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(11) Publication number:

0 346 771 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: **26.10.94** (51) Int. Cl.⁵: **C22C 1/10**

(21) Application number: **89110482.0**

(22) Date of filing: **09.06.89**

(54) **Method for making solid composite material particularly metal matrix with ceramic dispersates.**

(30) Priority: **17.06.88 US 208086**
17.06.88 US 208084

(43) Date of publication of application:
20.12.89 Bulletin 89/51

(45) Publication of the grant of the patent:
26.10.94 Bulletin 94/43

(84) Designated Contracting States:
AT BE CH DE ES FR GB IT LI NL SE

(56) References cited:
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Description

This invention relates to methods for making solid composite materials, preferably metal-matrix composites (MMC), which comprise a continuous metal matrix material having a plurality of separate particles, i.e. dispersates, dispersed throughout the matrix and to methods for making such composites having an extremely uniform dispersion of the dispersates and simultaneously having essentially no porosity. The invention is particularly directed to metal-matrix composites having ceramic dispersates substantially uniformly distributed throughout the matrix phase. The present invention provides a solution to the problems of excessive porosity in the MMC's and excessive clustering of the particulates, which problems have prevented as-cast composites from having properties comparable to powder metallurgically forged or extruded composites.

It is well known that composite materials often have better mechanical properties than either the matrix or the dispersates alone. The extent of the improvement in properties has generally been found to be a function of the degree of distribution of the dispersates. However, making composites with reproducible and extremely uniform dispersions of dispersates has proved extremely difficult.

One method of making composites in which a matrix material is obtained from a fluid state is to disperse dispersates in a precursor liquid and then form the composite by solidifying the liquid part, i.e. dispersion medium, of the dispersion. In practice, however, it has been quite difficult to perform due to density differences which normally are present between the dispersates and the dispersion medium. These differences generally lead to a non-uniform distribution of the dispersates in not only the liquid dispersion, but also the final composite.

U.S. Pat. No. 4,735,656 suggests overcoming the density segregation problem by (i) mixing metal particulates with ceramic particulates and then (ii) heating the mixture to a temperature high enough to cause partial melting of the metal (so that it fuses into a dense matrix when cooled) but not so high as to cause the ceramic particulates to float therein. The care required to utilize this process makes the process undesirable for large scale commercial operations. Also the process does not inherently preclude the presence of voids in the final product.

Another problem with using the simple mixing concept is that many of the most desirable dispersates are difficult to wet by known fluid precursors of desirable matrix phases. Int. Pat. Appln. WO 87/06624 teaches the use of specific dispersing and/or sweeping impellers to promote high shear

mixing while minimizing the introduction of gases into the mixture as well as the retention of the gases at the interfaces of the dispersates and the matrix material. U.S. Pat. No. 4,662,429 teaches the addition of lithium to an aluminum matrix alloy melt to facilitate wetting and dispersing of the dispersate in the matrix alloy. EP-A-0 256 600 describes composites of a zinc-aluminum alloy reinforced with silicon carbide powder which "surprisingly" has good mechanical properties without the difficulties often experienced with other similar composites. EP-A-0 104 682 discloses a method for distributing insoluble material in a liquid or partially liquid metal by providing a combination of a first metal and insoluble particles and introducing the combination into a second metal above the solidus temperature of both metals.

The difficulties in making good quality composites, and some of the methods attempted for dealing with the problems are generally reviewed in "Solidification, Structures, and Properties of Cast Metal-Ceramic Particle Composites," P.K. Rohatgi et al., 31 *International Metals Reviews* 115-39 (1986). One method, described in more detail by B. C. Pai et al., 13 *Journal of Materials Science* 329-35 (1978), involves pressing together dispersates with powdered matrix material to form a pellet, introducing the pellet with stirring below the surface of the fluid matrix precursor material for a sufficient time both to melt the pellet and to disperse the dispersates within the total amount of fluid precursor material, and then solidifying the dispersion. Analogously, J. Cisse et al. in 68 *Metallurgical Transactions* 195-97 (1975) describe the use of a "master alloy" of sintered aluminum powder rods which contain 10 w/o aluminum oxide.

A. Mortensen et al., *Journal of Metals*, February 1988, pp. 12-19, also reviews the field and refers to Rohatgi et al. as listing a number of techniques for introducing particulates, including pre-infiltrating a packed bed of particulates to form a pellet or "master alloy" and redispersing and diluting it into a melt of a matrix material or its precursor.

All of the prior art methods initially produce composites which contain substantial porosity unless the dispersates are easily wet by the fluid matrix material. And if the dispersates are easily wet, the subsequent properties of the composite are often degraded as a result of chemical reactions which occur between the matrix material and the dispersates.

It is therefore an object of this invention to produce composites containing as little porosity as possible. It is a further object to minimize the time required to make the composites so that chemical degradation of the dispersates by the final matrix material or a precursor thereto in the composite is

precluded or at least substantially minimized.

SUMMARY OF THE INVENTION

Composites having the most desirable properties for many purposes are produced when the dispersates therein are sufficiently widely dispersed that most of them do not touch another dispersate particle. This class of composites is characterized herein as having "discrete" dispersates or as a "discrete dispersion." It has now been found that many of the difficulties in the prior art of making discrete composites can be overcome by using an indirect method of preparation which entails (i) making a concentrated dispersion of dispersates and a precursor to the final matrix in which there is intimate contact between the precursor matrix material and the dispersates and then (ii) dissolving the concentrated dispersate dispersion in additional matrix precursor material.

Thus in the present invention a concentrated dispersion is used to form the more dilute dispersion of the desired composition by mixing it with additional matrix fluid precursor and dispersing it therein. If the mixing is done while the dispersion medium of the concentrated dispersion is still fluid, this embodiment is referred to as a "continuous" method. Sometimes, however, it may be more convenient to solidify the dispersion medium of the concentrated dispersion producing a "concentrated composite" before beginning the mixing step to prepare the finally desired composite. Composites made in this way are denoted as being made by the "concentrated composite" embodiment of the invention.

In accordance with the present invention there is herein provided a process for manufacturing a final composite product as defined in claim 1. Further embodiments are defined in claims 2 to 15.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a cross-section view of one type of apparatus useful in the practice of the instant invention. Figures 2-3 and 6-7 are cross sections of composites produced according to the invention or of comparison composites. Figures 4 and 5 are cross-section views of another type of apparatus useful for the practice of this invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

It has been found that satisfactory concentrated dispersions according to this invention are made by packing dispersates into a porous bed in which most dispersates touch at least one other dispersate, and then infiltrating the packed bed with a

fluid precursor to the desired final matrix such that (i) the reasonably uniform distribution of dispersates characteristic of the packed bed is maintained during the infiltration and (ii) most, if not all, of the gas existing in the interparticle volume of the bed is displaced during the infiltration step. In this way, the infiltrated packed dispersate bed becomes a concentrated dispersion of dispersates useful in this invention. Preferably the concentrated dispersion will have no more than about five volume per cent voids and/or gases. Still more preferably, porosity is substantially entirely eliminated from the concentrated composite. Dispersions having these characteristics are made having higher concentrations of dispersates than is normally desired in the final product.

If the packed porous bed of dispersates is evacuated before infiltration, the fluid infiltration into the bed may be accomplished from multiple directions, if desired. Often it is more convenient to omit the evacuation step and infiltrate the dispersate bed only from a single direction while permitting the displaced air to escape through an open part of the bed. When the fluid precursor does not spontaneously wet the dispersates, infiltration may still be successfully achieved by applying a pressure to the fluid. Although the infiltration process generally does separate some of the interparticle contacts between the packed dispersates, the resultant dispersion is more concentrated than the final dispersion made therefrom.

The infiltration of the packed porous bed of dispersates is usually performed under minimum suitable conditions of both temperature and pressure. By using a low temperature, any reaction between the dispersate powder and the matrix precursor will be minimized. Thus the temperature of the matrix precursor fluid used should be about 25 to about 200, preferably about 50 to about 175, and most preferably about 75 to about 125 °C. above its melting point. Preferably the dispersates are preheated to essentially the temperature of the matrix precursor fluid to prevent metal cooling or freezing off which otherwise could occur and reduce or prevent the complete infiltration. With regard to pressure, the use of pressure has been found to be advantageous to reduce/eliminate porosity in the concentrated dispersion but simultaneously increase the cost of performing the infiltration. Thus the pressure used is chosen to balance the level of porosity of the concentrated dispersion and the cost. Generally, an overpressure of at least 1.72 bar (25 psi) will be preferred with still higher overpressures being more preferred as the particle size of the dispersates is reduced. Also to help minimize any porosity due to too low a pressure having been used, it has been found to be desirable to increase the overpressure to about 13.79 to

20.68 bar (200 to 300 psi) near the end of the infiltration process.

