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Potential new experiments on fabrication of cast particulate composites in space

P K ROHATGI, M K SURAPPA and M R MADHAVA
Regional Research Laboratory, Trivandrum 695 019, India

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Abstract. Recent developments in fabrication of cast metal ceramic particle composites by liquid metallurgy techniques are outlined. Difficulties encountered in preparing cast composites in the ground environment (including non-uniform distribution and agglomeration of dispersed particles and relatively poor bonding between dispersoids and matrix) and how these can be overcome in a microgravity environment have been discussed. This paper also reviews experiments performed by various space agencies including NASA and ESA on fabrication of composites in space. Some new experiments concerning fabrication of cast composites like dispersion of submicron ceramic particles in molten metals, preparation of cermets with very large volume fractions of ceramic particles and dispersion of flake-type ceramic particles to achieve grain refinement have been proposed.

Keywords. Submicron particle ; flake type particle ; powder metallurgy ; dispersion strengthened and particulate composites ; wetting ; liquid metallurgy technique ; space environment ; grain refinement.

1. Introduction

There are three classes of composite materials as distinguished by their microstructures. In each class an elemental or alloy matrix has a second phase distributed in it.

(i) Dispersion strengthened composites

($r = 0{\cdot}01$ to $0{\cdot}1\,\mu$m; V_f 1 to 15%),

(ii) Particulate composites

($r = 1$ to $200\,\mu$m ; V_f 25 to 85%) and

(iii) Fibre reinforced composites

($L/d > 50$; V_f 40 to 70%).

Among the particulate composites there are metal-matrix ceramic particle composites such as metal-graphite, metal-oxide and metal-mica (named as METCERPS) with low volume fraction dispersoids (< 10%). These composites possess good properties like low coefficient of friction, improved seizure and wear resistances (adhesive and abrasive wear), improved machinability and high damping capacity.

However, dispersion-hardened composites with submicron particle dispersions are known mainly for their superior creep properties at high temperatures.

So far, these metal-ceramic composites (particle and dispersion-hardened) were generally made by powder metallurgical methods. Recently attempts have been made to produce these composites by direct casting route. This technique has many advantages compared to powder metallurgy methods.

In this paper we summarise (a) work on the preparation of cast METCERPS composites and their properties; (b) some of the difficulties encountered in preparing these cast composites in the ground; (c) work done so far by various space agencies to fabricate particle and dispersion-hardened composites in outer space. Some new experiments on fabrication of cast composites that could be performed in outer space have also been listed.

1.1. *Metal-ceramic particle composites by liquid metallurgy techniques*

Liquid metallurgy technique consists of introduction and dispersion of ceramic particles in molten metal/alloys and solidifying the resultant composite melts in suitable moulds. Introduction of ceramic particles could be accomplished either by (a) mechanical stirring (Badia 1971), (b) gas injection process (Badia and Rohatgi 1969a), (c) pellet technique (Pai and Rohatgi 1978) or (d) injecting powders into a stream of molten metal entering the mold (Rohatgi 1969).

However, generally most ceramic particles are not wetted by liquid metals and under such circumstances, even if the particles are introduced by some means they have a tendency to get separated from the melt. One approach to overcome the wetting problem was to coat the ceramic particles with metal coatings like copper (Pai and Rohatgi 1975) and nickel (Badia and Rohatgi 1969a) before introducing the particles in the melts. Another approach to improve the wetting is to add anions of refractory particles (Herald and Scruggs 1969) to the melt or/and an element like magnesium (Pai *et al* 1976; Gorbunov *et al* 1974) to the melt which will react with the ceramic particles or to add uncoated and untreated ceramic particles to a vigorously agitated, partially solidified slurry of the alloy (Flemings *et al* 1974).

Recently a more simple and direct method in which uncoated but suitably-treated ceramic particles are introduced into alloy melts has been developed (Surappa 1979; Krishnan 1980). Using this process, ceramic particles such as graphite (Krishnan 1980), alumina (Surappa 1979), mica (Deonath *et al* 1980), silica (Rohatgi *et al* 1979), silicon carbide (Surappa 1979), clay (Surappa 1979), shell char (Murali *et al* 1979) and zircon (Banerjee *et al* 1979) particles have been dispersed in molten aluminium and its alloys and corresponding cast aluminium composites have been made. Aluminium and its alloys have been used as matrix material in most of our work. However, some work has also been done with other matrix materials like magnesium (Rohatgi 1969), copper (Suwa *et al* 1977), zinc (Badia 1971), steel (Hasegawa and Takeshia 1978) and tin (Sartor 1974).

1.2. *Properties of metal-ceramic particle composites*

The physical, mechanical and tribological properties of cast metal-ceramic particle composites made on the ground at 1 g have been studied in detail. It has been reported that graphite particle additions to aluminium and its alloys lead to improved damping capacity and machinability (Rohatgi *et al* 1976). Addition of a minimum of 1·8% graphite to Al–12Si alloy enables its bearings to run under boundary lubrication without galling (Badia and Rohatgi 1969b) and results in decreased wear (Pai *et al* 1974). A 5% reduction in specific fuel consumption has been reported (Krishnan *et al* 1979) using aluminium-graphite pistons in IC engines. A similar reduction in fuel consumption in IC engines using cylinder liners of aluminium graphite has been reported (Bruni and Ignera 1978).

