



Solidification curves and structure of heterophase composite

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ABSTRACT

Purpose: This paper presents results of solidification tests for homo- and heterophase composites. Researches concerned influence of reinforcement particle for solidification process of composite materials. Curves of solidification composite reinforced with (Al_2O_3) and heterophase reinforcement (mixture of Al_2O_3 particles + glassy carbon) were compared with aluminium alloy matrix (AlSi12CuNiMg2). Based casting spiral test the castability of composite were put to the test. Also macro and microstructure ingots after solidification on equal thermal conditions were presented.

Design/methodology/approach: Solidification process was recorded with 0.4 sec. period by analog-digital converter connected to PC. Temperature was controlled by K thermocouple (NiCr-Ni) installed in standardized thermoelectric cup core QC4080. That equipment made possible to realised solidification tests on the equal thermal conditions. The castability was tested on standard spiral duct formed at self hardening phosphate mould.

Findings: Results of researches confirm influence of glassy carbon on solidification of composite suspension. Compared to aluminium oxide particles (Al_2O_3) glassy carbon accelerate solidification process of composite material. Probably, it results from disparate properties of glassy carbon.

Practical implications: Glassy carbon particles change characteristics of composite crystallization and decrease shrinkage of the casting. Moreover application of mixture of Al_2O_3 and glassy carbon as heterophase reinforcement allows to segregation and sedimentation particles in the matrix and it guides in results of solidification to gradient structure of composite material.

Originality/value: Employment of heterophase reinforcement allows to get segregation and sedimentation in the matrix, which results in the occurrence of a gradient structure.

Keywords: Composites; PAMMC; Solidification; Ceramic particles; Glass carbon particles

MATERIALS

1. Introduction

Aluminium-based metal matrix composites with different kind and size reinforcing phases are well-known for their high specific strength, stiffness, hardness also lower wear and corrosion resistant. These so good properties contribute to the many applications, especially in the aerospace, military, and especially in the automotive industries.

Particle aluminium metal matrix composites (PAMMCs) produced by casting method, for its good tribological properties, have found applications in particular in the automotive industries. Composites reinforced with ceramic particles (i.e. SiC, Al_2O_3) are most often fabricated by casting methods with the use of suspension forming processes. Economical reasons has contributed to a wide application of the liquid methods moreover of mechanical stirring of components [1-4].

Many authors link the shaping of the structure of Al alloy-ceramic particles composites with the use of casting techniques with two problems. The first one refers to particles' behavior in a liquid metal until crystallization commencement (floating, sedimentation or agglomeration), where wetting of ceramics by a liquid matrix is a significant and predominant factor. The other one is the description of factors connected with direct action of the growing crystallization front with reinforcing phase particles [3-15]. From the moment of crystallization and solidification commencement, the crystalline phase begins to grow. Its growth proceeds in a direction opposite to the particles' movement. Thus, apart from the geometric factor, i.e. the type, volume fraction and size of reinforcing particles, it is the crystallization rate and the casting's solidification time that determine the structure obtained and particles' distribution in the matrix.

The study aimed at determining and comparing the cooling curves obtained for the matrix (AlSi12CuNiMg2) and for composites containing one type of reinforcing particles (Al_2O_3) and heterophase composites, where two types of ceramic particles, a mixture of aluminium oxide and glassy carbon, were used as the reinforcement.

2. Research methodology

As the matrix material, a casting alloy of aluminium AlSi12CuNiMg, modified with a 2% magnesium addition, was used. In single-phase composites, aluminium oxide (Al_2O_3) of 15% fraction and a $25\mu m$ particle size was used for reinforcement. In heterophase composites, two types of ceramic particles were applied in the form of a mixture of $25\mu m$ aluminium oxide and glassy carbon of $160\mu m$ size. For that group of materials, a 10% fraction of each powder was applied. Composite suspensions were fabricated by the traditional stirring method, described in detail in paper [3], were then subjected to degassing and homogenization under lowered pressure.



Fig. 1. View of casting area used for degassing and homogenizing composites suspensions [17]

A testing stand designed and built at the Institute of Composites and Powder Metallurgy, Silesian University of Technology, was used to this end (Fig.1). As former research has shown [16,17], the

application of vacuum technology with simultaneous homogenization of a composite suspension changes the properties of the liquid suspension, including first of all its castability, and allows removal of gassy regions formed during composite production.