In order to promote good displacement of inter-particle gases at relatively low infiltration pressures during the formation of the composites, it has been found beneficial to incorporate known wetting agents to the matrix precursor materials used to form the concentrated dispersion. The specific wetting agents and amounts thereof utilized will normally depend upon the specific matrix material and dispersate and can be determined by routine experimentation. For instance, when preparing a composite of silicon carbide and an aluminum alloy, tin and potassium hexafluorozirconate are preferred wetting agents since they are known to promote the wetting of silicon carbide and can be readily added to the aluminum alloy precursor material.

The mixing of the concentrated dispersion with additional precursor fluid is then accomplished in a way that avoids the difficulties with dispersing small particles directly in an open container of fluid, as discussed above. With the favorable concentrated dispersions of this invention, mixing becomes very easy. Portions of the concentrated dispersion can simply be placed atop a second matrix fluid, if the dispersates are denser than the matrix, or covered with the second matrix fluid, if the dispersates are less dense than the matrix. A combination of gravity and stirring then mixes the dispersates into the total amount of fluid matrix precursor. It has been found that concentrated dispersions made by the methods described herein often have the very favorable property that, at some temperatures, they behave as if the dispersates were so well bonded to the matrix that each dispersate tends to carry a substantial amount of matrix material with it when moved.

Preferably a non-vortex generating mixer will be used to minimize any air entrapment. The mixer must obviously be made of a material which will not react with or degrade the matrix precursor fluid. Suitable such materials will depend upon the particular composition of the composites. For example, with an aluminum alloy matrix, mixers prepared from graphite or steel coated with a nonwetting wash or spray such as carbon, sodium chloride, or mica wash are preferred. In some alloys, Contronics 902 machinable ceramic from Contronics Co., Brooklyn, NY, may be used.

High shear mixing is preferred to disperse any small clusters or agglomerates of dispersates remaining from the concentrated dispersion and also to provide a more uniform distribution of the dispersates in the final composite. During mixing, the temperature is normally maintained within the range at which the mixtures containing the dispersates exhibit thixotropy so that efficient mixing is achieved while reducing the possibility of disper-

sate reseggregation due to density differences as the mixed material moves away from the mixing zone.

Certain procedures for accomplishing the mixing step have been found either convenient or preferred. In performing the continuous embodiments of the invention, as shown in Examples 9 and 10 below, it is often convenient to provide a pressurizable reservoir of the concentrated dispersion from which it is injected into at least a portion of the second precursor fluid. To facilitate mixing, it is generally preferred to inject the concentrated dispersion into a flowing stream of the second precursor fluid. For the concentrated composite embodiment, for which Example 1 below describes a suitable apparatus, a portion of the concentrated composite may be held mechanically below the surface of a body of second precursor fluid, maintained at a temperature high enough to reliquify at least part of the matrix of the concentrated composite, and portions of the two components of the concentrated composite can be mixed into the second precursor fluid as liquefaction occurs. Alternatively, the concentrated composite can be heated to, and held at, a temperature sufficient for partial liquefaction of its matrix, and additional fluid precursor added with mixing.

The invention provides the first discrete dispersions that are (i) substantially free of pores and (ii) have an essentially uniform dispersion of the dispersates. In particular, new dispersions and also not more than about 40 volume percent of dispersates and also not more than about 5 volume percent voids, pores and gases are provided herein.

In connection with this invention, it should be understood that a "precursor of a matrix material" refers to any material that can be converted into the desired matrix material by chemical or physical treatment without dislocation of any dispersates contained therein. Thus, a liquid alloy or a thermoplastic resin is a precursor to the solid alloy or resin into which it hardens on cooling; or fluid mixtures of polyfunctional isocyanates and polyfunctional alcohols are precursors to the polyurethanes that they form by chemical reaction after mixing; or fluid acrylated materials are precursors of the polymer that they form after exposure to an electron beam. "Matrix" is used herein to refer to the continuous phase of any dispersion or composite, be it in a fluid or a solid state.

The solid composite materials of the present invention are comprised of two primary materials - the matrix and the dispersates. Matrix materials useful in the present invention include metals, metal alloys, and thermoplastic resins. Suitable metals and metal alloys include aluminum, aluminum alloys, magnesium, magnesium alloys, bronze, cop-

per, copper alloys, zinc, and zinc alloys. Suitable thermoplastic resins include polyester polyurethanes, polyether polyurethanes, and acrylic polymers and copolymers. Preferably the matrix material is selected from aluminum, aluminum alloys, magnesium, magnesium alloys, and magnesium-aluminum alloys. Most preferably the matrix material is an aluminum alloy containing less than about 12 w/o, preferably less than about 8 w/o, and most preferably less than about 1.5 w/o silicon.

Suitable dispersates for use herein are ceramic materials such as silicon carbide, silicon nitride, aluminum nitride, alumina, titania, silica, boron carbide, borides, carbides, silicides, diamond and the like. These materials are characterized as having a modulus, strength and wear resistance which are substantially higher than that of the matrix materials. Preferably the dispersates are either silicon carbide, silicon nitride, aluminum nitride, or aluminum oxide. Most preferably the dispersates are silicon carbide. The dispersates, irrespective of the particular chemical composition, are used in the form of fine particles, generally having an average particle size of about 0.1 to about 45 micrometers, preferably about 3 to about 20 micrometers, and most preferably about 7 to about 15 micrometers.

The concentrated dispersion will generally be prepared to contain as much dispersate as possible. Thus, about 25 to about 85 w/o, preferably about 45 to about 60 w/o, and most preferably about 53 to about 56 w/o dispersates will be used.

Some of the most useful applications of this invention are in the manufacture of composites of silicon carbide dispersed in aluminum or magnesium alloys. Such materials are valuable construction materials for applications such as airplane bodies and other components in which a combination of low density, high toughness, and high flexure resistance at temperatures not too far below the melting point of the alloy are needed. Previously it had been very difficult to make composite products having about 20 w/o silicon carbide, the most mechanically desirable range, with the substantially uniform dispersion of silicon carbide particles that is needed. This was particularly true when the silicon carbide particles were mostly less than ten micrometers in size and/or had a wide distribution of sizes. Such composites can be readily produced with the present invention.

Alloys of aluminum containing from about 1 to 4% silicon are known to make stronger composites when reinforced with ceramic materials such as silicon carbide than do aluminum alloys containing less silicon. This is so even though unreinforced silicon-aluminum alloys containing less than 1% silicon are stronger than those containing more than 1% silicon. The difficulty of making composites with low-silicon aluminum alloys and silicon

carbide is believed caused by a reaction between the low-silicon aluminum alloy and the silicon carbide dispersates which produces aluminum carbides. The aluminum carbide formation weakens the matrix/particulate interface and makes SiC a less effective reinforcement material.

Similarly, when silica dispersates are used with a low-silicon aluminum alloy they can react with the aluminum to form silicon and alumina. And when titania dispersates are utilized to reinforce low-silicon alloys, they can react to form Ti_2Al and alumina. Similarly, other deleterious reactions can occur with various ceramic and metal combinations, as is known.

The difficulties caused by such deleterious interactions between desirable dispersates and either the precursor to the matrix material or the matrix material itself, such as occur when silicon carbide dispersates are used to reinforce a low-silicon aluminum, can be overcome with an embodiment of the present invention. The deleterious reaction is inhibited sufficiently, or ideally prevented, by coating the dispersate particles with a material that (i) will not react with either the precursor or the matrix material, but (ii) will produce an adherent coating on the dispersates, and (iii) will promote wetting of the dispersates by the matrix precursor.

Suitability of particular dispersate coating materials can be determined by routine experimentation by coating particular dispersates, then attempting to infiltrate a packed porous bed thereof with the desired matrix precursor fluid, and if the infiltration succeeds then attempting to disperse the concentrated dispersion into additional matrix precursor material. Examples of suitable coatings for use with low-silicon aluminum alloys include metals, metal oxides, metal nitrides, metal carbides and metal borides. When a metal coating such as copper, molybdenum, nickel, zinc, tin, or titanium is used, it is preferably extremely thin, i.e. up to about 2 micrometers, to minimize any detrimental intermetallic interactions. Metal oxides useful herein include such as silica, alumina, chromia, nickel oxide, copper oxide, mullite, spinels, titania, magnesium silicate, lithium silicate, and the like. Metal nitrides useful herein include silicon nitride, titanium nitride, boron nitride, and aluminum nitride. Metal silicon compounds useful herein include molybdenum, copper and titanium silicides.

