PRACTICAL ASPECTS CONCERNING THE SOLIDIFICATION OF CAST METALLIC COMPOSITES
(A review)

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Lucrarea cuprinde principalele aspecte privind solidificarea compozitelor metalice procesate prin turnare şi se bazează pe cunoştinţele generale referitoare la turnarea compozitelor, pe rezultate practice şi date experimentale proprii, în special în legătură cu compozitele realizate din aliaje de aluminiu şi diferite tipuri de particule: grafit, carbură de siliciu, sticlă şi alumină. Sunt, de asemenea, prezentate informaţii privind problemele tehnologice care apar, defecte specifice determinate de un proces de solidificare insuficient controlat, ca şi câteva metode de evaluare a calităţii acestor materiale .

The paper deals with the main aspects regarding the solidification of metallic composites processed by casting method and it is based on the general knowledge about casting of composites, practical results, and our own experimental data, especially in connection with the composites made up from aluminium alloys and different kinds of particles: graphite, silicon carbide, glass, and alumina. Information about technological problems which appear, specific defects caused by an insufficiently controlled solidification process, as well as some quality evaluation methods are also presented.

Keywords: solidification, casting, composites, metallic matrices, particles

1. Introduction

Aluminium and many of its alloys have a low density and high resistance at oxidizing and therefore they are the most used metallic matrix of composites with particles (Table 1) [1].

On the other hand, the mixing of components (matrix with particles) and the processing to near net shape by casting and solidification mean a low production cost (Table 2) [2].

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This is the reason why the paper deals especially with the composites with aluminium base matrix with particles, processed by casting-solidification.

Table 1

Some application of Al matrix/particle composites

System	Applications	Characteristics
Al/graphite	Cylinder block, connecting rods and pins, fan bearings, pistons and liners	Self-lubricating, cheap and light materials
Al/silicon carbide	Pistons, bearings, brake disks, bushings, manufacturing tools, hydraulic components used in aircraft, sport equipment	High tensile strength and wear resistance, good stiffness
Al/glass	Casting models, light and wear resistant parts	Good wear resistance, low weight
Al/alumina	Automotive components	High wear resistance

Table 2

Relative cost of composites

Method	**Cost**	**Complementary material**
Solid state diffusion Powder metallurgy Spraying deposition Casting	BIG ↑ SMALL	Mono-crystalline filaments Fibres Particles

2. Basic Principles

The dispersed material can determine a significant diminution of the crystalline grain size if it intensifies the nucleation process. Thus, for example, hypereutectic Al-Si alloys germinate heterogeneously on carbon, SiC, SiO_2, or Al_2O_3 supports. When a substratum participates to the nucleus formation: the number of atoms to be transferred from liquid to solid during the nucleus formation is smaller than for the homogeneous crystallization; the work necessary to create a separation surface nucleus-melt is less; the probability for nuclei with critical radius to appear is higher; the undercooling necessary to initiate the crystallization is smaller [4].

If θ is the wetting angle, the following relationship can be written:

$$\Delta G_{cr} = \Delta G'_{cr}\ f(\theta) \qquad (1)$$

where: ΔG_{cr} and $\Delta G'_{cr}$ are the critical free energy variation (necessary for a new heterogeneous and respectively a new homogeneous crystallization nucleus to appear) for the heterogeneous and respectively homogeneous nucleation;

$f(\theta) = \frac{1}{4}\left(2 - 3\cos\theta + \cos^3\theta\right)$, θ being the solid-liquid contact angle.

When the complementary phase does not stimulate the heterogeneous nucleation and does not intensify the heat transfer significantly, the crystalline grain size is even larger in comparison with the structure of the unreinforced material [3]. This phenomenon is due to the absence of convective streams.

One expects the reinforcement to affect the stability of the plane solidification front because it represents a diffusion barrier and determines a certain contact angle between the liquid alloy, solid alloy, and reinforcement surface. Thus, a curvature of the solid-liquid interface is produced. Dendrites developed into interstices smaller than the distance between the main branches (in free growth conditions) will have contorted secondary branches, trying to grow in the narrow space between fibres. When the solidification front meets an insoluble particle, two situations can appear:

- the solidified material retains the particle which remains inside the crystalline grain (obviously, if the particle is considerable smaller than the crystalline grain);
- the solidified materials does not include from the beginning the particle, which is pushed in the liquid phase and it is found between crystalline grains.

