MIXING AND SOLIDIFICATION PROCESSING OF Al- Al_2O_3 COMPOSITE

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Abstract

In the present investigation, mixing has been carried out by dispersing fine alumina particles (Avg. size- 25 μm) in molten Al-Mg alloy using a turbine blade stirrer with impeller diameter 'd'. The molten alloy has a depth of H when kept in a crucible of diameter D and the stirrer is placed at height 'h' from its bottom. The dimensionless variables, d/D and h/H, have been varied to observe the dependence of optimum values of these variables on stirrer geometry. These studies are expected to contribute to optimum processing route for stircasting of composites. In the current investigation, it has been observed that a turbine stirrer incorporates higher amount of particles in the ingot cast out of a slurry mixed by it as compared to a flat blade stirrer in the range of experimental parameters used. But the pore-particle ratio is also higher in the cast ingot processed by a turbine stirrer. In the cast ingots processed by a turbine stirrer the particle content increases when d/D ratio increased from 0.33 to 0.48 but a further increase of d/D to 0.62 did not change significantly the particle content. A similar trend in pore-particle ratio is also observed and the lowest pore-particle ratio was observed for d/D=0.62. For mixing a slurry of molten alloy with poorly wetting particles it appears that the recommendation of chemical engineers to keep the stirrer at h/H=0.35 may not work as well from the point of view of particle content in the ingot cast with the slurry. By enhancing h/H to 0.5, the particle content increased significantly and a lower pore-particle ratio was also achieved.

The microstructural evidence clearly indicates formation of bubbles with particles adhering to it and even their coalescence during processing. Some of these bubbles are eventually invaded by the molten alloy. When the manner of stirring is changed from continuous four minutes to an interrupted one of two minutes each intervened by a two minute period of cessation of stirring, it has been observed that the particle content at the top reduces but that at the bottom increases making particle content more uniform along the length of the ingot. This has been attributed to enhanced floatation of bubbles containing particles and reincorporation of the latter. During interrupted stirring the porosity did not reduce as much as the particles and the pore-particle ratio at the top increased. Enhanced processing time may have contributed to

increased dissolution of gases contributing to porosity and so, a reduced extent of bubble-particle combine did not reduce the overall porosity as much as it was expected. When the stirring was interrupted for a longer time of 10 minutes the uniformity of the ingot in respect of particle content improved to an extent but the overall porosity increased significantly. These facts possibly indicate that most of bubble-particle combines form during mixing and not during solidification.

Introduction

Solidification processing of metal matrix composites containing ceramic particles is influenced by (a) wetting between molten alloy and the ceramic dispersoids [1,2], (b) segregation of dispersoids during its mixing into the melt [3] and during solidification [4] and (c) porosity [5]. While mixing relatively coarser alumina particles (Avg. size ~70μm) in molten Al-Mg alloy with the help of a flat blade stirrer, Ghosh and Ray [6] have investigated the influence of the process variables during mixing on particle content and porosity in a cast composite ingot. The most significant process variables for mixing, as identified by earlier investigators [7], are - (a) stirrer diameter, characterized by dimensionless parameter d/D, where d is the diameter of the stirrer and D is the diameter of the container at the surface of the liquid, (b) position of the stirrer inside the liquid, characterized by dimensionless parameter h/H, where h is the height from the bottom of the container at which the stirrer is placed and H is the depth of liquid in the container, and, (c) stirring speed. Ghosh and Ray [6] considered temperature, T, of the melt as an additional variable in the context of melt-particle mixing so as to include also the mixing of particles in semi-solid alloy.

In the earlier studies [8] it has been observed that the overall particle content in the cast ingot is maximum at optimum values of d/D, h/H and the stirring speed which determine the flow pattern developing in the molten alloy. El-Kaddah and Change [9] has observed that there is a critical stirring speed below which the lift force in the flow is not able to overcome the settling of particles. Thus, one understands broadly the importance of variables of stirring in controlling the particle content. Unfortunately, it has been observed that the porosity content in the cast ingot is also maximum at the same values of d/D, h/H and the stirring speed where particle content is maximum. Examination of particle content and porosity in a large number of composite ingots cast under a variety of conditions has shown an almost linear band of variation of these two parameters. On the basis of these findings it has been conjectured that either both particle and porosity enter the melt through the same mechanism or the particle stabilizes the porosity inside the melt or both the mechanisms are taking place during processing. Under such conditions one may expect an almost linear correlation of porosity with particle content in the cast ingot as it has been observed.

The chemical engineers use turbine stirrers extensively for their ability to handle effectively agitating operations under a wide range of viscosities [10,11]. It has been recommended that d/D of 1/3 should be used. However, it should be remembered that the chemical engineers generally use solid - liquid systems where the particles are wettable by the liquid whereas mixing in the context of solidification processing of metal matrix composites generally involves ceramic particles not wetted easily by the molten matrix alloy in systems of commercial interest. Ray [1] observed that magnesium promotes wetting of alumina by molten aluminum. A detailed investigation of contact angles by Lijun et al. [12] has shown that the contact angle between alumina and aluminum melt reduces by about 40 degrees at 700°C due to evaporation of magnesium weakening the surface film on molten aluminum but still, no wetting has been observed below 1000°C.

The present investigation has been carried out by mixing finer alumina particles (Avg.size ~ 25 μm) with molten Al-Mg alloy using a turbine blade stirrer. The d/D and h/H has been varied to observe the dependence of optimum values of these variables on stirrer geometry. The mode of stirring has also been varied to see the effect of it on particle and porosity content. These studies are expected to contribute to the evolution of optimum processing route for stircasting of composites.