Silicon dioxide, which can be formed on silicon carbide by heating in air, is currently preferred, partially because it is particularly convenient to produce by merely heating silicon carbide powder at about 1300°C. for about 30 minutes. Alumina, which can conveniently be coated onto silicon carbide particles from a seeded sol to form the coating is also preferred.

Such coatings have generally not been found necessary for final composites prepared with magnesium-based alloys, because the formation of deleterious products has not been found to be as extensive, even if the magnesium is alloyed with aluminum.

The practice of the invention can be further appreciated from the following non-limiting examples in which all parts and percents are by weight unless otherwise specified.

Example 1

A quartz tube 16 cm in length and with an internal diameter of 2.2 cm was coated internally for a distance of about 15 cm with a suspension of colloidal graphite available from Acheson Colloids, Ltd., Brantford, Ontario, Canada, under the trade name AQUADAG. A supporting rod, narrower than the inside diameter of the quartz tube but having on one end a gas-permeable plug of porous refractory fireclay brick that fits tightly within the coated tube, was then inserted from the uncoated end and positioned so that the porous plug was about 10 cm from the opening of the coated end. The container thus formed by the coated tube end and the porous plug was filled with Grit F600 green silicon carbide to a packing density of about 50 volume %, with the aid of a vibrating table contacting the container. (The size distribution of Grit F600 silicon carbide is described fully in publications of the Federation of European Producers of Abrasives, hereinafter "FEPA") The particular lot of Grit F600 used for this experiment was measured with a Coulter Counter and had 50% of its volume in particles with a size of more than 9.1 micrometers; 3% of the volume was made up of particles (grits) larger than 15.3 micrometers, 94% of the volume was made up of particles (grits) larger than 4.8 micrometers, and the central 75% of the volume was made up of particles (grits) with sizes between 6.2 and 12.2 micrometers. The central 75% of the volume is defined as the part of the sample excluding the largest and the smallest particles that each make up 12.5% of the total volume.

The top surface of the packed bed of SiC was covered with a layer of porous alumina paper (Product APA1 from Zircar Products, Florida, New York) and this end of the container was then wrapped with aluminum foil. The porous alumina paper is fitted tightly enough to keep the packed bed from falling out when the container is inverted and to serve as a filter to exclude oxides or other unwanted foreign matter when molten metal is later infiltrated into the packed bed. The aluminum foil allows the protected end of the container to be immersed in the molten aluminum alloy without contaminating the contents with a layer of oxide

that forms spontaneously on molten aluminum alloys. Shortly after immersion, the aluminum foil melts. The amount of aluminum foil is too small to change the composition of the molten aluminum alloy to any significant extent.

The wrapped container with its packed bed was then placed in a gas tight desiccator that was evacuated to a pressure of no more than 0.01 bar and then backfilled with argon. The container as thus prepared was positioned within an apparatus illustrated in Figure 1. The quartz tube 1 now has the alumina paper 5 at the bottom of the packed bed 4, with the porous fireclay plug 3 and the support rod 2 on top. The tube 1 is connected via a gas tight fitting 6 to a channel 7 that allows the input or exit of gas from the space above the porous plug independently of the space 8 in the upper part of the apparatus.

The tube with its packed bed was immersed as shown in Figure 1 in a bath of molten A357 aluminum alloy (which contains about 7% silicon) 9, with a melting point of 610 °C, maintained within a graphite crucible 10 at a temperature of 700 °C by a conventional heating element 11. The crucible and heater are within a gas tight space defined by container 12, which is protected from the heat of the heating element 11 by insulation 13. Space 8 was initially filled with argon gas at atmospheric pressure. After preheating the tube containing the packed bed for 5 minutes, at which point the thermocouple 15 showed that the temperature of the molten metal 9 had recovered to the desired value of 700 °C. after being cooled by introduction of the packed bed and its container, the pressure within the furnace above the layer of molten alloy was increased at the rate of 1.36 bar/min by admission of additional argon gas through input channel 14. This pressure caused the fluid alloy to flow through the packed bed from the bottom, displacing gas from the top of the bed through the porous plug into the separate channel 7.

When the pressure reached 13.6 bars, the increase in pressure was discontinued and the pressure maintained at that level for five minutes. The entire furnace was then depressurized through outlet 15 and the tube containing the packed bed of SiC, now infiltrated with molten alloy, was removed and cooled in the air to produce a concentrated composite, which was separated from the quartz tube, by pushing from one end, and then cut into pieces with a diamond saw. A photomicrograph of a polished cross section of the concentrated composite produced is shown in Figure 2.

In a separate graphite crucible, 91 g of A357 alloy was melted and heated to 720 °C, and a piece weighing 62 g of the concentrated composite prepared as described above was placed on top of the molten alloy. After ten minutes, the material

was stirred with a graphite rod to effect a preliminary break-up of the concentrated composite, and a graphite rotating stirrer preheated to 600°C was then immersed and used to stir the melt for 5 minutes at 300 rpm. The crucible was then removed from the furnace and its contents poured into another crucible and cooled. The resulting composite according to this invention with 20 volume percent (hereinafter v/o) SiC was examined microscopically after preparing a polished cross section. There was good distribution of the SiC dispersates throughout the composite, with apparently intimate contact at most SiC-metal interfaces and little porosity, as shown in a micrograph of a cross section of the composite in Figure 3.

Example 2

Particles of Grit F600 silicon carbide were put into the bottom of a steel crucible to give a packed bed with about 50 v/o SiC. A sufficient amount of molten alloy of 90% Mg - 10% Al to infiltrate the entire packed bed was poured over the bed, and the crucible with its contents placed inside a pressurizable furnace maintained at 700°C. Compressed argon was then admitted to the furnace until the pressure reached 34 bars. This was sufficient to cause the molten alloy to impregnate all of the packed bed except for a small pocket at the bottom into which the air originally present in the packed bed had been displaced.

A portion of the fully impregnated concentrated composite prepared as described immediately above was softened at 700°C. and mixed with an additional amount of molten 90% Mg - 10% Al alloy chosen to result in a final composite with 20 v/o SiC. Mixing was initially accomplished with a hand-held stirring rod until the concentrated composite was sufficiently low in apparent viscosity to allow effective mechanical stirring. Mixing was then continued with a double helical stirrer operated at 400 revolutions per minute. This avoided entraining gas through vortex formation. After about five minutes stirring, a semi-solid slurry that could be cast into a mold resulted. The material was then cast and allowed to solidify. A well dispersed final composite was formed.

Example 3

This was performed in the same manner as Example 2, except that Grit F500 (defined hereinbelow in Example 11) rather than Grit F600 silicon carbide was used.

Example 4

This was performed in the same manner as Example 2, except that Grit F320 rather than Grit F600 silicon carbide was used.

Example 5-7

The preparation of the concentrated composite for these examples was performed in substantially the same manner as in Example 2-4 respectively, except that (i) a slightly different apparatus, one that allowed evacuation as well as pressurization of the space within the container for the dispersates, was used; (ii) initially solid alloy material was added to the container above the packed bed; (iii) the space within the container for the dispersates was evacuated after the solid metal and dispersates had been placed within the furnace, which was maintained at 900°C, before pressurizing to cause infiltration; and (iv) a commercially pure magnesium alloy was used for the matrix.

The concentrated composite was mixed with additional molten matrix alloy in a special container, under an argon atmosphere, using an agitator similar to a turbine moving at 2,000 - 3,000 revolutions per minute. A system of baffles in the container prevented any significant gas entrapment during mixing. The dispersion was mixed for about five minutes at a temperature of about 700°C. The stirrer was then immediately removed and the dispersion promptly cast in a copper chill mold about 9 mm deep. A well-dispersed composite with about 15 v/o dispersates resulted.

Example 8

This was performed in the same way as Examples 5-7 except that a finer particle size of silicon carbide, averaging 3 micrometers in size, was used.

Example 9

This example illustrates application of the invention to continuous casting and is accomplished with apparatus shown in cross section in Figure 4. Molten alloy 100 and concentrated composite 101 are continuously fed into a chamber 102 maintained at a temperature that will keep the mixture at least partially fluid. From chamber 102 the mixture is pumped and blended by a rotor 103 into a mixing region 104, where it experiences vigorous agitation. The high shear rates in region 104 are achieved in a narrow gap 104 between chamber wall 106 and a rotor 105. Both the chamber wall and the rotor have surfaces including a conic frustrum with the same taper angle, so that the gap

width, and correspondingly the rate of shear, can be adjusted by relative vertical displacement between the rotor and the chamber wall. The well dispersed dispersion exits in region 107 and can be fed into a crucible for solidification processing, continuously cast into a billet, or the like.