When the ratio between the thermal conductivity of the particle and the same property of the matrix is greater than one, the solidification rate will be diminished in front of the particle. Thus, a hollow appears in the solid phase and the particle will be included inside the crystalline grain [5].

3. Solidification of Aluminium Alloys-Graphite Composites

The graphite particles introduced in the aluminium matrix improve the wear properties of the matrix (friction coefficient, losses by abrasion). The high wear resistance is due to the presence of graphite particles in the contact zone of surfaces, the particles playing the role of a solid lubricant, diminishing the friction forces and therefore the heat released by friction. Used in proportion of 1...5 vol.% (with the average diameter of 10...100 μm) in the liquid alloy, the graphite particles have a strong tendency to segregate because the graphite has a lower density and it is not wetted by the metallic melt. The covering of particles with Ni or Cu is sometimes recommended, although it is an expensive process, because in this way the wetting conditions are improved and the dispersed material is included easier in the matrix.

Analysing the solidification structure, in the case of Al-Si/graphite composites, the graphite particles of 63...120 μm are pushed by the primary dendrites to the eutectic liquid, which solidifies the last. Graphite particles are not entrapped among the dendritic branches, because the distance between these branches is small (of about 20 μm).

In Al-Si alloys, the graphite stimulates the heterogeneous nucleation. Thus, the eutectic and primary silicon is agglomerated in the surrounding zone of the graphite particles. The large quantity of eutectic silicon can be explained on the basis of the rejection phenomenon of graphite at the interface *eutectic-primary dendrites of aluminium*, where the graphite will delay the solidification process, due to its low thermal diffusivity (in comparison with the matrix).

The induced modification becomes more intensive when the graphite proportion increases or its size becomes smaller. In the case of composites Al-Si alloy (eutectic or hipereutectic)/graphite (particles) cast in metallic mould, the eutectic silicon diminishes its size of about 37%, and the primary silicon becomes smaller by about 44%. For a concentration of 15%, the alloy is completely modified.

Experimental data were obtained by using a mechanical mixing installation with an impeller having two blades of steel and sand as well as cast iron moulds. To study the solidification structure test samples, of ϕ25x100 mm, were cast [6]. The matrix was made up from aluminium with 12%Si and 2%Mg, and graphite particles, introduced in proportions of 1...6 vol.%, had the diameter ranging in the range of 63...150 microns. The eutectic silicon crystals close to the graphite particle have a different morphology in comparison with those found at a longer distance of 30 μm around the particle. Near the graphite particles the eutectic silicon crystals have reduced dimensions, almost a globular shape. In the more distant zones dimensions of these crystals grow and their shape becomes acicular (specific to this structure). Fine structures were found in the narrow spaces between graphite particles, where the eutectic silicon crystals cannot develop too much or they were fragmented. The effect of stimulation of the nucleation processes is demonstrated by the tendency of primary silicon crystals to germinate on the graphite particle surface. The graphite presence of 3 and 5% diminishes the hardness by about 20%, but the friction coefficient can reach 0.2 or even 0.1 values. The friction is reduced due to the lubricant property of graphite as well as due to the absorption of water vapour and hydrocarbons from the environment into the superficial layer of particles. Even 2...3 vol.% graphite particles can reduce significantly the resistance against bonding. The distribution of particles in the matrix can be described with a non-uniformity coefficient, β, given by the following formula [7]:

$$\beta = \frac{\sum_{i=1}^{m} n_i^S - \sum_{i=1}^{m} n_i^I}{\frac{1}{k}\sum_{j=1}^{k}\sum_{i=1}^{m} n_{ji}}, \tag{2}$$

where, n is the particles density (number of particles per unit surface area); k – number of cross sections (equal distance sections made in a cylindrical test sample); m – number of analysed zones on each cross section of the sample; S and I refer to the upper and bottom position, respectively.