The course of the solidification process was recorded by means of a system which enabled continuous control and measurement of the metal temperature during solidification of the composite suspension (Fig. 2). The system was equipped with a thermoelectric cup core QC4080, with an incorporated thermocouple of K type (NiCr-Ni). The cup core was placed on a tripod and permanently connected via a contactor unit and a thermoelectric conductor with an analogue-digital measuring system based on the MC201 module, configured to operate with a PC [18].

The application of disposable thermoelectric cup cores of identical heat abstraction coefficient and known, standardized dimensions, ensured identical conditions and rate of heat abstraction during the cooling of the castings.

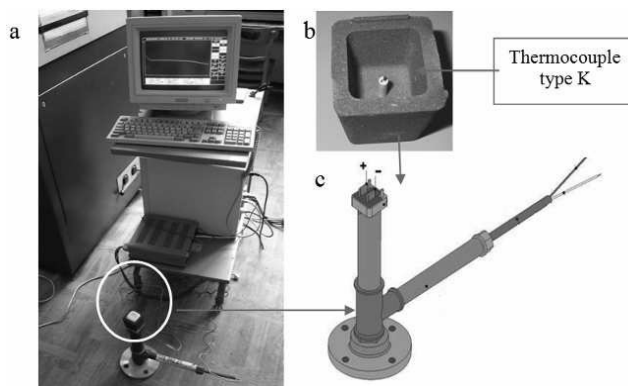


Fig. 2. Measurement system used for register of solidification curves a), thermo-electric cup with thermocouple type K b), column c) [18,22]

Thereby, the influence of the mould material on the solidification of the investigated composites was eliminated, which allowed an evaluation of the influence of the reinforcing particles' type on the course of cooling curves for the systems applied.

Also influence of ceramic particles on casting properties of composites was evaluated based castability test. Test duct was formed as spiral at self hardening phosphate mould.

The structure of composite ingots was examined on an MeF-2 Reichert light microscope and a Hitachi S-4200 electron microscope, applying properly made preparations.

3. Solidification curves and their analysis

The data obtained allowed plotting of cooling curves for the matrix and composites which, after numerical processing, are presented in Fig. 3. The differences in the course of composite materials' curves are particularly well visible for the heterophase composite, where apart from aluminium oxide particles, amorphous glassy carbon particles were used.

The differences refer to both time and temperature of the crystallization beginning. The matrix material solidified during 20s in the temperature range of 559-558°C. The composite containing a mixture of Al_2O_3 + glassy carbon particles solidified in the temperature range of 574-571°C, in the time of 7s. The temperature of crystallization beginning of the composite containing aluminium oxide particles was 557°C, with the composite solidifying for 18s in the temperature range of 557-556°C. As results from the data obtained, glassy carbon particles considerably increase the temperature at the beginning of crystallization and shorten its time, both when compared to the composite containing one type of particles (Al_2O_3) and when compared to the matrix. This testifies to a significant influence of glassy carbon particles on the solidification process course and thereby, to the disparate nature of the heterophase composite crystallization. The differences in composites' solidification time and temperature may be determined by different thermal conductivity coefficients applied for the particles, which amount for Al_2O_3 - 35W/(mK), for glassy carbon - 200W/(mK), and for the AlSi12CuNiMg matrix - 180W/(mK) [16].

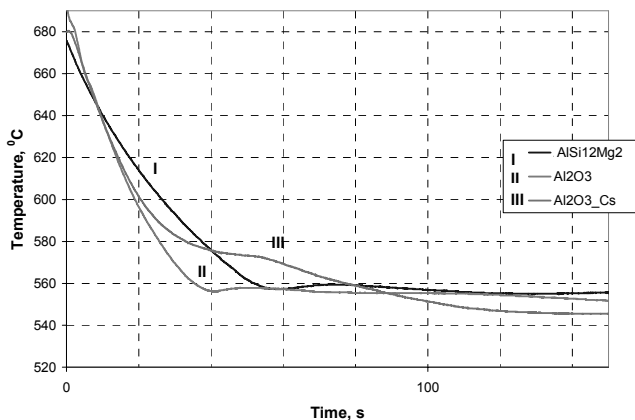


Fig. 3. Solidification curves of matrix and composites: I- AlSi12CuNiMg_2 , II- $\text{AlSi12CuNiMg}_2+10\% \text{Al}_2\text{O}_3$ 25µm, III- $\text{AlSi12CuNiMg}_2/10\% \text{Al}_2\text{O}_3$ 25µm + 10 % Cg [22]