Experimental

1. Solidification Processing

Solidification processing of aluminum based composite containing alumina particles has been carried out by stircasting using axial turbine impellers as shown in Fig.1. The following process variables have been varied to determine their influence on particle and porosity contents in the resulting composite - (a) d/D = 0.33, 0.5 and 0.62, (b) h/H = 0.35 and 0.5, (c) pouring temperature controlled in the range between 645°C and 700°C, and (d) manner of stirring - continuously stirred for four minutes before pouring denoted by C/4 or stirred for two minutes before ceasing it for two or ten minutes and then stirred again for two minutes, termed interrupted stirring, denoted respectively by I/2/2/2 and I/2/10/2. The schematic diagram of the experimental setup used in this investigation is shown in Fig.2. It consists of a clay-graphite crucible inside a resistance heated vertical muffle furnace and the crucible has a hole of size 12 mm at the bottom, plugged with graphite stopper. Pitched-blade mild steel stirrer of diameters as indicated above, are used. The blades are welded with a pitch angle of 45 degrees. In order to prevent dissolution of the stirrer in molten aluminum alloy, it is coated by a slurry of alumina powder in sodium-silicate and dried.

The speed of the stirrer is kept constant at 9.5 revolution per second (rps). A strobometer is used to measure the speed of the stirrer. The position of the stirrer inside the melt is varied by adjusting the length of the stirrer shaft. The depth at which the stirrer blades are placed from the surface of the melt and the total depth of the molten alloy are measured by dipping a graduated scale into the melt. The temperature of the melt is measured by using a digital temperature indicator with a chromel-alumel themocouple placed 15-20 mm inside the melt.

Commercially pure aluminum weighing about 500 g is melted in a clay-graphite crucible and heated to 730°C. Magnesium is added to the melt at 710°C and then the melt is cleaned by skimming. The melt is agitated vigorously by a stirrer preheated to prevent quenching of the melt. Depending on the particle content desired in the composite, weighed amounts of alumina particles of average size 25 µm, is wrapped inside aluminum foil and preheated to 400°C. These particles are then added to the molten alloy by immersing the aluminum wrapper containing the particles. The addition rate is so controlled that the particles enter the melt approximately at a rate of 1.5 to 2.5 g/sec. For continuous stirring, the melt and the particles are mixed by stirring continuously. While for interrupted stirring, the melt and the particles are stirred and then stirring is stopped for a definite interval of time before stirring the melt-particle slurry again. During stirring, the temperature of the slurry is maintained within ±5°C of the pouring to pour melt-particle slurry in a 30mm x 30mm x 300mm preheated steel mould coated by a slurry of graphite and bentonite clay in water. While pouring, the remaining melt-particle slurry in the crucible is continuously stirred. The ingot is quenched by spraying water on it.

2. Estimation of particle and void content.

For each cast ingot two specimens are taken, one from the bottom and the other from the top of the ingot just below the shrinkage pipe. The density of these specimens is determined by using weight loss method. To determine particle content, each specimen is dissolved in dilute hydrochloric acid and the alumina particles left behind as residue, are filtered out in ashless filter paper. The filter paper along with alumina particles is burnt in a ceramic crucible of known weight. The crucible is then cooled and weighed along with alumina particles to determine weight percent of alumina in the composite.

The volume of the composite, V_c, is equal to the total of the volume of the matrix alloy, V_m that of the alumina particle, V_a and that of the voids, V_p . But the weight of the composite, W_c , is equal to the total of the weight of the matrix alloy, W_m , and that of the alumina particles, W_a. Therefore, the density of the composite, ρ_c may be written as,

$$\rho_c = \frac{W_c}{V_c} = \frac{W_m + W_a}{V_m + V_a + V_p} \qquad (1)$$

The terms in Equn. (1) may be rearranged to find the volume of porosity as

$$V_p = W_m\left(\frac{1}{\rho_c}-\frac{1}{\rho_m}\right)+W_a\left(\frac{1}{\rho_c}-\frac{1}{\rho_a}\right) \qquad (2)$$

where, ρ_m is the density of the matrix alloy i.e., Al-4wt%Mg which is taken as 2.6812 g/cm^3, and ρ_a is the density of the alumina particles which has been measured as 3.4 g/cm^3.

Results and discussion

In order to correlate the particle content with processing conditions it is necessary to minimize the effect of solidification on particle distribution. The use of permanent metallic mould has been made to achieve a higher velocity of solidification front so that it may be higher than the critical velocity of particle pushing. Even if the particles are pushed during dendritic solidification it is expected to be only over microscopic distance without altering the macroscopic distribution significantly. Further, a reduced solidification time in permanent mould reduces the extent of settling or flotation of particles in the mould. So, the particles incorporated in the ingot may be assumed to have a similar distribution as that existing in the liquid metal after pouring. For the purpose of this investigation, the particle and porosity distribution in the ingot has been characterized by particle and porosity contents both at the top, just below the shrinkage pipe and at the bottom of the ingot.