2. Preparation of cast composites on ground

Despite the successes achieved in preparing a variety of cast composites by liquid metallurgy techniques several difficulties, some of which are listed below, need to be overcome.

(i) Introducing the as-received ceramic particles into molten alloys without resorting to ultrasonics, additions of surface-active elements, metal coatings or pre-heat treatment of particles and/or agitation of the bath.
(ii) Introducing submicron particles to produce dispersion-strengthened composites.
(iii) Eliminating or minimising gravity-induced floatation or sedimentation of dispersoids especially in slowly cooled castings.
(iv) Obtaining perfectly homogeneous distribution of ceramic particles on macro and microscale in castings.
(v) Getting grain refinement due to the dispersion of these particles.
(vi) Getting the particles entrapped by the freezing interfaces (this results in the particles being present only in the inter-dendrite regions than within the dendrites).
(vii) Obtaining good bonding between the particles and the matrix.

2.1. *Space environment in cast composites*

The low gravity (10^{-4} to 10^{-5} g) and high vacuum conditions prevailing in space would help overcome many of the difficulties described earlier since the absence of gravity would lead to marked reduction in (a) convection phenomena and associated thermal fluctuations, and (b) flotation or sedimentation rates of second phase particles. Due to considerable reduction in (a) and (b) a stable and homogeneous mixture of liquid metal and ceramic particles can be obtained. Hence, the distribution of second phase particles in composites (particle and dispersion hardened) prepared in space is expected to be uniform. Surface characteristics of some of the ceramic particles could be different in space, especially if particles are exposed to elevated temperatures in ultrahigh vacuum of space for long periods. This could bring about a significant improvement in wetting between molten metals and refractory particles and this would help in (a) strong cohesion between particles and the matrix; (b) grain refinement due to presence of dispersed particles; (c) preventing the formation of aggregates and agglomerates; (d) improved physical and mechanical properties.

3. Fabrication of particle and dispersion-hardened composites in space

Experiments to prepare composites in space have been conducted by many countries and the results appear meaningful. However, in certain cases the results were unexpected and inconclusive, perhaps due to the improper and inadequate design of experiments.

3.1. *Preparation of Mg–ThO_2 composites by casting*

These experiments were conducted on sounding rockets (SPAR) during a free fall in SPAR I and II flights (Ang and Raymond 1977).

The SPAR I experiment consisted of heating the composite sample containing 2·4 % vol ThO_2 particles (0·8 μm) in a matrix of Mg (75 μm) which was already made by PM route on the ground. The sample was held for 190 sec at a temperature above 800° C and allowed to cool slowly. As compared with ingots made on earth, it was found that the sample processed in space was relatively more sound and devoid of gas voids and shrinkage cavities. However, even under space conditions, the distribution of ThO_2 particles was not uniform and there were regions which were free from ThO_2 particles.

In SPAR II experiment the production of ThO_2 dispersions were based on the following reaction:

$$(2\,MgO)_S + [Th]_L = (ThO_2)_S + [2Mg]_L.$$

Composite sample consisting of 87·6 wt% Mg (75 μm), 9·2 wt% Th (0·8 μm) and 3·2 wt% MgO (0·8 μm) was made by hot pressing at 250° C at earth gravity. In space the sample was heated to temperature of 800–950° C and maintained for 180 sec followed by rapid quenching. Both the castings made in space and on the ground had little porosity and contained dispersion of ThO_2 particles. The size of ThO_2 particles in both samples was the same (0·6 μm) indicating the absence of agglomeration. However, the distribution of ThO_2 in the space-processed sample was more uniform and the Brinell microhardness of the sample was 31 ± 3 whereas that processed in space was only 21 ± 7.

3.2. *Experiments on processing of Be–BeO*

This is one of the most carefully planned experiments and the results are conclusive (Wouch *et al* 1978), although the objective of grain refinement was not achieved. These experiments were performed in SPAR III flight to prepare cast beryllium having very fine grain structure and improved properties. The grain refiner tried was BeO. Beryllium alloy containing 1·5 wt BeO was melted using NASA electromagnetic containerless processing payload (ECPP) and re-solidified. The specimen of diameter 0·922 cm was made by powder metallurgy (PM) method hot isostatic pressing (HIP). Another specimen from the same billet was melted and resolidified at earth gravity for comparison. More agglomeration and segregation of BeO particles was found in the specimen prepared on the ground, whereas the distribution of oxide particles was much more uniform in the specimen made in space. However, despite the uniform despersion of BeO particles in the space-processed samples the cast structure of beryllium matrix was not refined. One of the reasons for agglomeration of BeO particles in ground-processed specimen is poor wettability between BeO and molten Be. Results indicate that microgravity environment does not totally eliminate agglomeration but only lengthens the time for the onset of agglomeration. The agglomeration time in space is lengthened due to reduction in particle collisions, velocity gradient in the melts and reduced velocity of Stokes flotation. Other factors like poor wettability, surface tension driven convection and electromagnetic stirring caused agglomerates. Absence of grain refinement in the casting despite good dispersion of BeO has been attributed to the spheroidal shape of BeO particles, since normal grain refining agents are generally angular or flake type.