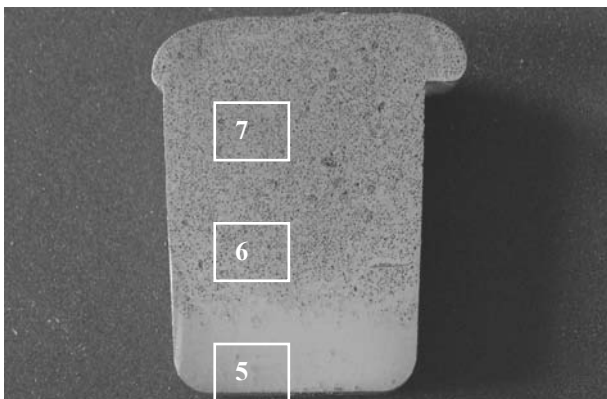


Fig. 4. Macrostructure of $\text{AlSi12CuNiMg}_2/10\% \text{Al}_2\text{O}_3$ 25µm + 10 % Cg heterophase composite with ceramic particles displacement visible in the matrix [22]

4. Microstructure of composite ingots

The crystallization and solidification processes of composite casts are very important in determining the microstructural features such as: phase composition, grain size and structure, first of all distribution of second phase particles. All of these factors influence the final material properties.

Macrostructure and microstructure of homo- and heterophase composite ingots obtaining by the solidification and crystallization processes are presented in Figures 4-15.

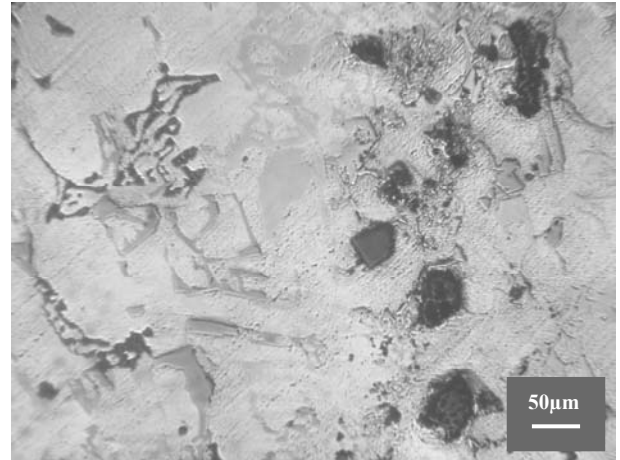


Fig. 5. Structure of matrix-reinforced interface area in the $\text{AlSi12CuNiMg}_2/10\% \text{Al}_2\text{O}_3$ 25µm + 10 % Cg heterophase composite, OM, [22]

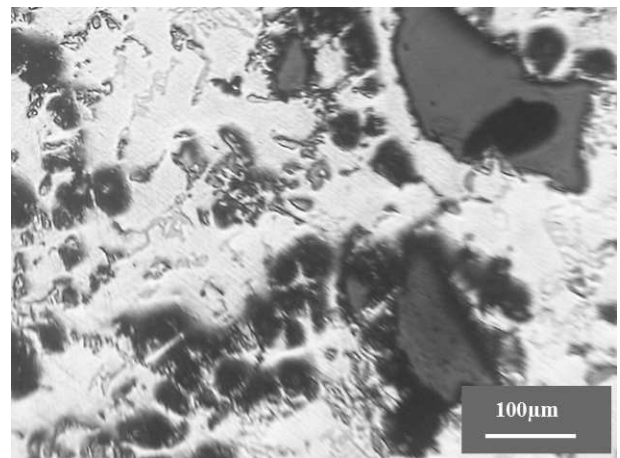


Fig. 6. Structure of ceramic particles displacement in the middle of ingot part of $\text{AlSi12CuNiMg}_2/10\% \text{Al}_2\text{O}_3$ 25µm + 10 % Cg heterophase composite, OM

On the cross-section of a heterophase composite ingot, floatation and segregation were found (Fig. 4), which in consequence, enabled the formation of a gradient structure. The lower part of the ingot does not contain particles and the matrix-composite interface is flat and parallel to its base (Fig.4).

