 7 and 8, respectively. The microstructural observations were carried out using the light microscope and at various microscope magnifications. It was noticed that in the case of gravity casting the shrinkage porosity is clearly visible. It was also observed that this porosity increased with the remelting number. The main geometrical parameters determined by means of image analysis are put together in Table 1.

The lowest porosity content [V_v (vol.%)] was noticed in the as-received state (Fig. 7a and b; Table 1), content of about two times greater was found after the fifth remelting (Fig. 7c and d; Table 1), and the greatest after the final remelting (Fig. 7e and f; Table 1). The number of porosity areas related to the unit area [N_A (1/mm²)] was changed in the similar (Table 1) way, whereas the sizes of porosity areas [F (μm)] were in the nearby level (Table 1).

Conversely, if the squeeze casting method was used, no microstructural changes were observed (Fig. 8); therefore, the multiple remelting did not cause any disadvantageous microstructural differences.

It should be underlined that using squeeze-cast procedure it was possible to obtain the excellent microstructure without any shrinkage porosity or other microstructural discontinuities identifiable in the light microscopy range.

4. Conclusions

Based on the results, it was ascertained that A359 matrix composite reinforced with 22 vol.% Al₂O₃ particles could be subjected to multiple remelting processes. Squeeze-casting methods leads to the reduction of shrinkage porosity and elimination of other microstructural defects or discontinuities compared to gravity casting. As a consequence, the wear resistance of the investigated composite was practically on the same level as in the as-received state. Simultaneously, it should be underlined that the proposed method could be treated as an economically well-founded alternative for other recycling methods such as composite component separation (Ref 9), disintegrated melt deposition technique (Ref 10), the method of pushing-out the metal matrix from the molten composite (Ref 2), and others.

Acknowledgments

This research was carried out in the frame of the Project No. N N507 242740 with financial support from the Ministry of Science and Higher Education of Poland.

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References

1. K.R. Ravi, R.M. Pillai, B.C. Pai, and M. Chakraborty, Separation of Matrix Alloy and Reinforcement from Aluminum Metal Matrix Composites Scrap by Salt Flux Addition, *Bull. Mater. Sci.*, 2007, **30**(4), p 393–398
2. M. Muzimoto, T. Oghai, and A. Agawa, Separation of PRMMC into Matrix Alloy and Reinforcement by Nozzle Filtering Method, *J. Mater. Process. Technol.*, 2009, **209**(9), p 4264–4267
3. A. Klasik, A. Wojciechowski, J. Sobczak, and N. Sobczak, Effect of Multiple Remelting on Selected Properties of Dispersed Reinforced Aluminum Matrix Composite, *J. Kones*, 2008, **15**(3), p 233–238
4. A. Klasik, J. Sobczak, K. Pietrzak, N. Sobczak, and A. Wojciechowski, *Influence of Multiple Remelting of Particulate Reinforced Cast Aluminum Composites on Their Properties and Structure*, MTI-FRI, Warsaw-Cracow, Poland, 2012
5. A. Labib, “L’effet des niveaux de refroidissement (température de du moule) et des traitements Thermiques sur les propriétés mécaniques et sur la Microstructure des deux alliages composites Al-Si-Mg/SiC/10p,” PhD thesis, Université du Québec à Chicoutimi, Québec, 1993
6. D.J. Lloyd, Solidification Microstructure of Particulate Reinforced Aluminum/SiC Composites, *Compos. Sci. Technol.*, 1989, **35**, p 159–179
7. F. Tongxiang, G. Yang, J. Chen, D. Zhang, Z. Shi, T. Shibayanagi, and M. Naka, Recycling Mechanism of Discontinuously Reinforced Al Matrix Composites, *Mater. Sci. Forum*, 2005, **502**, p 205–209
8. A359 Al Alloy Reinforced with Irregularly Shaped SiC Particles. <http://bricad.com/aluminium/dur/guides/awg/index.html>
9. K.R. Ravi, R.M. Pillai, B.C. Pai, and M. Chakraborty, Separation of Matrix Alloy and Reinforcement from Aluminum Metal Matrix Composites Scrap by Salt Flux Addition, *Bull. Mater. Sci.*, 2007, **30**(4), p 393–398
10. P.S. Ling and M. Gupta, Recycling of Aluminium Based Metal Matrix Composite Using Disintegrated Melt Deposition Technique, *Mater. Sci. Technol.*, 2000, **16**(5), p 568–574