Synthesis of semi solid slurry and effect of inoculants on microstructure of A356 alloy cast by rapid slurry forming (RSF) process

A. Sharma

Semi solid slurry of Al-356 alloy with 30% solid fraction is prepared by rapid slurry forming process. Effect of Al-5Ti-0.15C grain refiner alone and combined with Al-10Sr modifier is also studied on slurry produced by RSF process. Grain refining and modification of Al–Si alloys offer substantial benefits in casting processes. Finer grains ensure better mechanical properties, improved machinability, better feeding, while with modification the silicon morphology changes from flake to fibrous, resulting in improved properties, especially ductility. Combined addition not only replicates both effects but also gives the added bonus of better globularity in the SSM process.

Parole chiave: A356 alloy, RSF process, heat balance, enthalpy exchange, grain refiner, modification, microstructure

INTRODUCTION

Since the early 1970s, the rheological nature of metal was accidentally discovered during an investigation of hot tearing during casting of steel modeled by Sn-15Pb by Spencer under the supervision of Fleming at MIT [1]. Rheocasting involves the application of shear force during solidification to produce non-dendritic semi solid slurry that can be used to produce final cast product. The advantages associated with semi-solid metal casting involve improvement in the casting quality and technology. Uniform mixing of alloying elements in both liquid and solid phase minimizes tendency for macro and micro segregation. It also allows for eliminating micro-porosity of casting to the large extent and for reducing the solidification shrinkage. The process also improves the pressure-tightness of castings and diminishes the risk of hot cracking as compared with conventional casting techniques. At higher shear rate the viscosity of the alloy remains at a relatively low level even while the solid phase fraction reaches 50%.

Grain refining and modification of Al-Si alloys offer substantial benefits in casting processes [2-4]. Finer grains ensure better mechanical properties, improved machinability, better feeding, while with modification the silicon morphology changes from flake to fibrous, resulting in improved properties, especially ductility.

A number of rheocasting processes ("slurry on demand") are proposed for aluminium alloy; (i) Direct slurry forming(DSF),[5](ii) Sub liquidus casting(SLC),[6] (iii) New rheocasting(NRC),[7] (iv)Thixomoulding,[8] (v) Twin-screw rheocasting,[9] (vi)Semi-solid rheocasting(SSR),[10] (vii) Continuous rheocasting(CRP)[11] and Rapid slurry forming(RSF)[12]. The RSF process differs from other rheocasting processes because heat extraction and temperature control are not necessary.

RSF method has several advantages as easy control over the slurry temperature and initial temperature of melt, control over slurry cooling rate and enhances formation of globular primary phase. Besides this there is control over slurry process parameters like, size and amount of solid fraction, viscosity and castability by changing the thermal properties of a system.

The RSF process is based on enthalpy exchange of two alloy systems where one alloy is the low superheat melt (high enthalpy) and the other one act as the cold solid stirring material (low enthalpy) and is also known as enthalpy exchange material (EEM). An effective EEM dissolution is related to the temperature difference in the melt at the EEM interface. During the process, the EEM is immersed into the melt while stirring action is applied. Heat is absorbed from the melt during stirring operation. The crystallization nuclei arise at the surfaces of EEM during mixing process and are dispersed and uniformly distributed within the alloy volume by the centrifugal force, and the EEM itself melts. At slurry formation, during melting and dissolution of EEM, a new alloy system will form with a certain enthalpy level and solid fraction depending upon selected process parameters. The main advantage is that slurry can be produced without temperature control or outer cooling in a short time [13].

A. Sharma
Malaviya National Institute of Technology - Dept. of Met. & Mat. Engg. Jaipur-302017, (Rajasthan) India (email; ashok.mnit12@gmail.com)

The advantage of the method is that the relatively low solid phase fraction (20-30%) provides for achieving a globular structure with simultaneous retaining the high fluidity of suspension, and the preparation of the suspension is much shorter and cheaper than for other methods.

ASSUMPTIONS AND EQUATIONS USED IN RSF PROCESS

Some assumptions are taken to get the SSM temperature for the RSF process with the help of heat balance equation.