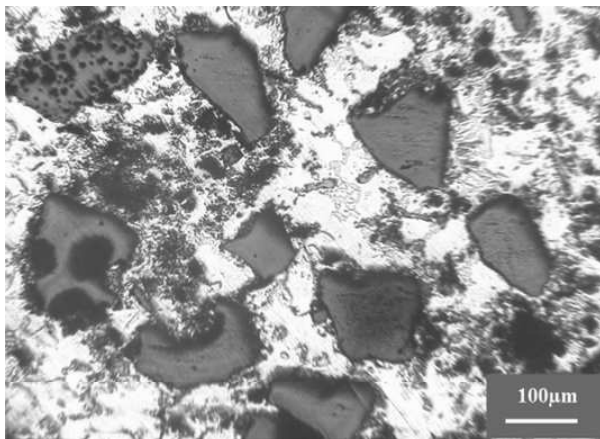


Fig. 7. Structure of ceramic particles displacement in the top of ingot part of AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg heterophase composite, OM [22]

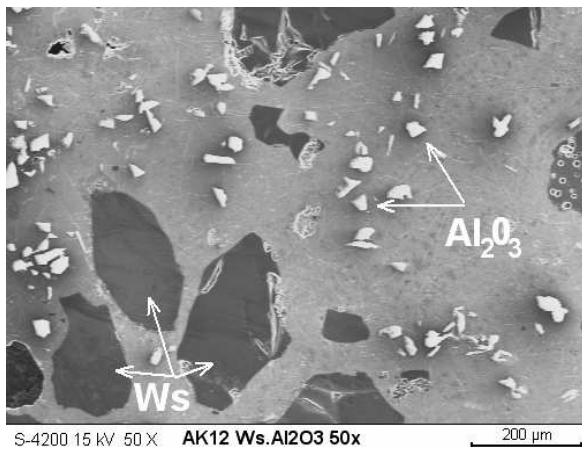


Fig. 8. Microstructure of AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg heterophase composite, SEM

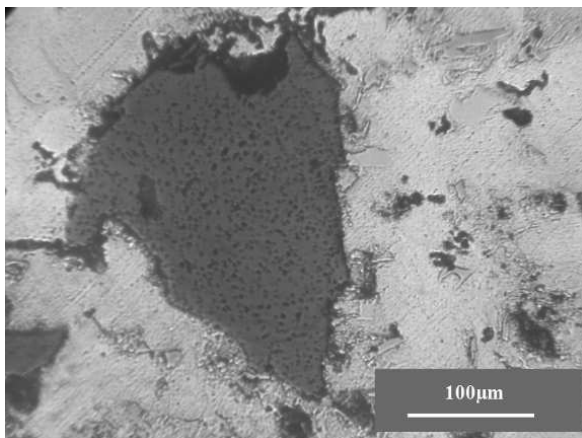


Fig. 9. Microstructure showing size and morphology of glassy carbon particle in the AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg heterophase composite, OM

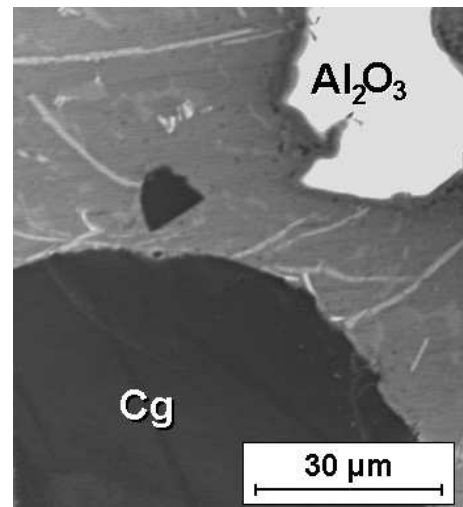


Fig. 10. SEM micrograph of the glassy carbon-matrix and aluminum oxide-matrix interface in the AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg, composite, mag. 500x, SEM

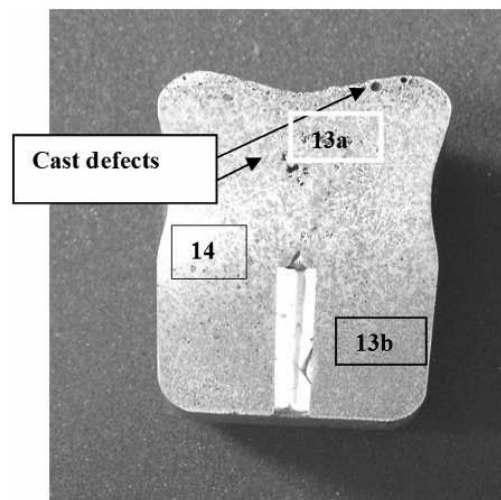


Fig. 11. Macrostructure of AlSi12CuNiMg2/10% Al₂O₃ 25µm composite with ceramic particles displacement visible in the matrix

The structure of the part of matrix - reinforcement interface is shown on Figure 5. The microstructure of ceramic particles displacement in the middle of ingot part of AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg heterophase composite is presented on Figure 6.