Technological aspects

- Graphite particles have the density of 2.18 g/cm^3 and consequently there is a difference of about 0.5 g/cm^3 in comparison with the density of molten aluminium alloy; that means their tendency to float and segregate to the upper side of the liquid material.
- In the case of centrifugal casting (with vertical axis) of cylindrical tube type castings from Al-graphite composite, the complementary material will be concentrated toward the rotation axis, that means to the internal cylindrical surface; this phenomenon can be useful to liners casting when a differentiated distribution of graphite in the castings wall is desirable.
- Improvement of wetting conditions can be realized by the following methods: the preheating of graphite particles, overheating of molten alloy, alloying of aluminium with magnesium and covering of particles with a thin layer of copper or nickel.
- The usual size of particles is ranged between 10 and 100 μm.

4. Solidification of Aluminium Alloys-SiC Composites

The aluminium – silicon carbide particle composites are frequently used because they are cheap and easy to be produced and have high tensile strength and elastic modulus. Nevertheless, the most important improved property is the wear resistance [8, 9]. Al-SiCp composites have low weight and a reduced thermal expansion coefficient for high particles contents (for example the thermal expansion becomes 12.10^{-6} °C^{-1} for 40 vol.% SiCp) [10].

Al/SiC composites have many applications, especially to realize moving parts of vehicles such as pistons, bearings, or brake rotors. For example, the brake rotor made of Al-9Si-0.55Mg/20 vol.% SiCp has the weight about half, conducts heat three times more efficiently, and reduces noise and vibration in comparison with the brake rotor made of cast iron (commonly used to produce this part).

In the Al-SiC system, in accordance with the equilibrium diagram, the compounds Al_4C_3 and Al_4SiC_4 can be formed as separate particles or like a continuous layer on the particles surface. The chemical reactions are:

$$4Al + 3SiC = Al_4C_3 + 3Si, \quad (3)$$

$$4Al + 4SiC = Al_4SiC_4 + 3Si. \quad (4)$$

The use of oxidized SiC particles assures improved wetting conditions, because SiO_2 reacts with Al and Mg.

In composites based on Al-Si-Mg and oxidized particles of SiC the compounds MgO and $MgAl_2O_4$ are formed and lead to the diminution of the Mg content from the matrix.

The formation of $MgAl_2O_4$ spinel is based upon the reaction:

$$2Al + 2SiO_2 + Mg = MgAl_2O_4 + 2Si. \quad (5)$$

The separation surface Al_4C_3-SiC is rough and encourages the phenomenon of stress concentration with several negative results on the properties of composites. The distribution of SiC particles in the solidified castings depends on [11]: the flocullation of SiC particles before being introduced in the liquid matrix, the agglomeration and segregation of SiC particles during and after their introduction into the melt, the movement of SiC particles in the melt, and the segregation of particles during the solidification. Detailed investigations made on Al-Si/SiCp composites have spotlighted the following aspects [12]. So, during the solidification of hypoeutectic Al-Si alloys, small SiC particles are rejected by the growing dendrites, especially at low cooling rates, but at high cooling rates, when the dendrite arm spacing is smaller than the particles size, they will not segregate. During the solidification of eutectic and hypereutectic Al-Si alloys, SiC particles are not rejected by the solidification front.

Particles of SiC, with oxidized surface, were added, in different concentrations, into an aluminium alloy with 2% Mg in a graphite crucible. The mixture was stirred by aid of a mechanical stirrer with two blades, the rotation speed being of 500 rot/min. Particles with size of 63…100 μm and smaller than 63 μm were used. The composite material was poured into a metallic mould (from cast iron), as well as in a sand mould and the obtained samples were cut in different sections on the height and processed for the metallographic analysis. Increasing the particles content (from 5 to 15 vol.%) the non-uniformity degree is greater, especially by the particles agglomeration phenomenon.