1. The temperature gradients do not exist in the slurry and its temperature depends only on time as mixing causing the temperature to be even within the volume of melt.
2. The ideal insulation between liquid metal and crucible wall is assumed. Therefore, there is no heat transfer between the melt and the crucible wall.
3. The solid fraction is known in the final slurry.
4. The slurry forms at a given temperature within solidus and liquidus region.
5. Temperature-independent thermo-physical properties of the system are assumed.
   The initial temperature of the melt which is to be kept for obtaining the desired solid fraction in the slurry is calculated with the help of the heat balance between the melt and the EEM. A schematic diagram used for the RSF set up is shown in the Fig. 1.

Amount of heat emitted by the liquid melt during cooling is given by,

$$dQ_1 = m_{melt} \times C_{P\,melt} \times (T^{melt} - T^{ssm})$$

Amount of heat emitted by the melt due to partial solidification is given by,

$$dQ_2 = \Delta H_{melt} \times f_s \times m_{melt}$$

Total amount of heat emitted by liquid

$$(dQ_{out}) = dQ_1 + dQ_2$$

$$dQ_{out} = m_{melt}\, C_{P\,melt} \times (T^{melt} - T^{ssm}) + \Delta H_{melt} \times f_s \times m_{melt} \qquad (1)$$

where, m_{melt}, $C_{P\,melt}$ are mass and the specific heat of melt respectively; f_s is final solid volume fraction in the slurry; T^{melt} and T^{ssm} are the temperature of the melt and the semi-solid slurry and ΔH_{melt} is latent heat of melt.

Amount of heat absorbed by the EEM during heating is given by,

$$dQ_3 = m_{eem} \times C_{peem} \times (T^{ssm} - T^{eem})$$

Amount of heat absorbed by the EEM during partial melting is given by,

$$dQ_4 = \Delta H_{eem} \times (1 - f_s) \times m_{eem}$$

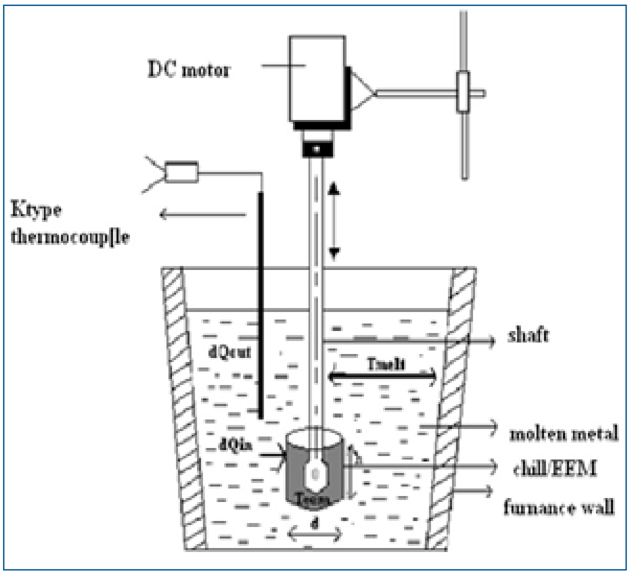

Fig. 1 - Schematic diagram of heat exchange during slurry formation in RSF process

Fig. 1 - Diagramma schematico dello scambio termico durante la formatura di slurry nel processo RSF

Total amount of heat absorbed by the EEM$(dQ_{in}) = dQ_3 + dQ_4$

$$dQ_{in} = m_{eem} \times C_{peem} \times (T^{ssm} - T^{eem}) + \Delta H_{eem} \times (1 - f_s) \times m_{eem} \qquad (2)$$

where, $m_{eem} = V_{eem} \times \rho_{eem}$; V_{eem}, ρ_{eem} and $C_{p\,eem}$ are volume, density and the specific heat of EEM respectively. fs is final solid volume fraction in the slurry. T^{eem} and T^{ssm} are the temperature of the EEM and the semi-solid slurry respectively. ΔH_{eem} is latent heat of the EEM.