Alternatively, the concentrated composite itself could be rotated vigorously in a bath of molten alloy so that portions of the concentrated composite are peeled off at the interface as the matrix of the concentrated composite softens under the influence of the higher temperature of the bath of molten alloy.

Example 10

This example illustrates another continuous method embodiment of the invention and may be understood with the aid of Figure 5, a cross sectional view of apparatus useful for the invention. A solid chamber 201 capable of withstanding the pressures involved is provided with conventional means for maintaining various temperatures in different regions in its interior and contains two inlets 202 and 203 and an outlet 204. At inlet 202, molten metal 205 is supplied under pressure. At inlet 203, dispersates 206 are supplied at an appropriate rate and also under pressure by means of a ram, screw feeder, or other appropriate device known to those skilled in the art.

During operation of the continuous process of this invention, chamber 201 is kept at a temperature that will maintain molten metal in regions 205 and 207 and at a temperature too low to melt the metal used at the top of region 206, which constitutes a packed bed of dispersates. The flow of dispersates from zone 203 is maintained in a downward direction by mechanical pressure exerted against the packed bed of dispersates, but this does not prevent metal from filling the interparticle space in the packed bed of dispersates in the lower part of the entry region for dispersates, where the temperature is sufficiently high to keep the metal molten. Thus a zone of concentrated composite according to this invention forms in region 207, but upward penetration of the metal is limited by its solidification in the upper part of the inlet 203, creating a more or less distinct boundary between region 207 containing concentrated composite and region 206 with dispersates and gas only.

In the region where the molten metal 205 contacts the concentrated composite 207, the flow rate of the metal is accelerated by a constriction caused by a bulge 208 in the chamber wall. In the region between 208 and 207, the concentrated composite is continuously entrained downstream by the rapidly flowing molten metal and it is sheared and

dispersed into the flowing metal. At a sufficient distance downstream from the constriction, a region 209 of substantially homogeneous and non-porous dispersion is obtained. This dispersion can be continuously cast from the outlet 204 to yield a solid continuous billet 210 of the finally desired composite. The volume fractions of metal matrix and dispersates are controlled by regulating the relative feeding rates of dispersates and molten metal at their inlets 203 and 202 respectively.

Instead of a constriction, separate mechanical or electromagnetic stirring could be used to disperse the concentrated composite into additional matrix precursor.

Example 11

This was performed in the same manner as Example 1, except that the SiC particulates used were a mixture of equal volumes of FEPA Grit F400, Grit F500, and Grit F600. The Grit F600 had the same size distribution as in Example 1. The Grit F500 material had 3% of its volume in particles larger than 22.5 micrometers, 50% of its volume in particles larger than 13.7 micrometers, 94% of its volume in particles larger than 8.7 micrometers, and the central 75% of its volume in particles with sizes between 10.6 and 17.7 micrometers, all as measured by a Coulter Counter. Using the same measurement technique, the Grit F400, material had 3% of its volume in particles larger than 25 micrometers, 50% of its volume in particles larger than 17 micrometers, 94% of its volume in particles larger than 12 micrometers, and the central 75% of its volume in particles with sizes between 13 and 20.5 micrometers.

The central 75% of the volume of the mixture had particles between 7.8 and 19 micrometers, 3% of the volume of the mixture was in particles smaller than 5.2 micrometers, and 94% of the volume of the mixture was in particles larger than 21 micrometers. The final composite produced had an apparently uniform distribution of all particle sizes of SiC within the matrix when examined in cross section.

Example 12

This was the same as Example 1, except that the dispersates used were boron carbide rather than silicon carbide. Good redistribution of the concentrated composite was obtained in the final composite.

Example 13

This was performed in the same as Example 1, except that (i) 100 g of concentrated composite and 215 g of additional A357 alloy were used, to

give a 15 v/o composite; (ii) the melt temperature during the mixing of the concentrated composite into the additional molten alloy was only 670°C rather than 700°C; and final stirring was for only 2.5 minutes instead of five. The difference in temperature considerably increased the apparent viscosity during the mixing of the concentrated composite with additional matrix material, and some large air pores were introduced during the stirring and preserved in the final composite. Therefore, even though the SiC dispersates were again well dispersed within the final composite, the results were less preferable than for Example 1.

Example 14

This was performed in the same way as Example 1, except that (i) the alloy used was Type 6061 alloy rather than the A357 and (ii) 100 g of concentrated composite and 149 g of additional molten alloy were used in the final mixing step.

Type 6061 alloy contains 0.6% Si, 1.0% Mg, 0.3% Mn, and 0.2% Cr, with the balance aluminum. Presumably because of the very low silicon content, the dispersion of the concentrated composite within the final composite was not nearly so good as in Example 1. A micrograph of a cross section of the final composite produced in this example is shown in Figure 6.

Example 15

This was performed in the same way as Example 14, except that the alloy used was 10% Si - 90% Al. The result contrasted sharply with that of Example 14, in that the distribution of the SiC dispersates within the final composite was very uniform.

Example 16

This was performed in the same way as Example 14, except that the alloy used contained 99.9% aluminum. The dispersion of silicon carbide in the final composite was less uniform than that achieved in Example 14.

Example 17

This was performed in the same way as Example 16, except that (i) the temperature both for preparation and for mixing of the concentrated composite was raised to 800°C; and (ii) 71 g of concentrated composite and 152 g of additional alloy were used in the mixing step, which should have given a final composite with only 15 v/o SiC. In fact, however, mixing the concentrated composite with additional metal proved to be practically

impossible. X-ray diffraction of a sample of the concentrated composite showed the presence of aluminum carbide at the interfaces between the dispersates and the matrix. This is believed to be the reason that the concentrated composite was so difficult to break up.

Example 18

This was performed in the same manner as Example 17, except that (i) the final mixing was at 720°C rather than 800°C and (ii) the SiC particulates used were heated in air at 1300°C for thirty minutes before being infiltrated to form the concentrated composite. This treatment is known to form a layer of SiO₂ on the surface of silicon carbide. The silica surface greatly retards the formation of aluminum carbide, as confirmed by an X-ray analysis of this concentrated composite, and thus the concentrated composite could easily be dispersed in the final mixing step. A micrograph of a cross section of the final composite thereby produced is shown in Figure 6.

Example 19

This was the same as Example 1, except that the SiC particulates, before forming the concentrated composite, were coated with alumina in the following manner: a boehmite sol at 10 w/o total solids containing 0.15 w/o of fine alpha alumina seeds, prepared in a state of incipient gellation as described in detail in U.S. Patent 4,623,364 was prepared. One liter of this sol was mixed with one kilogram of FEPA Grit 600 SiC, and the mixture then pumped through a NIRO spray drier, which caused the SiC to be coated with an apparently uniform coating of dried alumina gel when examined by as scanning electron microscope. The coated particulate was then heated at 1200°C for thirty minutes to convert the alumina gel to alpha alumina. Conversion was confirmed by x-ray diffraction analysis that showed alpha SiC and alpha alumina as the only phases present.

The final composite prepared in this Example showed an excellent uniformity of dispersion of the SiC within the matrix.

Comparative Example A

SiC was coated with Cr metal by an evaporation-condensation process to 29%. Infiltration attempts with both A357 and Al 99.9 were made using quartz tubes 20 cm. long and .5 cm. ID. The temperature of the infiltrating aluminum was varied between 700°C and 800°C, and the pressure varied up to 27.58 bar (400 psi). In both cases infiltration was negligible. It is believed that the chrome

metal was dissolved by the molten aluminum producing an aluminum chrome alloy. Small additions of chrome to aluminum significantly raise the melting temperature, thus freezing the metal front before infiltration can occur. Thus chrome metal is not a useful coating material.

Comparative Example B

SiC powder, coated as in Comparative Example A, was calcined in air at 1200 °C for 1/2 hour to promote the formation of chromia around each particle. The calcining only converted the surface of the chrome coating to chromia. This powder was packed and infiltrated with A357 as in Example 1 using a pressure of 375 psi and a temperature of 750 °C. 89 g of this master composite was heated to 850 °C with 209g of A357 to give a final composition of 14 %. After 2 1/2 hours, the master composite pieces had not yet broken up; thus little dispersion occurred. The protective barrier of Cr₂O₃ on each particle dissolved, allowing the Al to react with the chrome, as in Comparative Example A, to form a high melting alloy and to prevent the dispersion of the master composite. Thus a surface chromia layer on a predominantly chrome coating was not suitable.