The particles with the size of 63…100 μm from the cast samples obtained in the metallic mould were not rejected by the solidification front because their size was larger than the dendrite arm spacing. In the case of specimens cast in the sand mould a part of small particles (< 63 μm) segregates at the grain boundary and the large particles (63…100 μm) segregated gravitationally.

Technological aspects

- Silicon carbide particles have the density of 3.21 g/cm^3, the expansion coefficient of $5.4 \cdot 10^{-6}{}^{\circ}C^{-1}$ and the elastic modulus of 324 GPa (at 1090°C), having the tendency to segregate by sedimentation in the molten aluminium alloy.
- To produce the hardening phenomenon of the matrix, silicon carbide particles must be small (of about 5...20 μm).
- Gases absorption during matrix processing is to be avoided during the matrix elaboration, because subsequently these gases are much more difficult to

be removed, taking into account that, generally, the silicon carbide content is large. Even when a simple mixing mechanism and gravitational casting the content of SiC particles can reach 10...20% (weight).

- Aluminium reacts with SiC, leading to Al_4C_3 compound formation, which diminishes the material casting fluidity and worsens mechanical properties.
- Agitation must be made till the beginning of casting, to avoid a large segregation by gravitational separation.
- Oxidation of SiC particles before their introduction in the molten aluminium alloy to prevent their rapid degradation due to the reaction between Al and SiC.
- The solidification duration must be as reduced as possible to diminish in this way the segregation of the disperse material into the matrix.
- Mechanical processing is very difficult and needs a big consumption of machining tools, so that is very important to use minimum processing additions.

5. Solidification of Aluminium Alloys-Glass Composites

Composites from aluminium-glass (particles) system represents materials that can be obtained by the simple mixing of components, because glass has the density very close to the density of aluminium alloys and can be used as light material with good wear resistance. Another advantage of glass is its almost nonreactivity with the molten aluminium alloys. Experimental data were obtained by using a matrix from Al-Si-Mg alloy. The agitation device, with controlled rotation speed (500...1000 rot/min) contains a rotating rod with the immersion zone made up from two stainless steel blades, covered with refractory paint. The diameter of the circle drawn by the free end of blades represents 82% from the agitation-casting ladle. After the assimilation of Mg and metal bath homogenizing, different amounts of glass particles, preheated at about 300°C, were introduced. Also, different sizes of particles were used [13], with the composition: 71.35 SiO_2, 15% Na_2O, 10% CaO, 2% MgO, 1.5% Al_2O_3, and 0.15 Fe_2O_3.

Glass particles can be used up to 700...750°C and have good mechanical resistance and elasticity modulus, reduced thermal conductivity (0.035...0.058 W/(m.K)), high corrosion resistance and they are not hygroscopic. The casting of the composite material, Al12Si2Mg/glass particles (30% weight, 75...150 μm diameter), was made at 650°C in cast iron moulds, preheated at 80°C. The test samples had cylindrical shape and several dimensions. In Figs. 1 and 2 some suggestive images regarding the particles inclusion as well as their influence on the matrix solidification are presented. Glass particles are found usually in the ultimate solidifying regions, due to their pushing toward the in developing dendrites. The rejection tendency of glass particles can be explained by their relatively large dimensions. So, permanently pushed by the in forming dendrites, the reinforcing particles will segregate at the grains boundary and in the ultimate solidifying regions. Glass particles have to a certain extent a modifying effect by the increase of the heterogeneous nucleation.

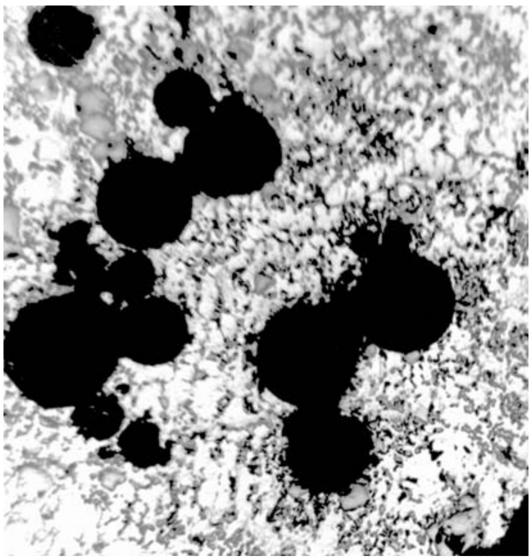

Fig. 1. Inclusion and sintering of glass particles in the matrix (x100); optical micrograph.