By equating equation (1) and (2), the initial temperature of melt can be written as,

$$dQ_{out} = dQ_{in}$$

$$T^{melt} = \frac{1}{C_{Pmelt}} \left[\frac{m_{eem}}{m_{melt}} \left(C_{Peem}(T^{ssm} - T^{eem}) + \Delta H_{eem}(1 - fs)\right) - \Delta H_{melt}\, fs \right] + T^{ssm} \qquad (3)$$

In the above equation all thermo-physical factors are known except the temperature of the slurry, which can be find out with the help of the Schiel's equation and is given by,

$$T^{ssm} = T_f - (T_f - T_L)(1 - f_s)^{k-1} \qquad (4)$$

where,

T^{ssm} is the final temperature of the slurry; T_f is the melting point of Aluminum; T_L is the liquidus temperature; fs is the solid fraction; k is the equilibrium partition coefficient.

The EEM is taken cylindrical for effective mixing. The mass of the EEM can be calculated by applying simple mathematics.

$$m_{eem} = V_{eem}\, \rho_{eem} = \frac{\pi d^2 h}{4}\, \rho_{eem}$$

EXPERIMENTAL PROCEDURE

Preparation of semi solid mass

The rod with EEM (5 cm diameter and 7.3 cm length) was attached to a motor having rotation speed up to 2000 rpm. 1.35 kg of A356 was taken in a graphite crucible and melted in an electric resistance furnace maintained at 720°C temperatures. A K-type thermocouple calibrated using a CAL-402-3/4 digital calibrator is used to measure the temperature of the melt. After melting, 0.2 % grain refiner as Al-5Ti-0.15C was added in to the melt to facilitate good nucleation potential. This was followed by adding 0.02% Al-10Sr as modifier. After which the degassed melt was kept for 15 minutes before starting stirring. The EEM initially at room temperature was slowly immersed into the melt and stirring started with preset rotational speed of 1000 rpm with the help of D.C motor. Fig. 2 shows various steps involved in RSF process.
After this, formation of semi-solid slurry took place within 35 seconds. This was followed by transferring the slurry in to water for quenching to restore the microstructure at a given temperature. Thus slurry having 30% solid fraction could be prepared. Later on solidified compact was cut for metallographic studies. Sample preparation for optical microscopy was done by standard metallographic technique and samples were etched with Keller's reagent. For SEM studies the metallographic samples were deep etched.

RESULTS AND DISCUSSION

Slurry formation through RSF process

RSF technology is able to extract a very large amount of heat through enthalpy exchange of similar material in a very short time. This also improves the castability of the alloy significantly. The reason being that through RSF technology, the morphology of the primary phase changes from coarse dendrites to near globular, which eliminates the feeding problem during solidification that is associated with conventional casting method[14].
The slurry formation assumed to occur below $T_{liquidus}$ when the EEM is completely dissolved. Stirring causes a formation of non dendritic primary α-Al phase. An effective EEM dissolution is related to the temperature difference in the melt at the EEM interface. As the EEM is immersed into the melt it becomes rapidly heated which reduces the melt temperature locally [15-16]. At the same time the temperature difference in the melt at the EEM interface is reduced. To obtain a quick dissolution, the melt around the EEM must be continuously replaced by new superheat melt. This is provided by the rotation of EEM giving forced convection in the melt. Nucleation during slurry formation is assumed to be massive and occurring close to the EEM. Proper nucleation is dependent on forced convection since the melt temperature initially decreases locally around the EEM. During stirring the nuclei will be thrown out in to the melt and the time spent in it decides if further growth is allowed or suppressed by the bulk liquid. At the same time as the nuclei are forced out into the melt, hot melt