3·3. *Ag–SiC composites*

These experiments were performed on *Skylab* (Takahiashi 1977) to obtain whisker-strengthened composites by dispersing SiC whiskers in silver. Results show that the dispersion of SiC whiskers was more uniform in space-processed sample which showed more uniform hardness, greater ductility and higher strength compared to the specimen prepared at 1 g on ground.

3.4. *Dispersion of non-wettable particles in InBi alloy*

Attempts were made during Apollo-14 flight to incorporate nonwettable particles like tungsten in InBi alloy melts. Results indicate (Yates 1972) that on the ground the tungsten particles were rejected by the melt but it was not so in space.

3.5. *Al-Pb bearing alloy (TEXUS-1, Dec. 1977)*

This experiment was designed to obtain cast Al-Pb alloy containing uniform and fine dispersions of lead particles in the matrix of aluminium (Hodes and Steg 1978). Such composites are difficult to process on ground due to their large density differences. Initially Al-8%, Pb-3·5%, Si-1·5%, Cu-1% Sn alloy was prepared on the ground by PM. The specimen was heated to 1100° C in space and allowed to re-solidify. The dispersion characteristics of Pb in Al were the same both in flight and ground specimens. This has been attributed to the presence of oxide layers on Pb particles in both specimens preventing fragmentation of lead droplets in the space environment.

4. Potential experiments on composites in space

In all the previous experiments performed in space, the starting material was a composite prepared by powder metallurgy method on the ground. In the case of a composite so prepared and remelted in space there may not be much difference in the surface characteristics of dispersed phases in PM composite, and in the composite processed in space, since the dispersed particles were not exposed to high vacuum environment prevailing in space. The formation of cast composites will be much easier if the dispersoids are exposed to elevated temperature in space vacuum.

4.1. *Dispersion of graphite, coconut shell char and zircon particles in molten aluminium*

Attempts should be made to disperse 3 wt % graphite, γ-Al_2O_3, coconut shell char and zircon particles (100 μm size) in molten aluminium to prepare cast composites through liquid metallurgy route in space. A suitable quantity of aluminium should be melted and its temperature raised to 720° C. The required quantity of graphite or Al_2O_3 or shell char or Zr SiO_4 particles (100 μm), previously heated to 900° C for 2 hr in space, should be added to the surface of molten aluminium. If necessary a mechanical stirrer could be employed to aid dispersion. Molten Al containing suspended particles could be allowed to solidify in moulds identical to those used in the ground. It would be interesting to examine the distribution of these particles especially to measure the interparticle distance in resulting composites, and compare the results with composites processed at 1 g. It would be instructive to examine the fracture surfaces of space-processed composites to determine if improved bonding is obtained between the metal matrix and ceramic particles.

4.2. *Dispersion strengthened cast Al–Al_2O_3 and cast Al–graphite composites*

Experiments mentioned earlier (§ 3.1) could be repeated with 0·01 μm size Al_2O_3 and graphite. It would be interesting to see whether the initial submicron sizes of dispersed particles is retained in the castings and if these particles lead to dispersion strengthening.

4.3. *Cast cermets*

Experiments mentioned in § 3.1 should be repeated with 60–98 vol % graphite, zircon, Al_2O_3 particle of 100 μm size to prepare cermets containing very high volume percentage of ceramic phase. It has not been possible to prepare these composites at 1 g by the casting technique.

4.4. *Dispersion of submicron size flake shaped a-Al_2O_3*

This experiment is conducted to obtain dispersion hardening and grain refinement in aluminium by dispersing flake-shaped and submicron size a-Al_2O_3 (and possibly other oxides) particles in molten aluminium and casting the melts. a-Al_2O_3 could act as grain refiner since it has the same crystal structure as aluminium (cubic) and there is a hypothesis that grain refining efficacy of flake-shaped particles is higher compared to granular particles.

4.5. *Interaction between moving solid-liquid interface and dispersed particle during solidification*

Previous experiments on this could be repeated with a slight modification. In the modified set-up the interaction between moving solid-liquid interface and single particle should be studied instead of multiparticle system. This would eliminate the effects due to the crowding of particles. Experiments should also be conducted in space on the unidirectional solidification of cast Al-1% graphite (40 μm) particle composite to study the interaction of moving solid-liquid interface, and dispersed graphite particles. On earth the major difficulty in conducting such experiment is flotation of graphite particles due to buoyancy effect.

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