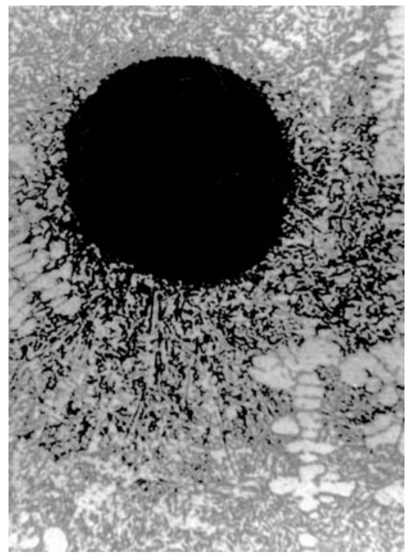

Fig. 2. Influence of glass particles on the heterogeneous nucleation (x200); optical micrograph.

Micro-shrinkage cavities have an un-regular aspect and appear in the ultimate solidifying regions, therefore just in the zone with an increased concentration of glass particles.

Technological aspects

- Glass particles have the density of 2.52 g/cm^3, close to the molten aluminium alloys, that meaning a reduced segregation tendency.
- Inclusion and entrapment of particles into the matrix are not technological problems; there are no defects in the separation surface of components.
- Spherical shape of particles eliminates the risk of stress concentration and microcracks to appear under the action of external forces.
- Castings must have a near net shape, as in the case of all composites with hard particles, because the mechanical processing is difficult.

6. Solidification of Aluminium Alloys-Al_2O_3 Composites

Alumina (the oxide Al_2O_3) is used frequently to reinforce the aluminium matrices. In comparison with SiC it is much more stable in the molten aluminium and much more resistant to oxidation. The covering of alumina particles with a thin layer of MgO improves the wetting conditions. The influence of alumina particles on the microstructure of Al-Mg/Al_2O_3 (particles) depends on their size [14]. Because the thermal conductivity of alumina particles is lower than the same property of the metallic matrix, the particles will block the thermal flow partially and will modify the thermal field. Due to its low thermal diffusivity, the alumina particles will delay the solidification process in their surrounding.

In Al-Mg/Al_2O_3 (particles) composites the large particles are surrounded by the eutectic phase. Fine particles of alumina (< 1 μm) are pushed by the solidification front at the grains limit, where they segregate and the particles with about 5 μm are included in the α phase. The grain size of the α phase is reduced when the agitation velocity increases, because the nucleation process is intensified by the fragmentation of dendritic branches (the fragments become nucleation nuclei).

Technological aspects

- Alumina particles have the density of 3.98 g/cm^3, the expansion coefficient of 7.92.10^{-6}°C^{-1}, the tensile strength of 221 MPa at 1090°C and the elasticity modulus of 379 GPa at the same temperature; the complementary material having the density significantly larger than the density of the usual molten aluminium alloys..
- For the inclusion of particles into the matrix, it is important to cover the reinforcing particles with a thin layer of Cu or Ni, the increase of mixing duration and the casting of mixture at as reduced as possible temperature, without affecting the quality of the mould cavity filling.
- In order to obtain a good dispersion of the particles into the matrix, it is recommended the casting in metallic moulds, where the solidification is produced rapidly, stopping in this way the segregation process.