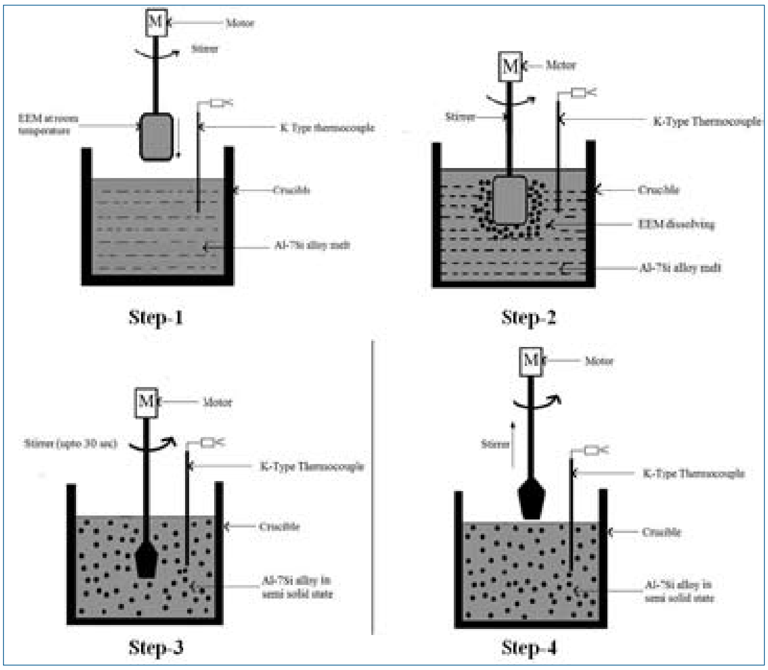

Fig. 2 - Steps of RSF process

Fig. 2 – Successione di passaggi del processo RSF

flows in towards the EEM. Local nucleation will therefore take place repeatedly. Proper nucleation is dependent on forced convection since the melt temperature initially decreases locally around the EEM. Addition of grain refiner further promotes nucleation and subsequent growth. Al-5Ti-0.15C grain refiner releases TiC and $TiAl_3$ particles which are stable in the melt. These particles act as nucleation sites for α-Al phase when solidification starts. Al-10Sr acts as modifier which restricts the growth of Si particles. Thus net result is fine α-Al globular phase with finer eutectic Si. Rapid heat extraction, potent nucleation and force shearing action during initial stage of solidification, lead to the formation of RSF slurry. The globular structure of α-Al is believed to a be a result of spherical growth under forced convection as is shown in Fig. 3.

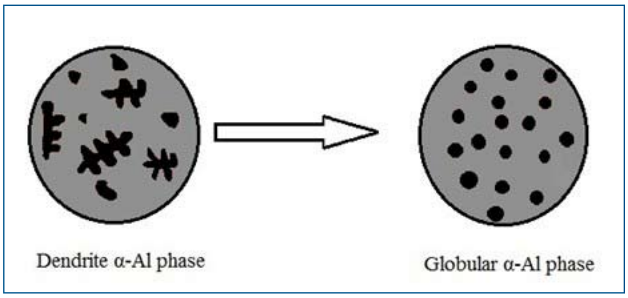

Fig. 3 - Morphology of the primary phase changes from coarse dendrites to globular

Fig. 3 - Cambiamenti nella fase primaria, da morfologia con dendriti grossolane a quella globulare

Analysis of coalescence by shear flow

During RSF process shear forces will induce two kinds of effects as given below;

(a)It will lead to collision of particles. Rate of coalescence is given by;

Rate of Coalescence= Rate of Collisions × Coalescence Efficiency

(b) Stirring will induce turbulence.

The stirring of the slurry might induce coalescence of the primary phase particles by turbulent diffusion. In turbulent diffusion, mass is transferred through the mixing of turbulent eddies within the fluid. This is basically different from the molecular diffusion. In turbulent diffusion, the random motion of the fluid is responsible for mixing. The eddy diffusion coefficient depends on the properties of the fluid flow, where flow velocity is very important factor. Turbulence is only present at flow velocities above a critical level. The degree of turbulence is correlated with velocity. In other words, the presence or absence of turbulence depends on the Reynolds Number, which depends on velocity, width of the crucible and the viscosity of the fluid. The degree of turbulence also depends on the material over which the flow is taking place. The flow over bumpy surfaces will be more turbulent than flow over a smooth surface. The increased turbulence will cause more rapid mixing. In addition to this, the value of the eddy diffusion coefficient depends to some degree on the size scale of the problem under question.