Particle incorporation has been parametrized in terms of particle recovery which is the ratio of weight percent alumina in the sample to that added in the melt expressed as percentage. Fig.3(a) shows the variation of particle recovery of alumina at different ingot height parameter l/L in an ingot when l is the height from the bottom of the ingot at which the sample has been taken to determine alumina content and L is the total usable height of the ingot from the bottom to the point below shrinkage pipe. Since 6wt% alumina has been added in all the experiments reported in Fig.3(a), particle recovery is directly proportional to particle content in the sample. It is observed that the extent of particle incorporation and particle recovery are significantly enhanced when one increases the d/D ratio of the impeller from 0.33 to 0.48. However, on further increase of d/D to 0.62 the extent of average particle recovery does not significantly change and it remains around 25-30% on the average. In case of flat blade stirrer it has been observed by Ghosh and Ray {6} that the particle content attains a sharp maximum and particle content reduces drastically when d/D is changed from the optimum value of d/D = 0.63 recommended for use in melt-particle stirring. Such a sharp variation of particle content with d/D has not been observed for turbine stirrer. The variation of pore-particle ratio i.e., the ratio of vo1% porosity and particle recovery, with ingot height parameter shown in Fig.3(b), reveals that a stirrer with higher d/D ratio of 0.48 and 0.62 result in much lower porosity relative to particle recovery than that for a stirrer with lower d/D of 0.33. Even if mechanisms like suction through vortex is considered responsible for incorporation of both the porosity and the particles, their relative proportion may be influenced by d/D ratio. The flow pattern and the velocity gradient are also influenced by the d/D ratio and it could affect the size of the bubble-particle combine and hence the pore-particle ratio. It has been recommended by the chemical engineers [11] that the turbine stirrer should be so placed as to have 35% liquid below and 65% liquid above it, the position of the stirrer has been kept fixed at a height ratio h/H = 0.35 for all the experiments whose results are reported in Fig.3. The pouring temperature for these experiments has been kept in the close range between 660°C and 680°C.

When the position of the stirrer is raised so that h/H changes from 0.35 to 0.5, it is observed that the particle content has more than doubled while the pore-particle ratio has reduced as shown in Fig.4(a). In this context it should be noted that for flat blade stirrer Ghosh and Ray[8] has recommended use of h/H = 0.8. Thus, it appears that recommendations in respect of h/H for mixing melt -particle slurry will have to be different from those of chemical engineers. In these experiments whose results are reported in Fig.4(a), stirrer with d/D ratio of 0.48 has been used and the pouring temperature has been about 670°C. A comparison of particle content and pore-particle ratio of turbine stirrer with those obtained by Ghosh[13] under the same h/H and d/D ratios shows (Fig.4(b)) that turbine stirrer is more effective in helping particle incorporation particularly towards the top although the pore-particle ratio also increased significantly. This is probably an indication of enhanced tendency for the formation of bubble-particle combine when stirred with a turbine stirrer.

Ghosh and Ray[14] in their earlier investigation has established that porosity varies with particle content within a broad band of data points corresponding to processing under a variety of conditions. It has been surmised that during mixing, bubbles are sucked along with particles through vortex created by the stirring. Further, a significant amount of poorly wetting particles gets attached to the bubbles. During solidification in the mould, absence of flow induced by stirring helps these bubble-particle combine to float freely towards the top increasing both particle content and porosity there in the cast ingot. It has therefore, been thought that if stirring is interrupted for an interval of time it may become possible for the bubble-particle combine to float to the top in the crucible itself and the particles may be released on the melt surface for mixing by subsequent stirring. However, the bubbles also form due to gas evolution caused by reduced gas solubility at lower temperatures during solidification. It is also possible that poorly wetting particles dispersed in the molten alloy may get attached to these bubbles because of lowering of interfacial energies. But it is not clear as to when most of the bubble-particle combine forms. Fig.5 shows that interrupted stirring (I/2/

2/2) increases the particle content at the bottom but reduces that at the top. So it appears that the number of bubble-particle combine which contributes to higher particle content at the top, reduces and there are more particles available for settling to the bottom to increase the particle content there. As a result the ingot has more uniform particle distribution along the height than that obtained in case of continuous stirring (C/4) when the total period of stirring has been the same. When the melt-particle slurry is poured in the mould the bubble-particle combine with lower overall density mostly floats to the top and enhances the particle and porosity contents there. A reduction of particle content at the top due to interrupted stirring may indicate lowering in the number of bubble-particle combine as mentioned above resulting from enhanced floatation during cessation of stirring. The particles released at the top are incorporated again by stirring and those particles unattached to bubbles, settle during solidification to enhance the particle content at the bottom. Therefore, the particle content in the ingot becomes more uniform along the height. However, the porosity content at the top reduces but only by a relatively small extent compared to the particles. As a result, the pore-particle ratio at the top increases as shown in Fig.5. Longer processing time at elevated temperatures contributes to higher dissolution of gases in the molten alloy which may enhance the number of pores not attached to particles. Therefore, the extent of porosity does not reduce considerably inspite of flotation of a number of bubble-particle combine. These experiments whose results are reported in Fig.5 have been carried out with d/D = 0.48, h/H = 0.5 and the pouring temperature in the range between 640°C and 670°C.

In order to enhance the effect of cessation of stirring the time for this period has been increased from two minutes to ten minutes and the results of these two sets of experiments denoted respectively by I/2/2/2 and I/2/10/2, have been compared in Fig.6. Increasing the period of cessation of stirring has enhanced the particle content both at the top and at the bottom. The particle content has become more uniform along the height but the percent porosity in the ingot has more than doubled as indicated by the pore-particle ratio in Fig.6 which is possibly due to enhanced dissolution of gases over a longer total processing time at higher temperature even though there was no stirring during the extended period. However, evolution of these gases during solidification did not contribute to the nonuniformity of particle distribution in the ingot and it may be an indirect indication that bubble-particle combine which significantly increases nonuniformity of particle content along the height due to their floatation, forms mostly during mixing by stirrer. The water modeling of mixing of nonwettable particles by stirrer carried out by Ghosh and Ray[7], also indicate that bubble particle combine forms below the stirrer at the center during mixing. Also, the flow induced by stirring possibly enhances the probability of encounter between the bubbles and particles and extent of formation of bubble-particle combine. These experiments whose results are reported in Fig.6, have been carried out with d/D = 0.48, h/H = 0.5 and pouring temperature of about 645°C. However, when the pouring temperature is increased to 700°C for I/2/10/2 it has been observed that particle distribution becomes more nonuniform along the height as shown in Fig.7. Lower viscosity of the melt and the resulting higher Stokes velocity and an increased tendency for flotation of bubble-particle combine and settling of particles during mixing at higher processing temperatures may have contributed significantly to nonuniformity of the ingot both in respect of particle content and pore-particle ratio as shown in Fig.7.