The microstructure from the upper part in the top of ingot, with visible glass carbon particles in the aluminium matrix, is shown in Figure 7.

Figure 8 shows the typical microstructure of an AlSi12CuNiMg2/10% Al₂O₃ 25µm + 10 % Cg composite cast obtained by the using scanning electron microscopy. The picture was made in the middle of the ingot part. In these area presence of aluminium oxide and glass carbon particles homogeneously distributed in the matrix, (Fig. 6,8). Figures 9 shows the size and morphology of glassy carbon particle and its good connection with the matrix.

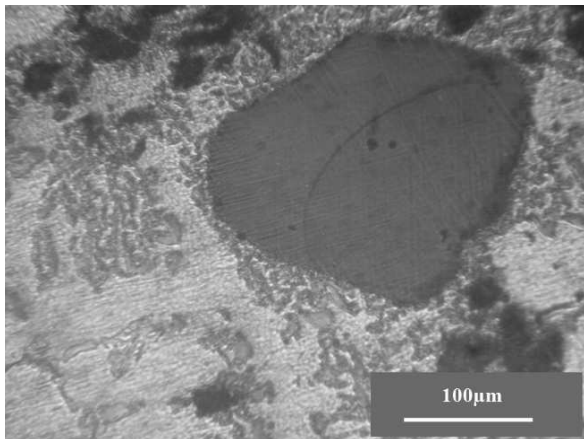


Fig. 12. Size and morphology of alumina oxide particle in the AlSi12CuNiMg2/10% Al₂O₃ 25μm composite, OM

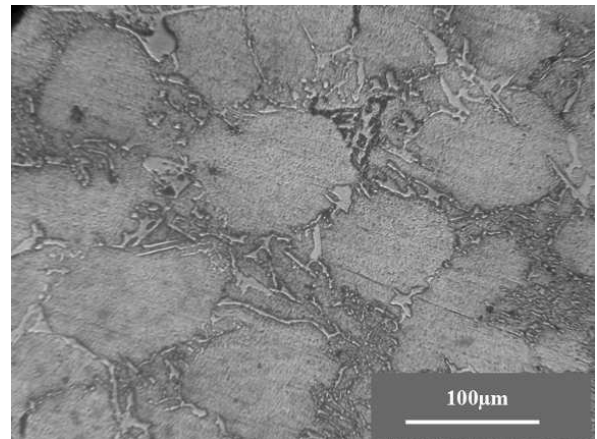
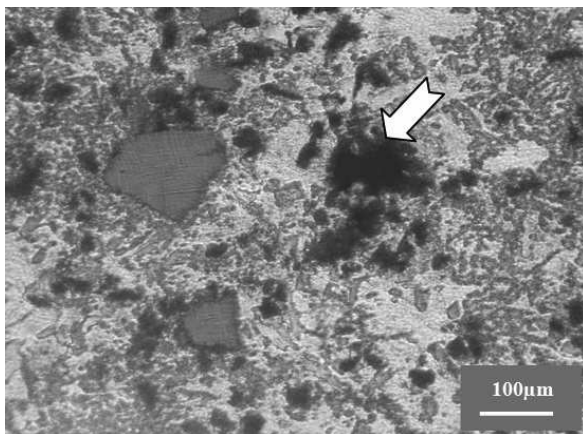
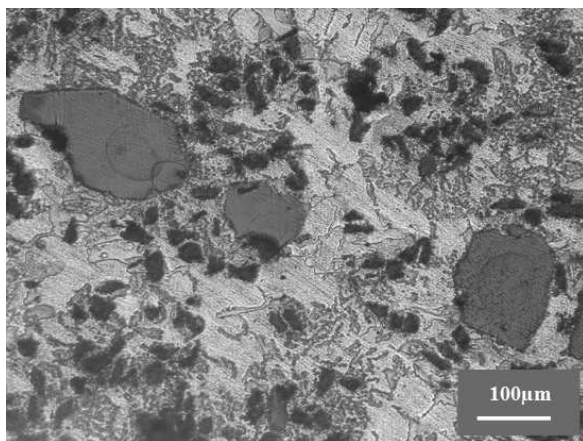