Example 20

A master alloy was prepared using A357 and SiC coated with chrome to 3.5 wt% and calcined as in Comparative Example B. In this case, however, the Cr was completely oxidized to chromia. The infiltration occurred at 750 °C with 400 psi. 94.9 g of this concentrated dispersion sample was dispersed into 235 g of A357 to give a composite of 13.4 wt% SiC. This took 15 minutes at 850 °C. Micrographs indicate a good dispersion with a few clumps of master alloy remaining which can be broken down with improved stirring. In this case the chrome oxide coating prevented any reaction between the aluminum metal and the SiC to allow dispersion of the master alloy to occur. The absence of any chrome metal in the coating also prevents the melting point of the alloy from rising which had been found to prevent dispersions of the master composite in Comparative Examples A and B.

Example 21

A coating of Si₃N₄ on SiC particulate was produced by heating SiC in flowing nitrogen gas to 1425 °C. A master composite was produced as in Example #1 using A357 at 20.68 bar (300 psi) and 725 °C. 75 g of this master composite was stirred into 192 g of A357 as in Example 20 to give a final

fraction of 13.1% SiC. Dispersion occurred easily. Micrographs of the final composite indicate good dispersion of the coated SiC particulate without any noticeable reaction products being formed between the metal matrix and the coated SiC dispersates.

Comparative Example C

SiC was coated to 8.3% Ni metal using a NiB electroless plating solution. Infiltration was accomplished using quartz tubes, as in Example #19, with the A357 at 725 °C and applying a pressure of 200 psi. 9.3 g of this master alloy was dispersed into 57.9 g of A357 using a small porcelain crucible and a graphite stirring rod. Furnace temperature was set to 800 °C. The master composite did not disperse evenly, presumably due to nickel-silicon alloy formation, thus indicating that a nickel metal coating is not suitable with this aluminum alloy.

Example 22

The procedure of Comparative Example C was repeated reducing the nickel content on the dispersates to 2% and using Al 99.9 as the matrix material and in both the infiltration and the dispersion. A good dispersion results.

Example 23

SiC was given a total 9.5 % coating of a chromia-alumina solid solution. This coating was produced by mixing equal weights of SiC and a 30 % solids solgel of Cr(NO₃)₃·9H₂O and Al₂O₃·H₂O. The resulting slurry was dried to a powder and fired at 1250 °C to remove structural water in the coating materials and to convert the nitrate to an oxide. This SiC was infiltrated with A357 as in Example 1 using a temperature of 725 °C and a pressure of 11.03 bar (160 psi). 5.8 g of this master alloy was then stirred into 60.7 g of A357 to give a composite having 4.0% SiC. Dispersion was good.

Example 24

A 6% molybdenum coating was placed on silicon carbide particles by mixing coarse molybdenum powder with F600 SiC in an appropriate weight ratio and heating in a vacuum furnace to evaporate the molybdenum and redeposit it on the SiC particles. The coated particles were then processed as in Example 1 with A357 alloy to form a concentrated dispersion thereof containing 54% dispersates. The concentrated dispersion was then stirred into further A357. Good break-up of the concentrated dispersion is observed and a uniform final composite is produced.

Example 25

The procedure of Example 24 was repeated to produce a 6% titanium metal coating on silicon carbide dispersates, to use those dispersates to prepare a concentrated dispersion, and to use the concentrated dispersion to prepare a final uniform dispersion. A good dispersion resulted.

Example 26

The procedure of Example 25 was repeated to produce the titanium metal coated silicon carbide dispersates and then the titanium coating was oxidized to convert it to titania. Infiltration with A357 at a pressure of 11.03 bar (160 psi) was barely adequate to produce a void-free concentrated dispersion, but when repeated at 20.68 bar (300 psi) the process proceeded smoothly. Good final dispersions were produced in each case.

Example 27

A titanium nitride coating was placed on silicon carbide dispersates by (i) forming a silica and carbon diffusion layer by calcining the SiC in air at 1100°C., (ii) coating titanium metal thereon by a pack diffusion process, and then (iii) nitriding at 925 - 1025°C. to convert the metallic Ti to TiN. The resulting dispersates were readily infiltrated with pure aluminum to form a concentrated dispersion which was stirred into further pure aluminum to produce a well-dispersed final composite product.

Claims

1. A process for manufacturing a final composite product having a plurality of discrete solid ceramic dispersates within a solid metal matrix which comprises:

(a) forming a concentrated dispersion of the ceramic dispersates within a first quantity of a fluid material which is one portion of said solid metal matrix material, wherein said forming is by pressure infiltration of a packed porous bed of said discrete solid ceramic particles with said first quantity of said fluid metal material;

(b) placing at least a portion of the concentrated dispersion into a second quantity of said fluid metal material which is a second portion of said solid metal matrix;

(c) processing the product of step (b) so that the ceramic particles in the concentrated dispersion separate and disperse within a mixture of both of the first quantity and the second quantity of said fluid metal

material; and

(d) solidifying the product of step (c) to form the final composite product.

2. A process according to claim 1, wherein said packed porous bed is evacuated before said infiltrating.
3. A process according to claim 1 or 2, wherein said packed porous bed contains interparticle gas and said infiltrating is performed from one side of said bed at a sufficiently slow rate to allow displacement of the gas originally within said packed bed by said first fluid material.
4. A process according to any one of claims 1-3, wherein the mixing of at least a portion of the concentrated composite with the second quantity of fluid material is performed at a temperature at least about 70°C. higher than the melting point of the matrix of the final composite.
5. A process according to any one of the preceding claims, wherein the concentrated dispersion comprises 25 to 85 weight percent ceramic dispersates and 75 to 15 weight percent metal matrix precursor.
6. A process according to any one of the preceding claims, which includes solidifying at least part of said first quantity of said fluid material of said concentrated dispersion before step (b) is performed.
7. A process according to any one of the preceding claims, wherein said first quantity of said fluid material contains a material which facilitates wetting of the dispersates without promoting deleterious chemical reactions.
8. A process according to any one of the preceding claims, wherein said dispersates are comprised predominantly of silicon carbide or boron carbide and said matrix material is comprised predominantly of aluminum or magnesium.
9. A process according to any one of claims 1-7, wherein the metal matrix material contains predominantly aluminum and silicon.
10. A process according to any one of claims 1-7, wherein said matrix material comprises predominantly aluminum.
11. A process according to any one of claims 1-7, wherein said solid ceramic dispersates have a surface layer of a barrier material which re-

tards, at the temperature of formation of the concentrated dispersion and at the temperature of formation of the final composite, a chemical reaction that would otherwise occur between the material of the dispersates and said fluid metal material.

12. A process according to claim 11, wherein said barrier layer is comprised predominantly of a material which is a metal, a metal oxide, a metal nitride, a metal silicide, a metal carbide, or a metal boride.

13. A process according to claim 12, wherein said barrier material is copper, molybdenum, nickel, zinc, tin, titanium, silica, alumina, chromia, nickel oxide, copper oxide, mullite, spinel, titania, magnesium silicate, lithium silicate, silicon nitride, titanium nitride, boron nitride, aluminum nitride, molybdenum silicide, copper silicide, or titanium silicide, and wherein the surface layer is up to 2 micrometers thick.

14. A process according to any one of claims 11-13, wherein said first quantity of said fluid metal material contains predominantly aluminum and less than 12% silicon.

15. A process according to any one of the preceding claims, wherein the final composite comprises 5 to 40 volume percent discrete dispersates and not more than 5 volume percent pores, voids, and/or gases.

Patentansprüche

1. Verfahren zur Herstellung eines End-Verbundprodukts mit einer Vielzahl von einzelnen festen, dispergierten Keramikeilchen in einer festen Metallmatrix, welches umfaßt:

(a) Bildung einer konzentrierten Dispersion der keramischen dispergierten Teilchen in einer ersten Menge eines fluiden Materials, das ein Teil dieses festen Metallmatrix-Materials ist, wobei diese Bildung durch Druckinfiltration eines gepackten porösen Bettes dieser diskreten festen Keramikeilchen mit dieser ersten Menge des genannten fluiden Metallmaterials erfolgt;

(b) Einbringen wenigstens eines Teils der konzentrierten Dispersion in eine zweite Menge dieses fluiden Metallmaterials, das ein zweiter Teil dieser festen Metallmatrix ist;

(c) Verarbeiten des Produkts von Stufe (b), so daß die Keramikeilchen in der konzentrierten Dispersion sich trennen und in einer Mischung beider, der ersten Menge und der

zweiten Menge dieses fluiden Metallmaterials, dispergieren; und

(d) Verfestigung des Produkts von Stufe (c) zur Bildung des End-Verbundprodukts.