The microstructure at the bottom of the ingots cast out of the continuously stirred slurry (C/4) and the slurry prepared by interrupted stirring (I/2/2/2) are shown respectively in Figs.8(a) and 8(b). A comparison of the microstructures clearly reveal that the bottom of the ingot contains more dispersoid particles when the slurry has been mixed by interrupted stirring as compared to that in continuous stirring. There are also some particles arranged in extended clusters, sometimes in the shape of a circle or coalescence of two circles as shown by the arrows in Fig.8(a) and 8(b) respectively. It appears that the non wetting dispersoid particles get attached to the bubbles and possibly some thin layer of oxides also forms. Some of these bubbles may even coalesce while moving during mixing or solidification. Due to cooling during solidification, the pressure inside the bubbles start reducing and the surface gets punctured by the molten alloy which fills it displacing the gases inside. Since the contact angle between the molten alloy and the particles, often reduces with time [15], the particles have more easily parted with the bubbles. These bubbles so filled, are now heavier due to higher density of particles and oxides, and settle towards the bottom, to the extent allowed by the progress of solidification. The circles and coalescence of circles appear to correspond to planar sections of filled single bubbles and coalescence of two bubbles as shown respectively in Figs.8(a) and (b). The top of the ingot just below the shrinkage pipe, contains more of these clusters in the shape of circle, irregular closed loops or open loop as shown in Fig.9(a) for continuous stirring (C/4) but for interrupted stirring (I/2/2/2) the number of circular clusters are relatively less as shown in Fig. 9(b). Thus the assumption that some of the bubble-particle combine are able to float during cessation of stirring appears to be borne out by microstructural evidence also. There are further clear evidences of coalescence of two to three bubbles as evident from the circular clusters shown by arrows in Fig.9(a). At higher magnification the microstructure in Fig.10(a) clearly shows the dendritically solidified alloy within these clusters. Fig.10(b) shows part of an extended irregular closed loop which may have deformed during flow or during solidification as it is quite likely that solidification of the molten alloy in the bubble may be delayed.

Fig.11 shows that the Brinell hardness number (BHN) measured along the height of a cast composite (I/2/2/2) ingot decreases from the bottom towards the top which may look a little surprising because of enhanced particle content at the top as shown in Fig. 5, but a significant increase in the pore-particle ratio causes this reduction in hardness. A lower pore-particle ratio may show an opposite trend sometimes because the effect of particle content then dominates inspite of the presence of porosities.

Conclusions

The present study has aimed to examine the influence of process variables for mixing melt-particle slurry and a limited variation of stirring schedule on the particle incorporation, porosity and microstructure of the ingot cast from this slurry. The following conclusions emerge:

(i) In the range of experimental variables of the current investigation, a turbine stirrer incorporates higher amount of particles in the ingot cast out of a slurry mixed by it as compared to a flat blade stirrer used by Ghosh and Ray[6] in their earlier investigations. But the particle content at the top increases relatively more possibly due to enhanced number of bubble-particle combine. The pore-particle ratio is also higher in the cast ingot processed by a turbine stirrer.

(ii) In the cast ingots processed by a turbine stirrer the particle content increases when d/D ratio increased from 0.33 to 0.48 but a further increase of d/D to 0.62, did not change the particle content significantly. A reverse trend in pore-particle ratio has been observed and the lowest pore-particle ratio is observed for d/D = 0.62.

(iii) For mixing a slurry of molten alloy with poorly wetting particles it appears that the recommendation of chemical engineers to keep the stirrer at h/H = 0.35 may not work as well from the point of view of particle content in the ingot cast with the slurry. By enhancing h/H to 0.5, the particle content increases significantly and a lower pore-particle ratio is also achieved.

(iv) The microstructural evidence clearly indicates formation of bubbles with particles adhering to it and even their coalescence during processing. Some of these bubbles are eventually invaded by the molten alloy and the particles occur in clusters along the circular boundaries as it has been observed in the microstructure.

(v) When the manner of stirring is changed from a continuous one for four minutes to an interrupted one of two minutes each intervened by a two minute period of cessation of stirring, it has been observed that the particle content at the top reduces but that at the bottom increases making particle content more uniform along the length of the ingot. This has been attributed to enhanced flotation of bubbles containing particles and the mixing of these particles again into the molten alloy.

(vi) During interrupted stirring the porosity did not reduce as much as the particles and the pore-particle ratio at the top increased. Enhanced processing time may have contributed to increased dissolution of gases contributing to porosity and so, a reduced extent of bubble-particle combine did not reduce the overall porosity as much as it was expected.

(vii) When the stirring was interrupted for a longer time of ten minutes, the uniformity of the ingot in respect of particle content improved to an extent but the overall porosity increased significantly. These facts possibly indicate that an enhanced evolution of gas during solidification does not increase the number of bubble-particle combine which contributes to nonuniform particle distribution in the ingot.