In Al-7Si alloy, the primary α-phase particles are typically larger and are thus not entrapped into the turbulent eddies. Therefore coalescence by eddy diffusion is unlikely. However, in the initial stages of the nucleation and growth, when the growing spheroidal α-phase particles are smaller in size they might get entrapped in the turbulent eddies [17]. Hence, in the initial stages, coalescence by stirring plays a role which results in much higher growth rates.

Microstructures of A356 ally under different conditions

The optical photomicrograph of as cast A356 alloy is shown in Fig. 4(a). As cast alloy shows α-Al dendrites and

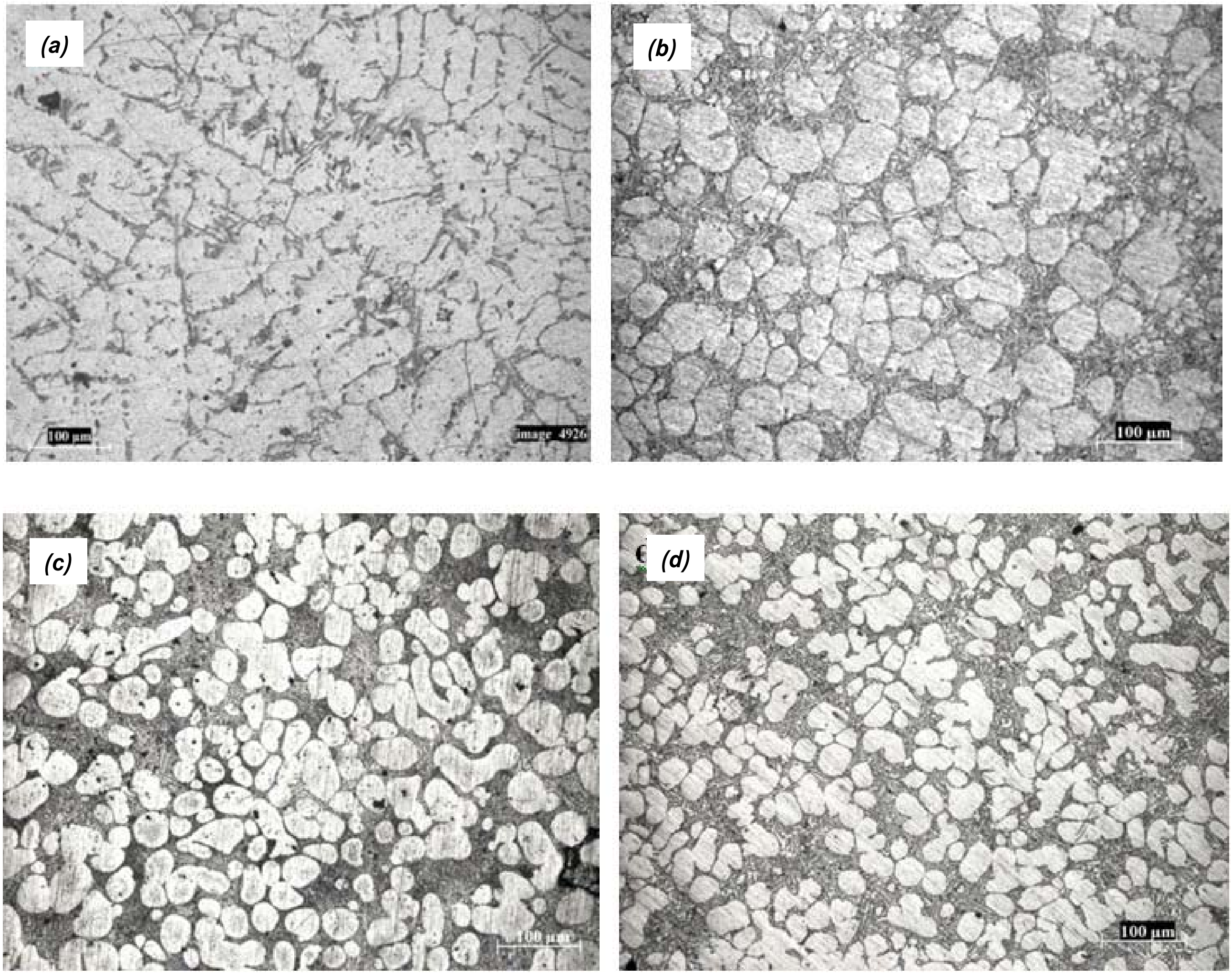

Fig. 4 - Optical microstructure of A356 alloy (a) in as cast condition and (b-d) with RSF process; (b) without any inoculation (c) with grain refiner (d) with grain refiner and modifier