2. Verfahren gemäß Anspruch 1, wobei dieses gepackte poröse Bett vor diesem Infiltrieren evakuiert wird.

3. Verfahren gemäß Anspruch 1 oder 2, wobei dieses gepackte poröse Bett zwischen den Teilchen Gas enthält und dieses Infiltrieren von einer Seite dieses Bettes in einer hinreichend niedrigen Geschwindigkeit erfolgt, um die Verdrängung des ursprünglich innerhalb dieses gepackten Bettes vorhandenen Gases durch dieses erste fluide Material zu erlauben.

4. Verfahren gemäß einem der Ansprüche 1 bis 3, wobei das Vermischen wenigstens eines Teils des konzentrierten Verbundes mit der zweiten Menge des fluiden Materials bei einer Temperatur von wenigstens etwa 70 °C höher als der Schmelzpunkt der Matrix des End-Verbundstoffs durchgeführt wird.

5. Verfahren gemäß einem der vorhergehenden Ansprüche, wobei die konzentrierte Dispersion 25 bis 85 Gew.-% keramische dispergierte Teilchen und 75 bis 15 Gew.-% Metallmatrix-Vorprodukt umfaßt.

6. Verfahren gemäß einem der vorhergehenden Ansprüche, welches die Verfestigung wenigstens eines Teils dieser ersten Menge dieses fluiden Materials der genannten konzentrierten Dispersion umfaßt, bevor die Stufe (b) durchgeführt wird.

7. Verfahren gemäß einem der vorhergehenden Ansprüche, wobei diese erste Menge des genannten fluiden Materials ein Material enthält, welches das Befeuchten der dispergierten Teilchen erleichtert, ohne daß schädliche chemische Reaktionen gefördert werden.

8. Verfahren gemäß einem der vorhergehenden Ansprüche, wobei diese dispergierten Teilchen vorwiegend aus Siliciumcarbid oder Borcarbid bestehen und das genannte Matrixmaterial vorwiegend aus Aluminium oder Magnesium besteht.

9. Verfahren gemäß einem der Ansprüche 1 bis 7, wobei das Metallmatrix-Material vorwiegend Aluminium und Silicium enthält.

10. Verfahren gemäß einem der Ansprüche 1 bis 7, wobei dieses Matrixmaterial vorwiegend aus Aluminium besteht.
11. Verfahren gemäß einem der Ansprüche 1 bis 7, wobei diese festen keramischen, dispergierten Teilchen eine Oberflächenschicht eines Sperrschichtmaterials haben, welches bei der Temperatur der Bildung der konzentrierten Dispersion und bei der Temperatur der Bildung des endgültigen Verbunds eine chemische Reaktion verzögern, welche sonst zwischen dem Material der dispergierten Teilchen und dem fluiden Metallmaterial auftreten würde.
12. Verfahren gemäß Anspruch 11, wobei diese Sperrschicht vorwiegend aus einem Material besteht, das ein Metall, ein Metalloxid, ein Metallnitrid, ein Metallsilicid, ein Metallcarbid oder ein Metallborid ist.
13. Verfahren gemäß Anspruch 12, wobei dieses Sperrschichtmaterial Kupfer, Molybdän, Nickel, Zink, Zinn, Titan, Siliciumdioxid, Aluminiumoxid, Chromoxid, Nickeloxid, Kupferoxid, Mullit, Spinell, Titanoxid, Magnesiumsilikat, Lithiumsilikat, Siliciumnitrid, Titanitrid, Bornitrid, Aluminiumnitrid, Molybdänsilicid, Kupfersilicid oder Titansilicid ist, und wobei die Oberflächenschicht bis zu 2 Mikrometer dick ist.
14. Verfahren gemäß einem der Ansprüche 11 bis 13, wobei die erste Menge dieses fluiden Metallmaterials vorwiegend Aluminium und weniger als 12 % Silicium enthält.
15. Verfahren gemäß einem der vorhergehenden Ansprüche, wobei der Endverbund 5 bis 40 Vol-% diskrete dispergierte Teilchen und nicht mehr als 5 Vol-% Poren, Lücken bzw. Lehrstellen und/oder Gase enthält.
- (b) l'introduction d'au moins une partie de ladite dispersion concentrée dans une seconde quantité dudit matériau métallique liquide qui constitue une seconde partie de ladite matrice métallique solide;
- (c) le traitement du produit de l'étape (b) de manière que les particules céramiques dans la dispersion concentrée se séparent et se dispersent dans un mélange de la première quantité et de la seconde quantité dudit matériau métallique liquide; et
- (d) la solidification du produit de l'étape (c) pour former le produit composite final.
2. Procédé selon la revendication 1, dans lequel ledit lit poreux tassé est mis sous vide avant ladite infiltration.
3. Procédé selon la revendication 1 ou 2, dans lequel ledit lit poreux tassé contient un gaz interparticulaire et ladite infiltration est accomplie depuis un côté dudit lit à une vitesse suffisamment lente pour permettre le déplacement par ledit premier matériau liquide du gaz initialement présent dans ledit lit tassé.
4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel le mélange d'au moins une partie du composite concentré avec la seconde quantité de matériau liquide est accompli à une température supérieure d'au moins environ 70 °C au point de fusion de la matrice du composite final.
5. Procédé selon l'une quelconque des revendications précédentes, dans lequel la dispersion concentrée comprend 25 à 85% en masse d'éléments céramiques dispersés et 75 à 15% en masse de précurseur de matrice métallique.
6. Procédé selon l'une quelconque des revendications précédentes, qui comprend la solidification d'au moins une partie de ladite première quantité dudit matériau liquide de ladite dispersion concentrée avant l'accomplissement de l'étape (b).
7. Procédé selon l'une quelconque des revendications précédentes, dans lequel ladite première quantité dudit matériau liquide contient un matériau qui facilite le mouillage des éléments dispersés sans favoriser des réactions chimiques nocives.
8. Procédé selon l'une quelconque des revendications précédentes, dans lequel lesdits éléments dispersés sont constitués principalement par du carbure de silicium ou du carbure

Revendications

1. Procédé de fabrication d'un produit composite final ayant une pluralité d'éléments céramiques solides discrets dispersés dans une matrice métallique solide, qui comprend:
- (a) la formation d'une dispersion concentrée des éléments céramiques dispersés dans une première quantité d'un matériau liquide qui constitue une partie dudit matériau de matrice métallique solide, ladite formation étant accomplie par infiltration sous pression d'un lit poreux tassé desdites particules céramiques solides discrètes avec ladite première quantité dudit matériau métallique liquide;

de bore et ledit matériau de matrice est constitué principalement par de l'aluminium ou du magnésium.

9. Procédé selon l'une quelconque des revendications 1 à 7, dans lequel le matériau de matrice métallique contient principalement de l'aluminium et du silicium. 5
10. Procédé selon l'une quelconque des revendications 1 à 7, dans lequel ledit matériau de matrice comprend principalement de l'aluminium. 10
11. Procédé selon l'une quelconque des revendications 1 à 7, dans lequel lesdits éléments céramiques solides dispersés ont une couche superficielle constituée par un matériau formant barrière qui, à la température de formation de la dispersion concentrée et à la température de formation du composite final, retarde une réaction chimique qui, autrement, se produirait entre le matériau des éléments dispersés et ledit matériau métallique liquide. 15
20
12. Procédé selon la revendication 11, dans lequel ladite couche formant barrière est constituée principalement par un matériau qui est un métal, un oxyde métallique, un nitrure métallique, un siliciure métallique, un carbure métallique ou un borure métallique. 25
30
13. Procédé selon la revendication 12, dans lequel ledit matériau formant barrière est le cuivre, le molybdène, le nickel, le zinc, l'étain, le titane, la silice, l'alumine, l'oxyde de chrome, l'oxyde de nickel, l'oxyde de cuivre, la mullite, le spinelle, l'oxyde de titane, le silicate de magnésium, le silicate de lithium, le nitrure de Silicium, le nitrure de titane, le nitrure de bore, le nitrure d'aluminium, le siliciure de molybdène, le siliciure de cuivre ou le siliciure de titane, et dans lequel la couche superficielle est épaisse de jusqu'à 2 micromètres. 35
40
45
14. Procédé selon l'une quelconque des revendications 11 à 13, dans lequel ladite première quantité dudit matériau métallique liquide contient principalement de l'aluminium et moins de 12% de silicium. 50
15. Procédé selon l'une quelconque des revendications précédentes, dans lequel le composite final comprend 5 à 40% en volume d'éléments dispersés discrets et pas plus de 5% en volume de pores, de vides et/ou de gaz. 55