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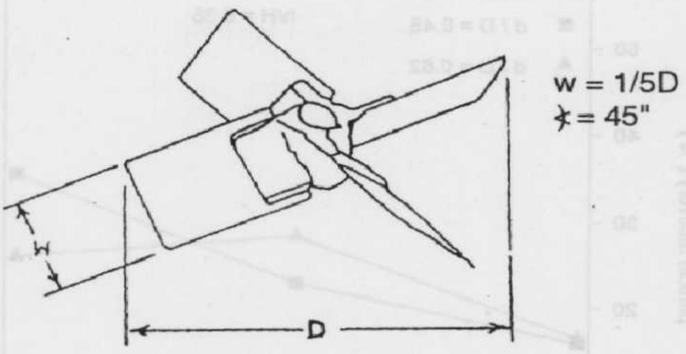


Fig. 1- Schematic diagram showing axial turbine stirrer

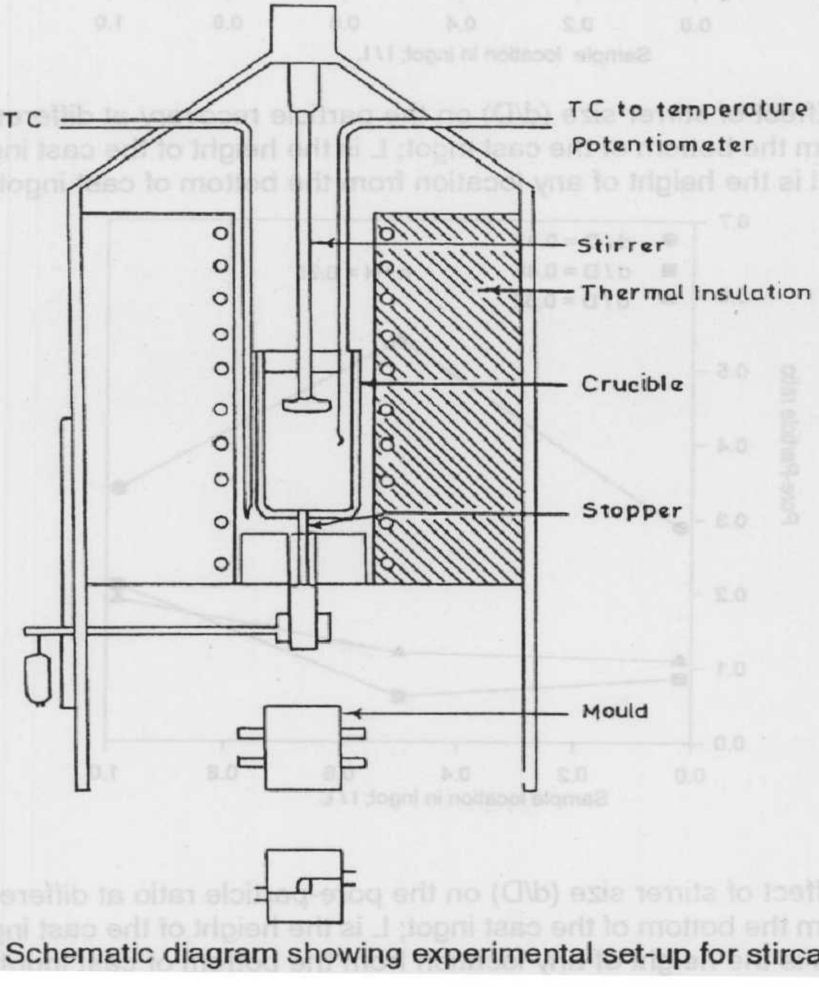


Fig. 2- Schematic diagram showing experimental set-up for stircasting

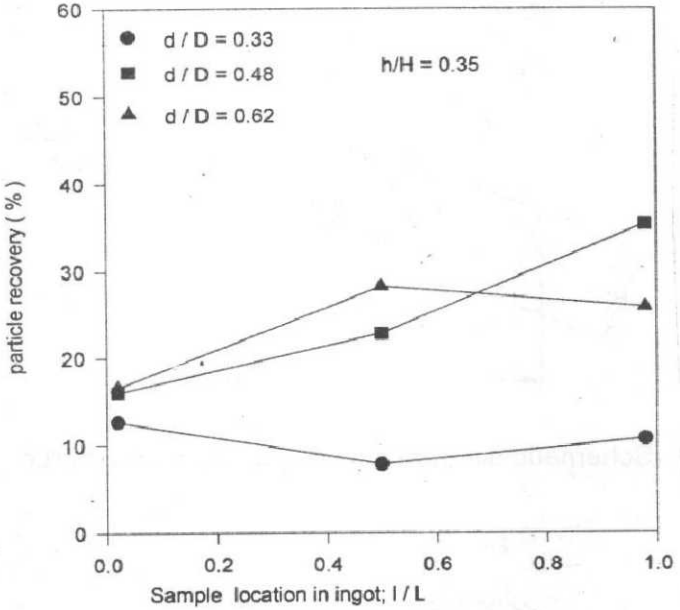


Fig. 3a - Effect of stirrer size (d/D) on the particle recovery at different locations (l/L) from the bottom of the cast ingot; L is the height of the cast ingot and l is the height of any location from the bottom of cast ingot.

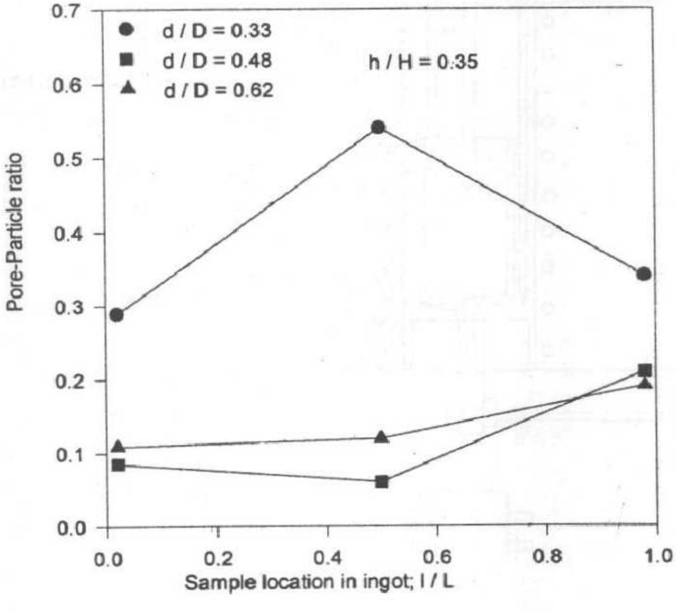


Fig. 3b - Effect of stirrer size (d/D) on the pore-particle ratio at different locations (l/L) from the bottom of the cast ingot; L is the height of the cast ingot and l is the height of any location from the bottom of cast ingot.