7. Specific Defects and Quality Control Aspects

In the field of quality control of composite materials there are not yet unitary and general norms. Only some material categories prevalently used in military and aeronautic fields are subjected to control in accordance with some local norms or in finalizing stage norms. Typical defects in metallic composites raised during the solidification process or in connection with this process are:

- *draws*, usually found in gravitational cast composites, because commonly alloys have a coefficient of contraction at solidification of 3...6% and the presence of particles or fibers brakes the complete compensation of shrinkage micro-cavities with liquid from adjacent zones;

- *occluded gases* (*blowholes*), specifically in composites obtained by *VORTEX* proceeding, because gases from the environment are included in the matrix together with the complementary material and their solubility decreases dramatically during the liquid to solid transition;

- *agglomeration of particles or discontinuous fibers* that appear when the dispersion of complementary material is insufficient, specifically in applying *COMPOCAST* proceeding (with the metallic matrix in semisolid state);

- *segregation of the dispersed material* pushed by the solidification front to the last solidifying regions;

- *damage of particles*, caused by dissolving, melting or chemical reactions;

- *fissures and hot cracks* due to the stresses occurring during the cooling of the cast alloys (in braking contraction conditions).

Synthetically, in Table 3 the main type of defects which occur in casting, as well as the reasons for such defects to appear and prevention measures are presented [15].

Table 3

Defects in cast composites

Defects	Reasons	Prevention
DRAWS	Gases absorption during alloy processing and components mixing	Processing of alloy in a controlled atmosphere, advanced degassing of melt, avoidance of turbulence to casting
BLOWHOLES	Presence of particles that brakes the alloy compensation of shrinkage cavities	Directional solidification, controlled solidification
AGGLOMERATION	insufficient dispersion of particles in the matrix	Longer time to mixing, ultrasonic treatment of the mixture
SEGREGATIONS	Floating or settlement of particles due to the density difference	Intensification of cooling process, mould with high heat diffusivity use
DAMAGE OF PARTICLES	Dissolving, melting or chemical reactions	Diminution of contact time of components, avoidance of excessive alloy overheating, protective coatings
FISSURES, HOT CRACKS	Internal stresses	Avoidance of free contraction

At present the ultrasonic testing is one of the most widespread method in the field of metallic or nonmetallic composites, although there are some complications regarding their large degree of non-homogeneity [16].

Gross structure or particles structures produce a strong attenuation and the occurrence on the oscilloscope screen of difficult decipherable signals. Echoes, revealing the flaw are almost lost in the background noise, produced by untidy and multiple reflections given by the grain boundaries of the particles of composite material. Scattering is diminished significantly if the wavelength is larger by 10 times than the particle size.

Composite materials with aluminium matrix reinforced with particles of silicon carbide or alumina with the maximum size of 0.1 mm are examined using frequencies smaller than 6 MHz in the case of longitudinal waves and smaller than 3 MHz in the case of transversal waves. These values are due to the propagation velocities of the ultrasounds in aluminium: C_L = 6320 m/s and C_T = 3080 m/s.

If ultrasonic impulses are long, to cumulate several echoes is possible due to the reinforcement, leading to the false indication occurrence. In order to diminish the amplification effect due to these cumulated echoes it is recommended to use strong damped impulses with the ratio $d = A_1/A_2$ (A_1 being the maximum amplitude of the oscillation and A_2 the first harmonic) larger than 4.

The content of silicon carbide, porosity, and complementary material orientation are determined by the measurement of propagation velocity of wave through the tested material, using the reflected impulse method.

Both the transversal and longitudinal waves increase for a larger content of complementary material. On the other hand, the porosity (generally, created during solidification by the shrinkage or gases) produces an opposite effect. Thus, its increase means a diminution of the relative and propagation velocity. It was ascertained that the modification of the longitudinal velocity is stronger than the change of the transversal velocity. Using a beforehand-determined correlation or a suitable inscription of the oscilloscope the porosity as a function of wave propagation velocity can be established.

8. Conclusions

The analysis showed that although the cast metal-particle composites production seems to be a very simple process, however there are enough technological problems, mainly in connection with:

- the wetting of solid particles by the liquid alloy;
- the segregation and agglomeration of particles during *mixing*;
- the segregation of particles during *solidification*.

Therefore, the main problem is the difficulty to obtain an uniform distribution of particles in the metallic matrix or a controlled segregation of the complementary phase.

Nevertheless, there are some technical measures which can be taken to reduce these uncontrolled phenomena and to improve the quality of cast metal-particle composites.

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