Fig. 4 - Microstruttura della lega A356 in microscopia ottica (a) in condizione as cast e (b-d) con processo RSF; (b) senza alcuna inoculazione (c) con affinatore di grano (d) con affinatore e modificatore di grano

coarse eutectic silicon in the interdendritic region. A large nucleation rate is necessary for effective grain refinement and also the survival rate of nuclei is important. Therefore for α–Al refinement it is necessary to ascertain that majority of heterogeneous nuclei can survive and contribute to final microstructure. This is possible through RSF process with addition of grain refiner. Fig. 4(b) shows globular α-Al grains of about 60 µm in size cast by RSF process.

Higher shear rate causes intensive mixing in the melt during stirring operation due to which homogeneous dispersion takes place. This increases tendency for heterogeneous nucleation. Hence grain refinement becomes very effective. With addition of Al-5Ti-0.15C master alloy the structure is not only refined but also the sphericity of the primary α-Al particles increases (Fig. 4c). The higher globularity coupled with smaller primary Al leads to better flow of semi solid slurry. The combined addition of grain refiner and modifier is shown in Fig.4d. Due to Sr modification, the eutectic silicon particles change from coarser to finer morphology. This is due to the growth nature of the fibrous structure of the Si in the eutectic mixture which encompasses the primary α-Al phase without being a continuation of it. Therefore, the globules are indeed true globules when Sr is added. Fig. 5 shows that due to grain refinement and modification α-phase particles size decrease.

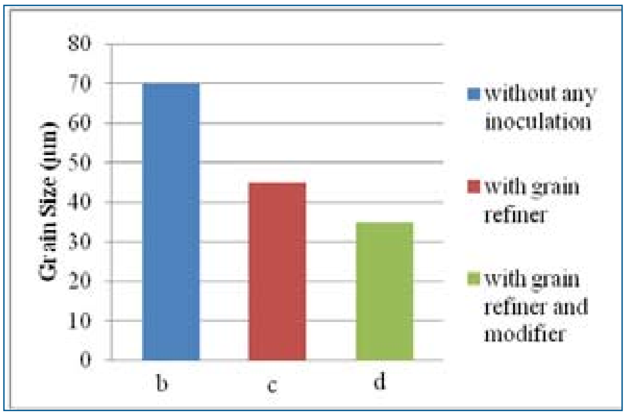

Fig. 5 - Effect of inoculation on α-phase particle size

Fig. 5 – Effetto dell'inoculazione sulla dimensione delle particelle di fase α

Fig.6 (a-c) show SEM micrographs of A356 alloy in as cast, refined and refined and modified conditions respectively. Fig. 6a shows SEM microstructure of A356 alloy cast through RSF process (with out addition of any grain refiner or modifier). In this case coarser Si needles are seen between globular primary Al phase. It appears that shear forces generated during process did not effect the morphology of Si particles and Si remained coarse. However some rounding of primary phase took place. Fig. 6b shows the effect of grain refinement on RSF cast A356 alloy. It is observed that grain refinement has changed the coarser Si particles to some what finer Si particles in addition to making primary phase more globular. Fig. 6c is showing combined effect of grain refiner and modifier on RSF A356 cast alloy. In this case the coarser Si particles have changed to very fine Si particles and globularity of primary phase has also increased. Grain refining efficiency is obvious through the size reduction of the globules and the presence of more primary particles.