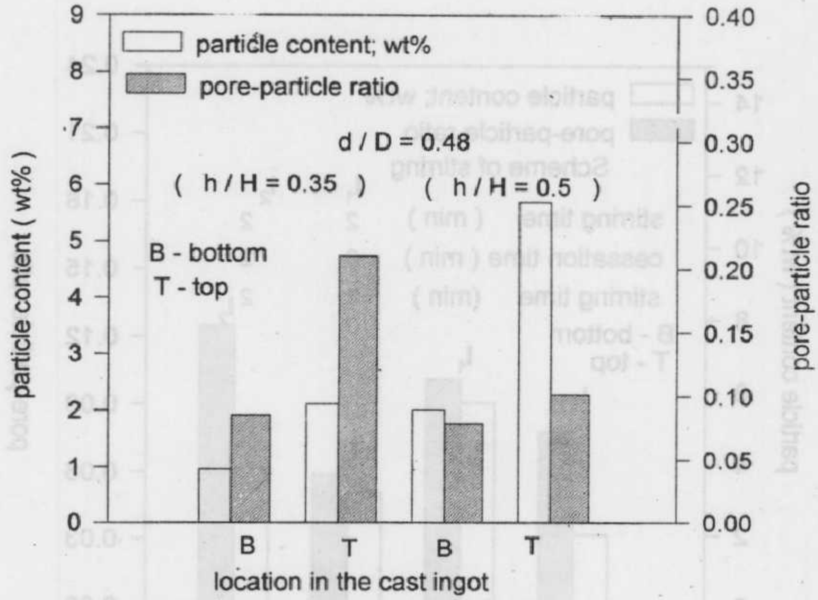


Fig. 4a - Effect of stirrer size (h/H) inside the molten alloy on the particle content (wt%) and the pore-particle ratio at the bottom and the top of the cast ingot; h is the height from the bottom of crucible, at which the stirrer is placed and H is the depth of molten alloy in the crucible.

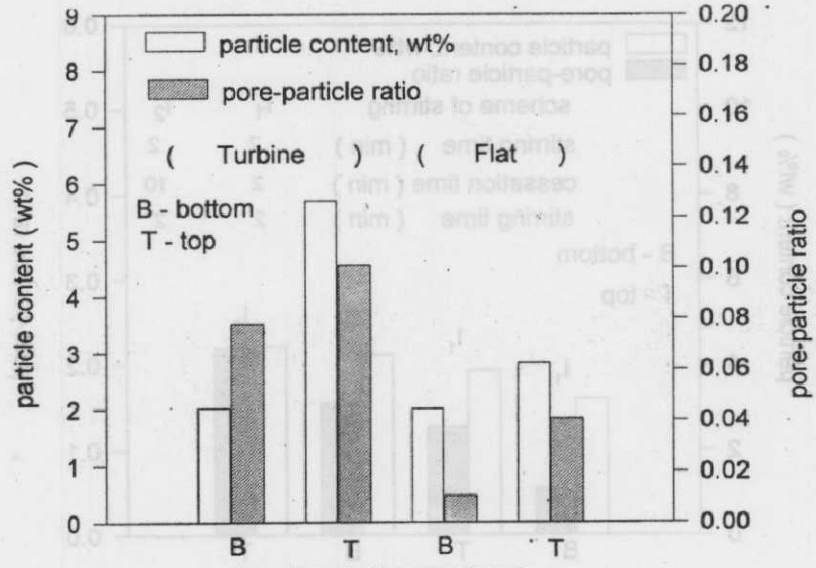


Fig. 4b - Effect of stirrer geometry on the particle content (wt%) and the pore-particle ratio at the bottom and top of the cast ingot.

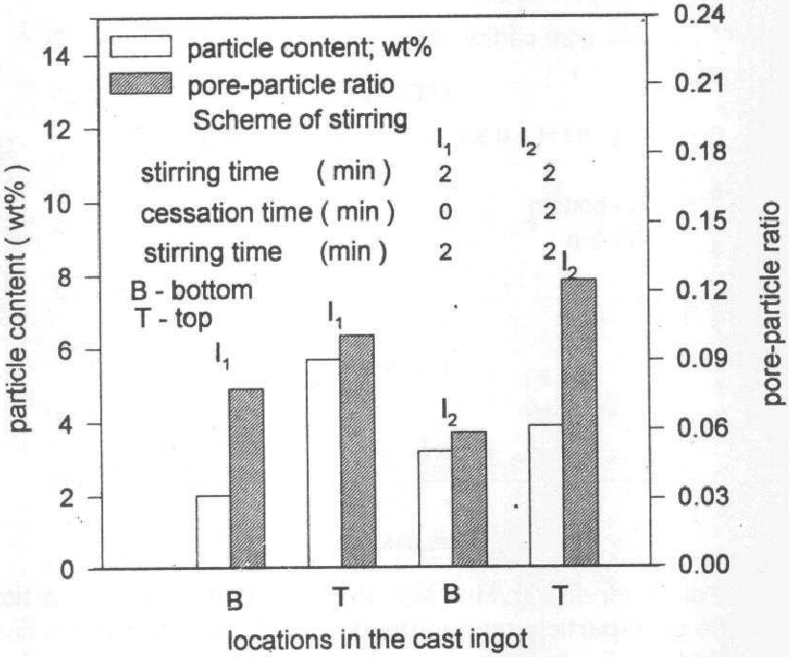


Fig. 5 - Effect of mode of strring on the particle content (wt%) and the pore-particle ratio at the bottom and the top of cast ingot.