FIG. 1

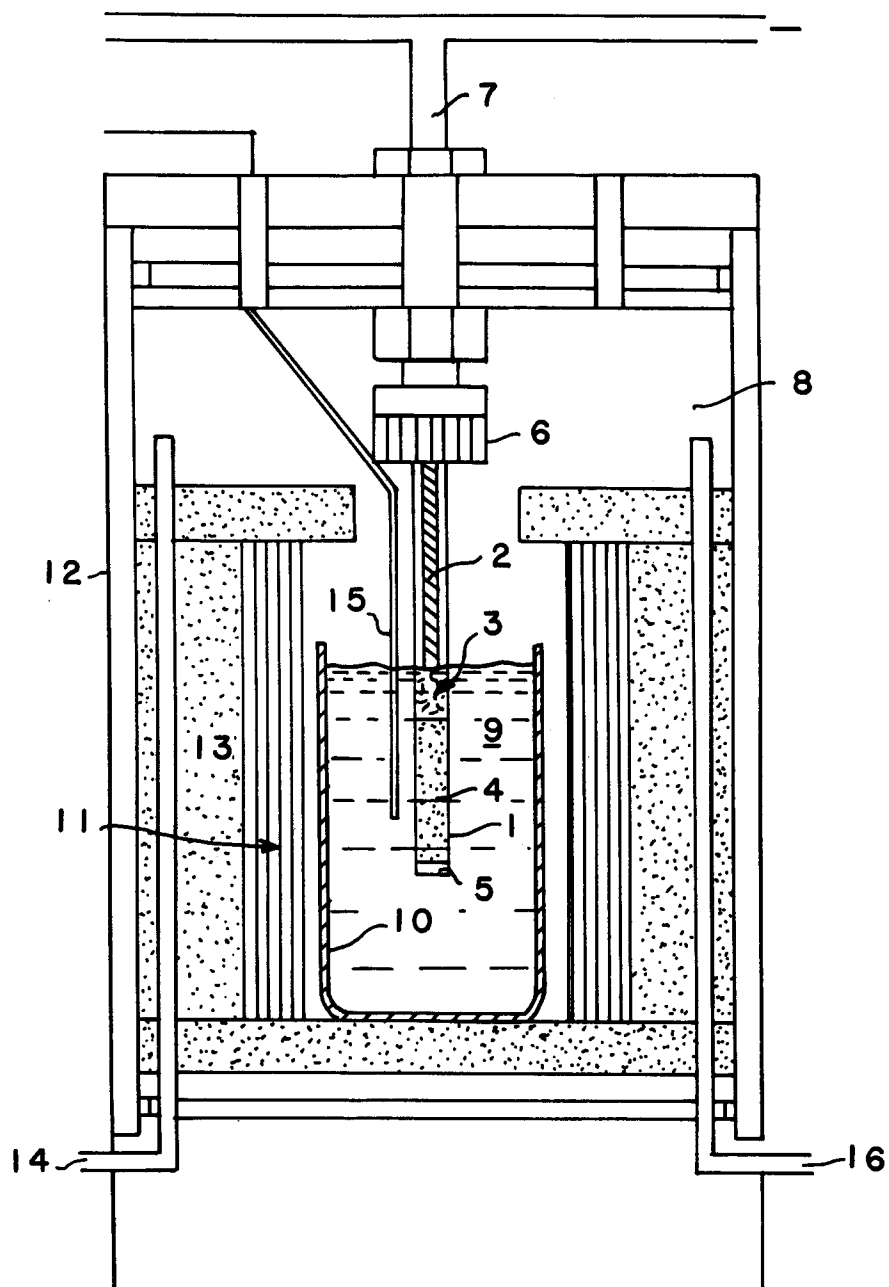
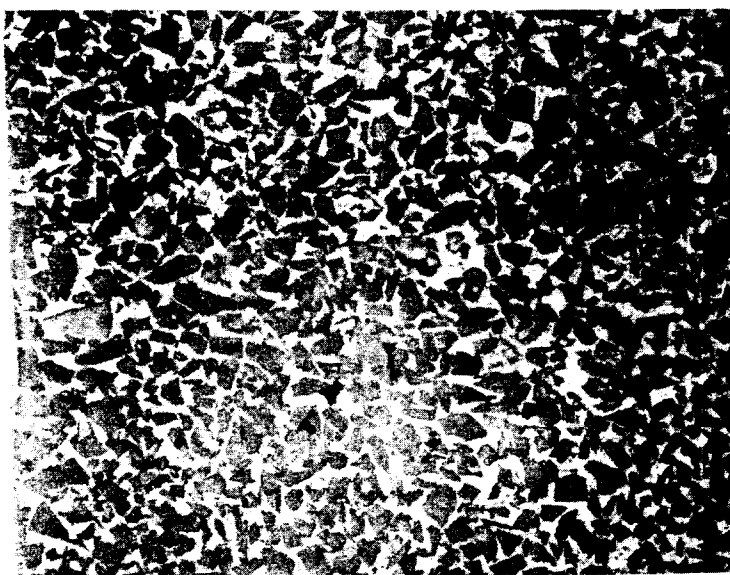
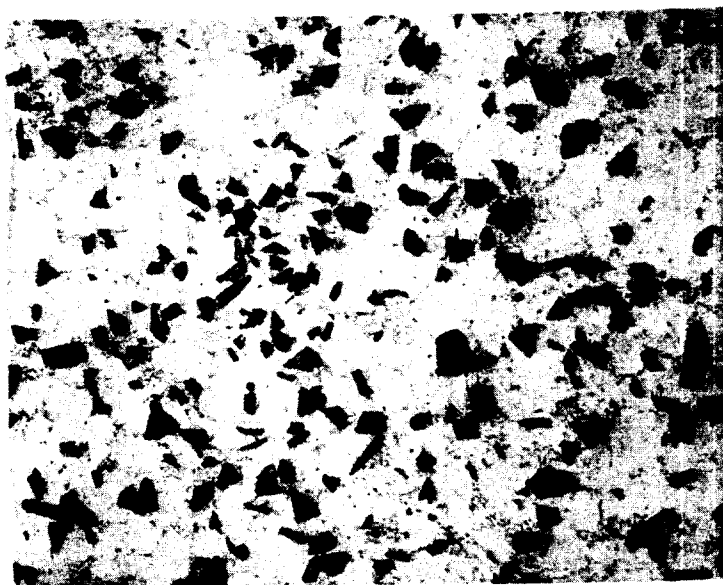