With addition of grain refiner, the nucleation and growth of α–Al particles takes place at little higher temperature (~4°C). The refiner addition gives rise to an increase in the α-Al nucleation temperature due to the presence of more potent and effective nucleants in the bulk liquid. Eutectic silicon is modified by the addition of 0.02 wt% Al-10Sr. With the addition of modifier the eutectic temperature line depresses to lower temperature (~7°C). If both inoculants perform in their respective way in the combined form, wider solidification range will be available. Wider solidification range may not be good for conventional casting. However, it may be quite interesting for semi solid forming. Thus grain refining and modification have lead to globular α-Al phase with very fine eutectic phase.

With the addition of the grain refiner, the number density of α-Al increased. The eutectic phase in microstructure seen in between the globular α–Al particles shows the melt before quenching. This was the liquid part of the semi solid slurry.

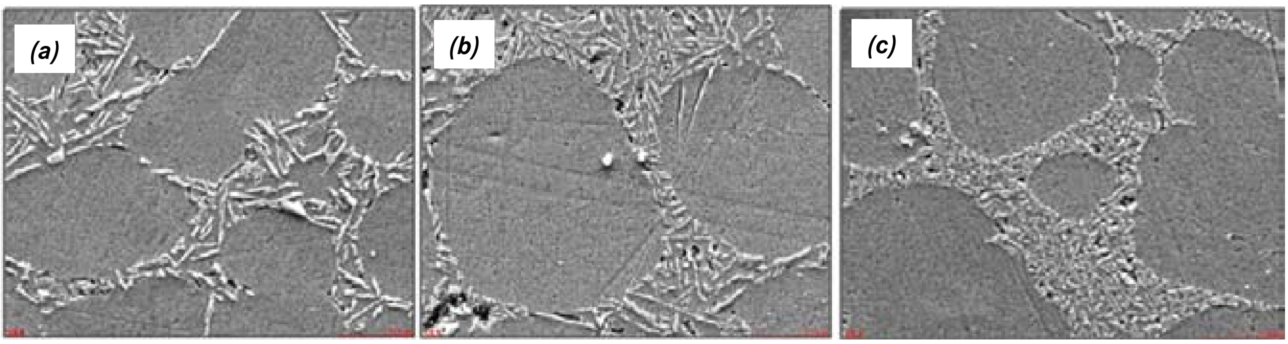

Fig. 6 - SEM micrographs of A356 alloy in different conditions; (a) as cast, (b) grain refined and (c) grain refined and modified

Fig. 6 - Micrografie SEM della lega A356 in differenti condizioni; (a) as cast, (b) con affinazione del grano e (c) con affinazione e modifica del grano

CONCLUSION

With addition of refiner and modifier, the nucleation and eutectic reactions not only replicates both effects but also gives the added bonus of better globularity in the RSF process.
Following factors also helped in obtaining the required microstructure.
(a) Uniform temperature and chemical composition maintained throughout the liquid alloy.
(b) Nucleating agents were well dispersed.
(c) Rapid extraction of latent heat. Under such condition each nuclei survived and contributed to refinement of final microstructure.

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Sintesi di slurry semisolido ed effetto di inoculanti sulla microstruttura di getti in lega A356 mediante processo RSF - (Rapid Slurry Forming)

Parole chiave: Alluminio e leghe, processi

Lo slurry semisolido in lega Al-356 con frazione solida del 30% è stato preparato mediante il processo RSF - Rapid Slurry Forming. Nel presente lavoro è stato studiato - sullo slurry prodotto mediante il processo di RSF - anche l’influenza come affinatore del grano di Al-5Ti-0.15C, da solo e in combinazione con il modificatore Al-10Sr. L’affinazione del grano e la modifica delle leghe Al-Si offrono notevoli vantaggi nei processi di fusione. Il grano fine garantisce infatti migliori caratteristiche meccaniche, migliore lavorabilità e alimentazione, mentre la modifica di composizione permette di ottenere cambiamenti nella morfologia del silicio da fiocco a fibroso, con conseguente miglioramento delle caratteristiche, in particolare della duttilità. Inoltre l’aggiunta combinata non solo replica entrambi gli effetti, ma comporta anche il vantaggio di una migliore globularità nei processi di fonderia in semisolido.