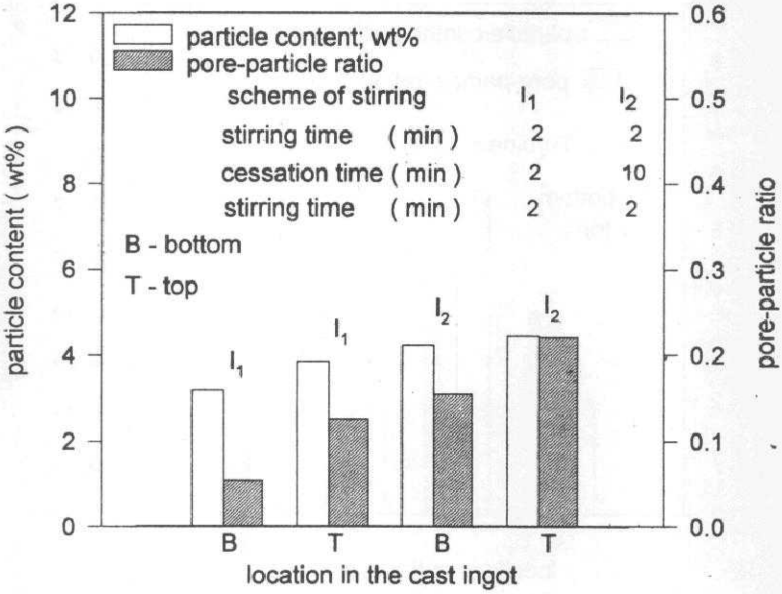


Fig. 6 - Effect of time of cessation of stirring on the particle content (wt%) and the pore-particle ratio at the bottom and top of cast ingot.

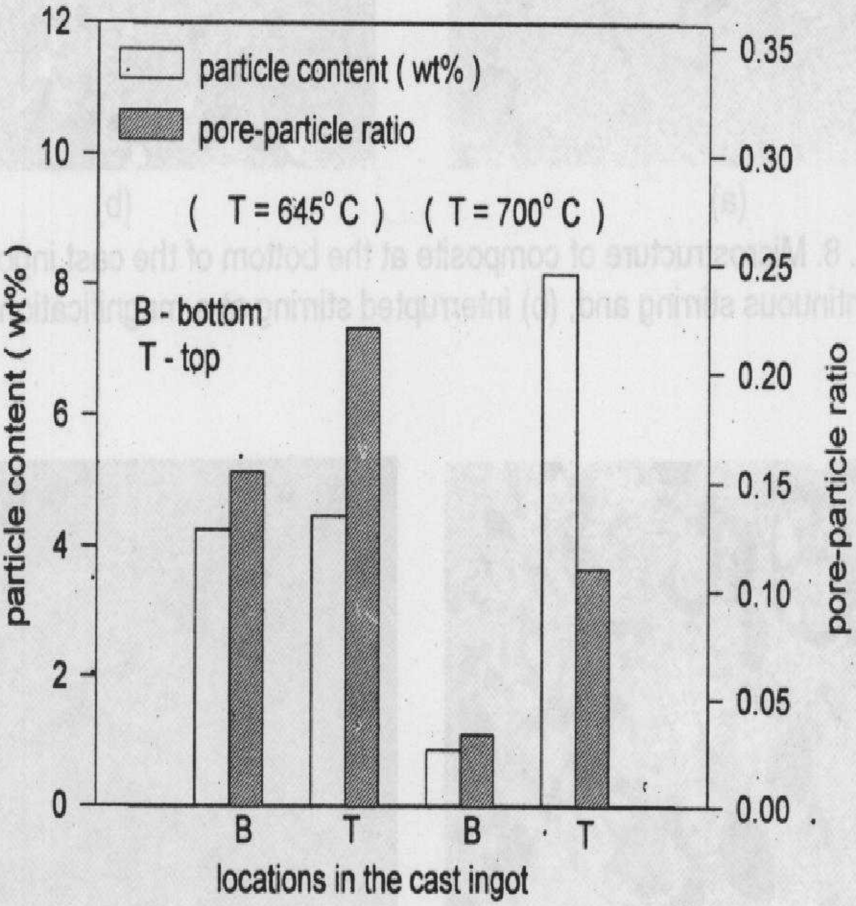
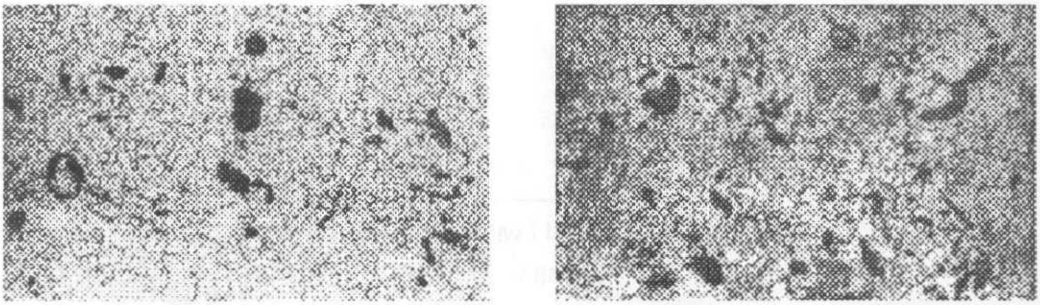
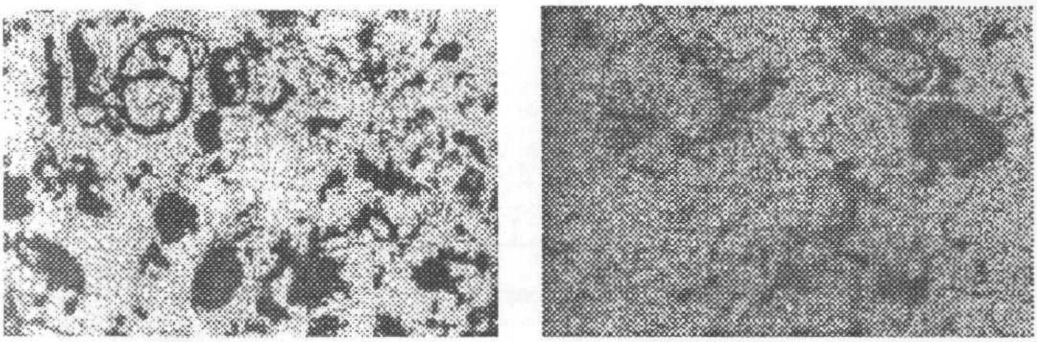