FIG. 2



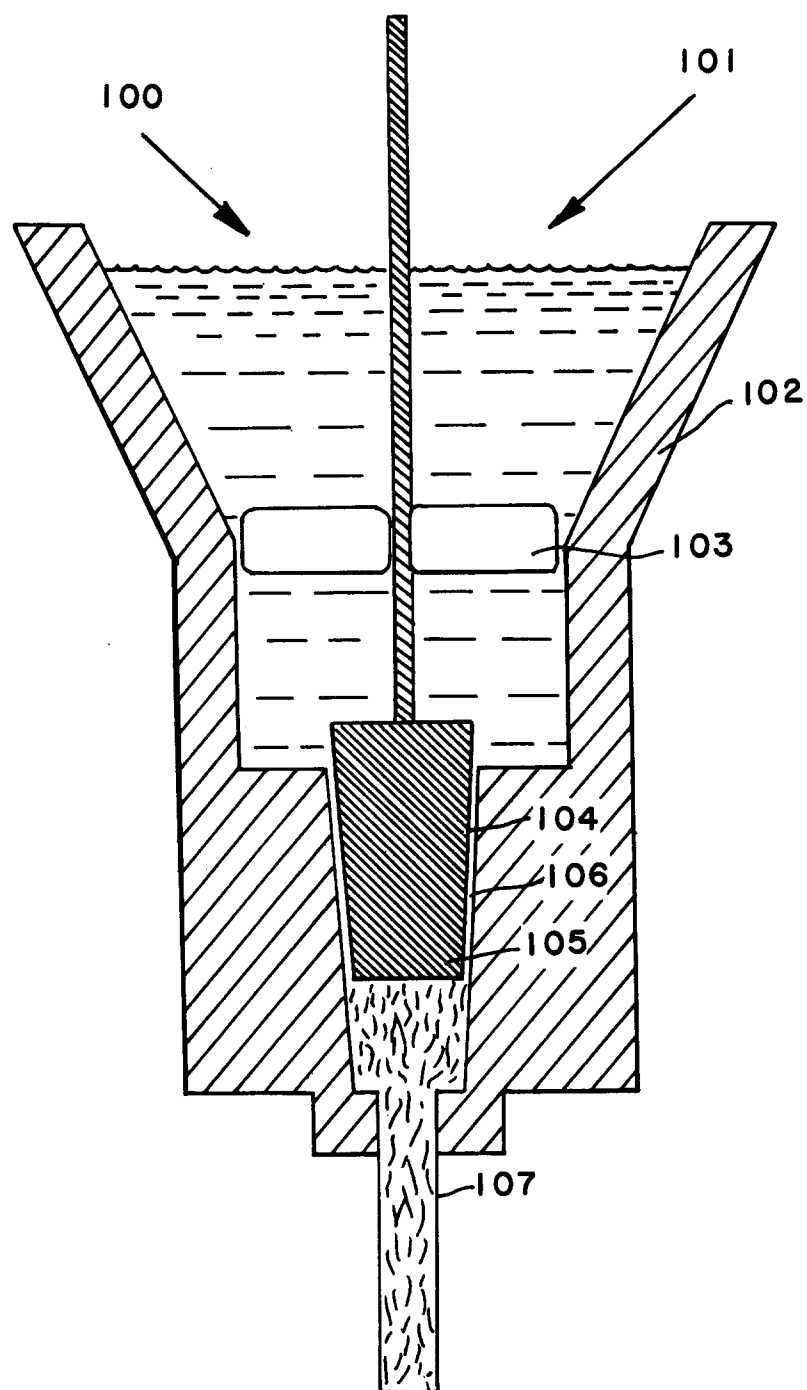
A357/SiC F600 M.A.
400X 3/21/88

FIG. 3



A357/F600 20% FROM M.A.
400X 3/22/88

FIG. 4



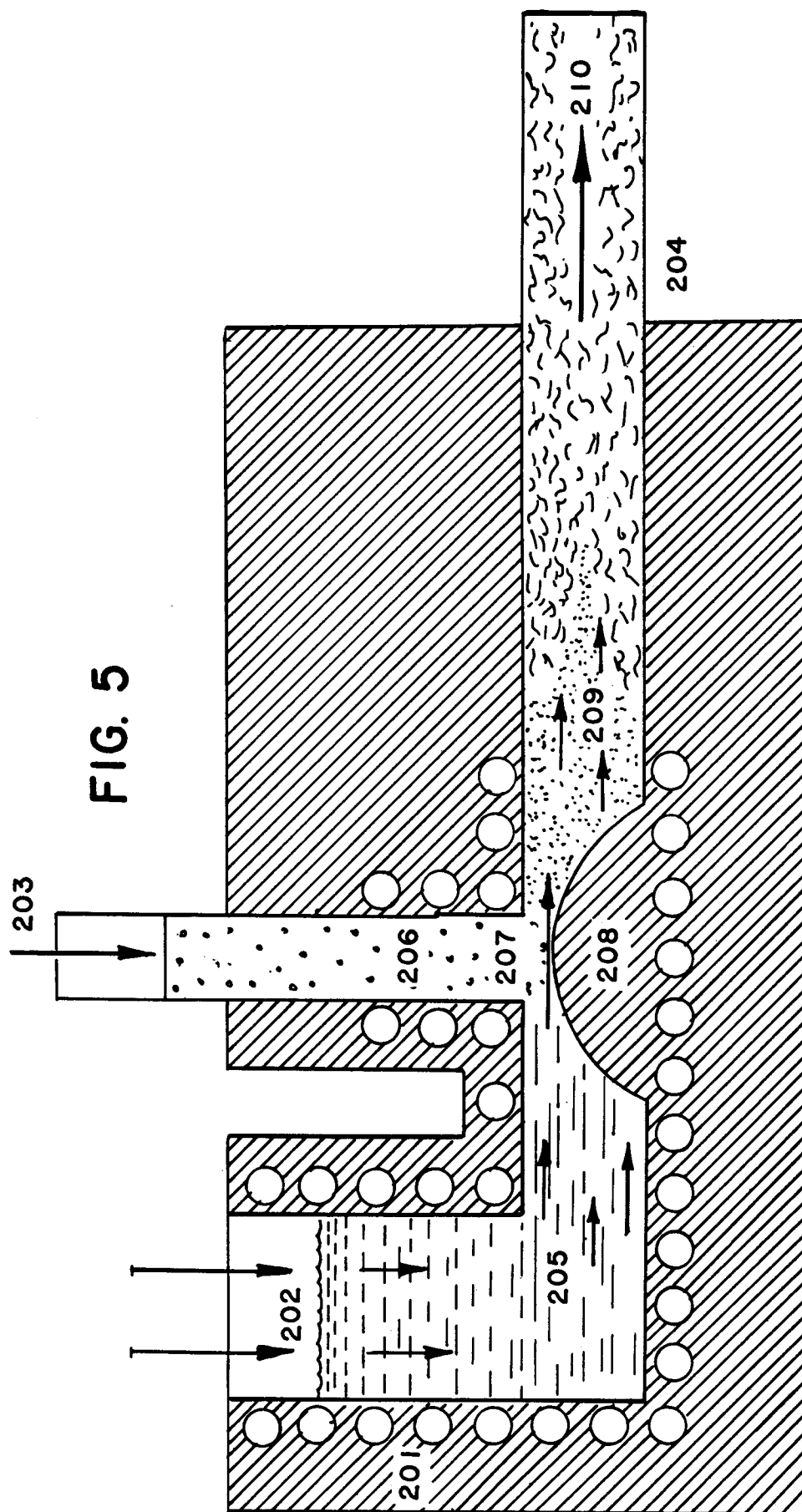
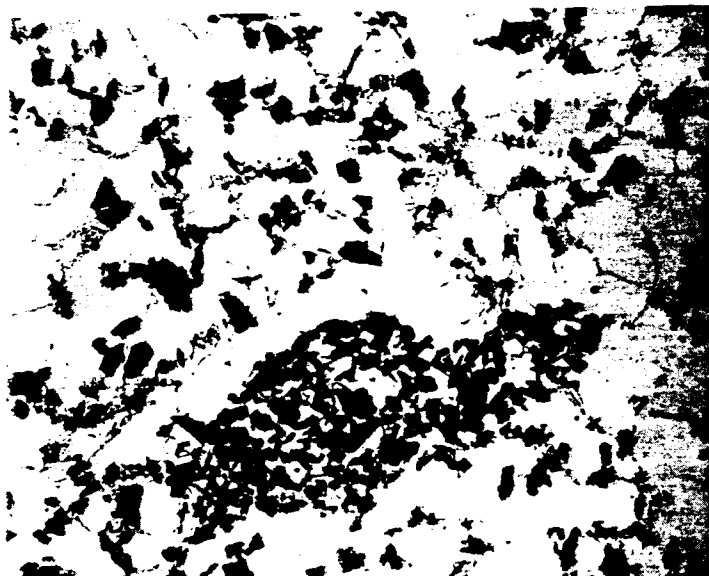
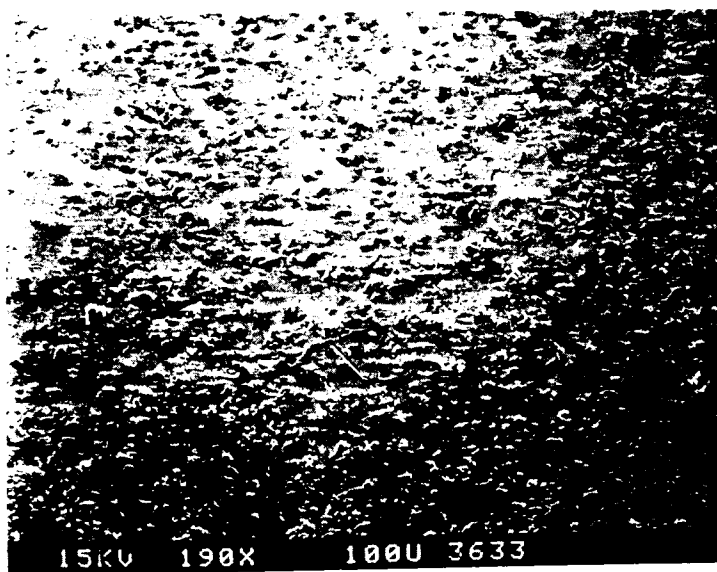


FIG. 6



6061/SiC F600 20% FROM M.A.
400X
3/21/88

FIG. 7



MICROSTRUCTURE OF 99.9Al/15VOL%
SiC COMPOSITE
SiC HEAT TREATED IN AIR 1300C
30 MIN. BEFORE INFILTRATION