Physical and Chemical Processes in the Production of Aluminium - SiC Whisker Composites by Squeeze-Casting

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Abstract

The results of a study of squeeze-casting SiC whiskers with aluminium melts are presented. Correlation is established between infiltration pressure, whisker and aluminium melt characteristics. Basic chemical characteristics in the process of whisker frame filling with the melt are defined. The effect of melt front movement direction on composite ingot quality is shown and the principal technological requirements for squeeze-casting are formulated.

Riassunto

Vengono presentati i risultati di uno studio della colata col sistema Squeeze casting di whisker e SiC con alluminio fuso. Una correlazione viene dimostrata fra la pressione d'infiltrazione, whisker caratteristiche del bagno d'alluminio. Vengono poi definite le caratteristiche chimiche di base del procedimento in cui la matrice preformata del whisher va riempita di alluminio. Si ilustra l'effetto sulla qualità del lingotto composito, del senso di movimento del fronte della colata. Infine, vengono formulati i principali requisiti tecnologici del metodo "squeeze casting".

Introduction

Manufacture of composites from aluminium alloys reinforced with discrete fibers or particles of refractory compounds, for example aluminium — SiC whisker composites, has recently become both the subject of detailed studies and widely used for products and structures [1-3]. These materials are characterized by a high level of physical and mechanical properties: high stiffness, high temperature strengh, thermal cycling resistance, wear resistance, low linear thermal expansion coefficient, etc., combined with moderate cost and suitability for mass production.

Both liquid and solid phase composite production processes are being developed simultaneously.

They exist in parallel and complement each other with regard to the further designation of a composite, ensuring its properties, production cost, etc.

A study of the basic physical and chemical processes for the production of aluminium — SiC composites by squeeze-casting is described in this paper.

Experimental results

SiC whiskers, used as the reinforcement, have been produced by the VLS process (fig. 1). This highly efficient method gives SiC whishers with a tensile strenght of 10 GPa and an elasticity modulus of 500 GPa, an average diameter of 0.12 μm and an average length of 15 μm.

Composite production requires the production of whisker preforms and their subsequent pressure infiltration with aluminium melts. The preform comprises the frame of discrete, spontaneously located fibers with a volume fraction from 8 to 20%.

Excessive pressure during infiltration results in the absence of spontaneous wetting and filling of the frame with aluminium melts. A preliminary theoretical estimation has made of the correlation between the required infiltration pressure and the preform and melt characteristics.

Calculations were based on the energy approach. Preform with the whiskers volume fraction (φ) is infiltrated under pressure (P) to a certain volume (ΔV).

Since the melt fills only spaces unoccupied by the preform, pressure work (A) is equal to:

$$A = P\,\Delta V(1-\varphi) \qquad (1)$$

The solid body-gas interface in the infiltrated part of the preform is replaced by solid body-liquid interface. This process requires the following energy consumption (E):

$$E = (\sigma_{sl} - \sigma_{sg}) S_{\Delta V} \qquad (2)$$

where: $S_{\Delta V}$ — area of whisker surface in the volume ΔV;

σ_{sl} — surface tension on solid body — liquid interface;

σ_{sg} — surface tension on solid body — gas interface.

$S_{\Delta V}$ can be expressed as follows:

$$S_{\Delta V} = S_{sp} \cdot \Delta V \cdot \varphi \cdot \varsigma_w \qquad (3)$$

where: S_{sp} — whisker specific surface; ς_w — density of SiC.

Equilibrium of surface tension forces in the contact point results in the well-known ratio:

$$\sigma_{sl} - \sigma_{sg} = -\sigma_{lg} \cdot \cos\theta \qquad (4)$$

where: σ_{lg} — surface tension on liquid — gas interface; θ — edge angle.

Equation 2 can now be written in the form:

$$E = \sigma_{lg} \cdot S_{sp} \cdot \cos\theta \cdot \varphi\ \Delta V \qquad (5)$$

Pressure work (A) is assumed to compensate energy losses due to interface changings, and is expressed by equality A = E.

By equation of the right-hand terms in equations 1 and 5 we obtain:

$$P_p = S_{sp} \cdot \varsigma_w\ \sigma_{lg}\ \cos\theta \cdot \frac{\varphi}{1-\varphi} \qquad (6)$$

Similar equations have been obtained with the pseudocapillary body approach to whisker preform approximation [4].

The experimental and calculated results display a sufficiently close agreement. The actual range of infiltration pressures is usually 5-7 MPa depending on preform density, whisher surface preparation, melt temperature, ect.