Fig. 14. Microstructure in the middle of ingot part; area without ceramic particles, the grain size of the matrix are visible on the picture, OM



a)



b)

Fig. 13. Ceramic particles displacement of AlSi12CuNiMg2/10% Al₂O₃ 25μm composite: a) in the top of ingot part, with cast defects; b) at bottom of ingot area, OM

On the basis of an optical and scanning microstructural observation you may say that the heterophase composite is

characterized by much lower shrinkage in the riser head region and lack of casting defects, which were identified in the composite with one reinforcing phase (Fig. 10). On the following pictures shown of ceramic particles displacement in different composite ingot part.

5. Results of spiral test

Results of spiral test was presented at Figure 15. It was found, that composites where one kind of reinforced particles were applied alumina oxide (Al₂O₃), after sand mould casting filled 15 spiral sections. Otherwise heterophase composites containing particles of aluminium oxide and also the particles of glassy carbon filled only 4 spiral sections. Probably that was results of influence of glassy carbon particles which accelerated solidification process of composites material and braking the stream of liquid phase in the channel of spiral mould.

6. Conclusions

As the research has shown, ceramic particles in the form of amorphous glassy carbon have an influence on both temperature change and the time of composite castings solidification. They also have influence on the casting properties of composites suspension, reducing they castability. The changes results, first of all, from disparate physical properties of the glassy carbon particles used (thermal conductivity, mass density) [19-22], compared to aluminium oxide particles (Al₂O₃). Moreover, glassy carbon particles decrease shrinkage of the casting and change the nature of its crystallization. Their low mass density (1,4 g/cm³) contributes to segregation and sedimentation in the matrix, which results in the occurrence of a gradient structure in the heterophase composite. The presented research results represent a preliminary study and they require completing. The on-going research refers to the influence of the type, size and volume fraction of reinforcing particles on solidification and crystallization of heterophase composites [23-24].

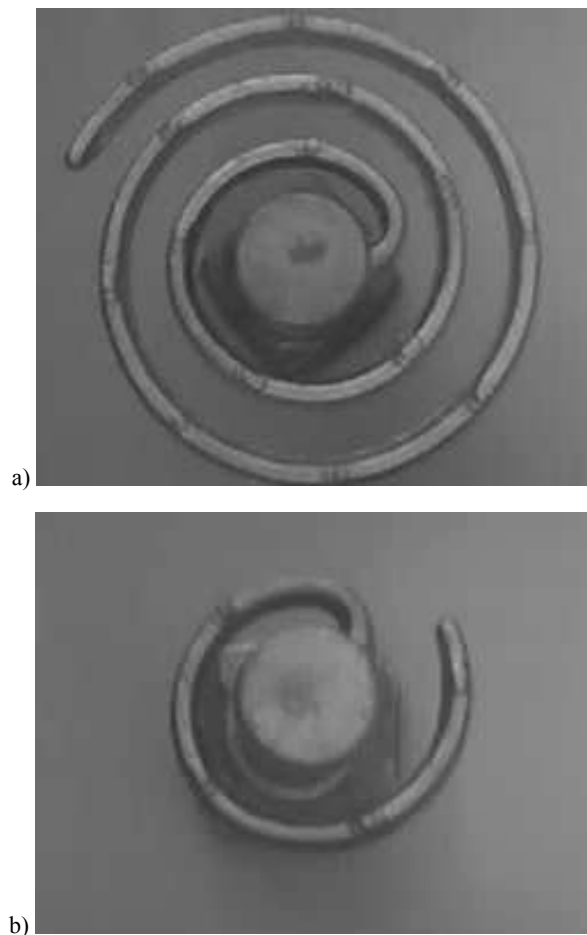


Fig. 15. Spiral test of composite suspension: a) for AlSi12CuNiMg2 / 10%Al₂O₃,25µm, b) for AlSi12CuNiMg2/ 10% Al₂O₃ 25µm + 10 % Cg heterophase composite

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