Fig. 7 - Effect of holding temperature on the particle content (wt%) and the pore-particle ratio at the bottom and the top of cast ingot.



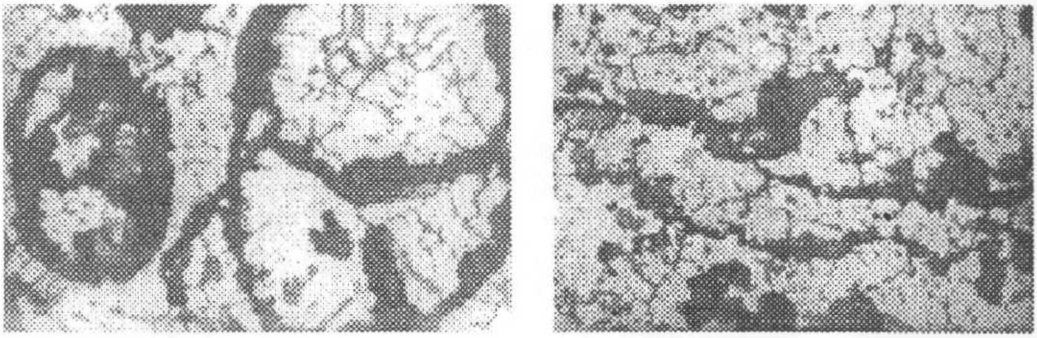
(a) (b)

Fig. 8. Microstructure of composite at the bottom of the cast ingot for (a) continuous stirring and, (b) interrupted stirring at a magnification of X25



(a) (b)

Fig. 9. Microstructure of composite at the top of the cast ingot for (a) continuous stirring and, (b) interrupted stirring at a magnification of X25



(a) (b)

Fig. 10. Microstructure of composite at the top of the cast ingot for (a) continuous stirring and, (b) interrupted stirring at a magnification of X100

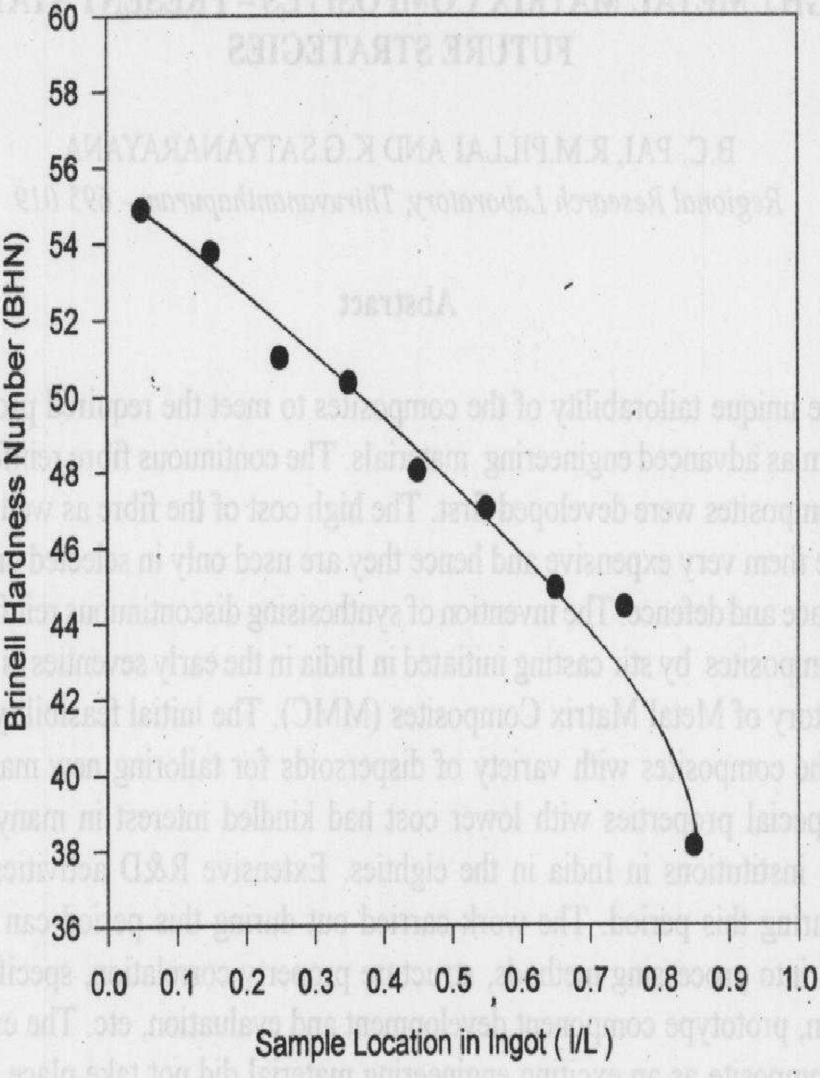


Fig. 11 - Variation of the Brinell hardness number (BHN) with the locations (l/L) along the height of the cast ingot, L is the height of the ingot and l is the height is the given location of the ingot.