One of the key factors that significantly affects infiltration quality is the direction and nature of motion of the aluminium melt front when it penetrates the SiC whiskers preform.

Fig. 2 shows the ingot section of an Al — Mg — Si alloy base composite reinforced with 15% volume fraction of SiC whiskers. After infiltration, the sectioned part of ingot was heated to 500 C for 30 minutes. The figure also shows the Mg and Fe content through the ingot section.

The ingot was obtained by whisker preform preliminary vacuum degasation, immersion into the melt at a temperature 50 C higher than the liquidus point, and three-dimentional infiltration with argon excess pressure of the order of 5 MPa into a melt mirror.

Analysis of the mechanical properties of these ingots revealed enhanced plasticity and a lower strength of the "bright" compared with the "dark" zone. Examination of the content through the ingot section (fig. 2) showed the practically total absence of magnesium in the "bright" zone, together with 5 to 7 fold enhancement of the iron content. Enhanced porosity was also noted near the "bright" and "dark" zone interface.

It was clear that formation of "bright" zones is due to chemical reaction of the magnesium in the melt with the silica film on the whisker surface as the melt moves through the preform:

$$4\ Mg + SiO_2 \rightarrow Mg_2Si + 2\ MgO$$

Visually observed initiation of "dark" and "bright" zones was found experimentally to be caused by differences in the degree of oxidation of the alloy with or without magnesium.

Additional reactions can occur during composite production:

$$3SiO_2 + 4\ Al \rightarrow 3\ Si + 2Al_2O_3$$
$$3\ SiC + 4\ Al \rightarrow Al\ _4C_3 + 3\ Si$$
$$SiC + 3\ Mg \rightarrow Mg_2Si + Mg\ C$$

The last two are extremly objectionable reactions with regard to unstable and non-corrosive aluminium and magnesium carbides. They are directly connected with silica film availability and thickness on the crystal surface.

Experiments showed, however, that aluminium carbide formation is virtually nil when developed composite production techniques are used.

Magnesium carbides formation is observed only with rather high content of the order of 5-6% in the starting matrix alloy.

Concentration saturation level (fig. 2) due to oxide film thickness is reached when the Mg content of the alloy is 1-1,5%. The relation of "bright" zone quantity or volume depending on preform and alloy properties was calculated and experimentally supported:

$$\frac{V_{lo}}{V_d} = \frac{S_{sp}\ \varsigma_w}{\varsigma_{al}} \cdot C_{sur}\ \frac{100 - C}{C} \cdot \frac{\varphi}{1 \cdot \varphi}$$

where: V_{lo} — ingot "bright" zone volume; V_d — ingot "dark" zone volume; C — Mg concentration in matrix alloy; C_{sur} — Mg surface concentration, appropriate to saturation; ς_w — SiC density; φ — whisker volume fraction in the ingot.

"Bright" zone quantity obviously increases in function of ∅ and decreases in function of C. It rises linearly with C_{sur} on the whisker surface.

Increased iron content in the "bright" zone is presumably connected with the iron silicide content in the starting crystals. During penetration of the Mg-containing melt front, the following reaction occurs:

$$FeSi_2 + 4\ Mg \rightarrow 2Mg_2Si + Fe$$

It is followed by iron transfer due to the melt moving into the "bright" zone, where a new phase is formed:

$$Fe + Al + Si \rightarrow FeAlSi$$

Its composition is 20% Fe, 12% Si and 68% Al by weight.

Increased porosity near the "dark" and "bright" zones interface is due to shrinkage crystallization porosity, since the two zones have a different chemical composition, and different solidification temperatures, so that two crystallization fronts are formed near the interface.

Conclusion

Faulty phase initiation in the ingot center is observed in the composite manufacturing process by through infiltration, including preliminary vacuum degasation of SiC whisker preforms.

Reliable gas phase removal is very difficult to ensure on account of the free and critical moisture on the extremely developed whiskers surface, and the possibility of gaseous product formation as a result of contact between the melt and the crystal surface contact.

Squeeze-cast ingot porosity may reach 7-10%. Autoclaves are conventionally used in this process to meet enhanced safety engineering requirements and result in high cost and rather low efficiency.

In the most advantageous, the melt front moves plane — parallelly from the preform end. Removal of the gas phase and, in some cases, the objectionable interaction products, takes place beyond the ingot. The "bright" zone obtained with magnesium alloys can be shifted into the ingot end (fig. 3) or beyond it. In this case, high infiltration quality can be ensured without the use of vacuum equipment in open-casting moulds.

A production technology giving fault-free composite ingots with porosity $< 1\%$, uniform whisker distribution and ∅10-25% has been developed from these studies.

References

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Fig. 1:
SiC whisker X 5000.

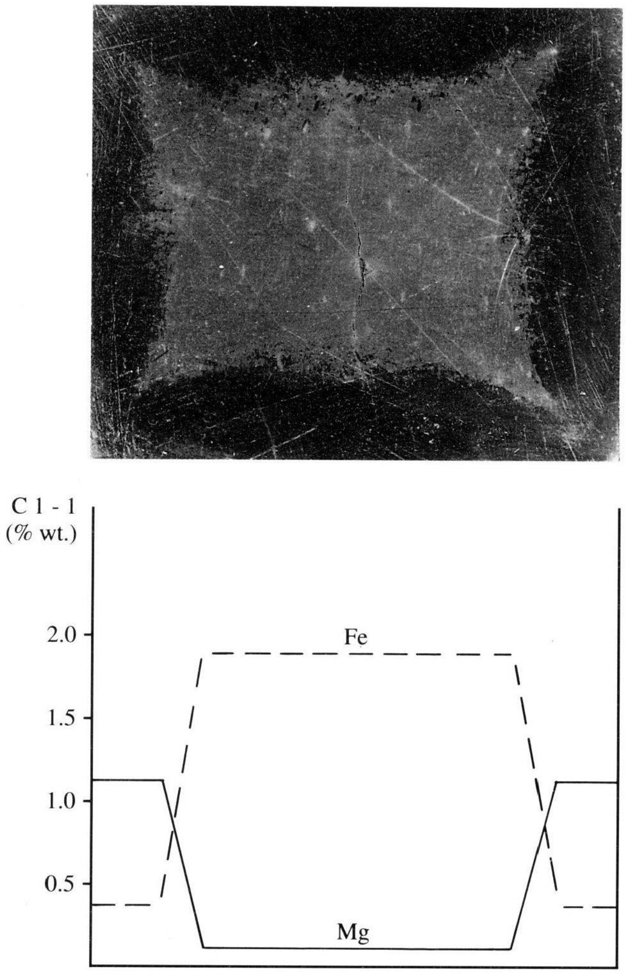

Fig. 2:
Ingot cross-section obtained in three-dimensional infiltration of preform and Mg and Fe distribution curves through section I-I.

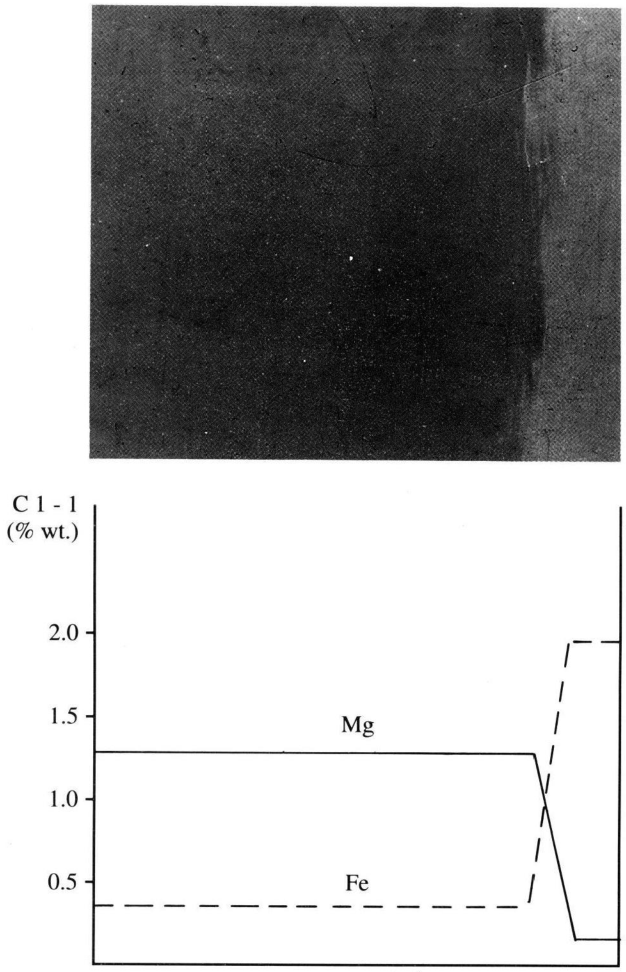

Fig. 3:
Ingot cross-section obtained in one dimensional infiltration of preform, and Mg and Fe distribution curves through section I-I.