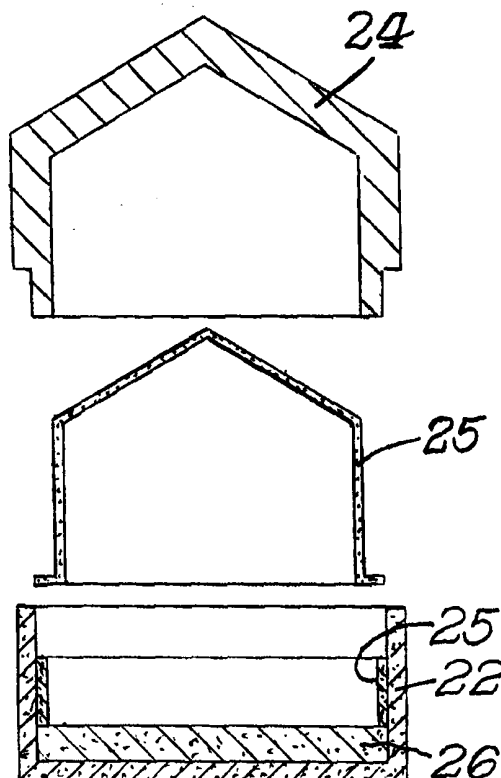




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(54) Title: METHOD FOR MAKING A METAL MATRIX COMPOSITE BODY COMPRISING A REINFORCEMENT PHASE PRODUCED <i>IN SITU</i> (57) Abstract <p>A new technique is described for producing a metal matrix composite body whose reinforcement phase is formed in situ. Specifically, an infiltration enhancer material is applied to a surface of a substantially non-reactive material, e.g., a mold surface. A molten matrix metal is then contacted to the infiltration enhancer material. The molten matrix metal spontaneously infiltrates the infiltration enhancer material. Concurrent with infiltration, at least a portion of the infiltration enhancer material is reduced chemically by the matrix metal to form as a reaction product a ceramic material. The ceramic material, embedded by matrix metal, essentially constitutes a metal matrix composite (MMC) material. Upon formation of the MMC material having the reinforcement phase produced in situ, the process may be repeated by coating or depositing fresh infiltration enhancer material on the previously formed MMC material. In a particularly preferred embodiment, the matrix metal is forced to infiltrate against the force of gravity, the infiltration enhancer is applied by a chemical vapor deposition technique, and the surface against which the MMC is formed is readily separated intact from the MMC body upon completion of spontaneous infiltration and solidification of the matrix metal, thereby permitting the surface, e.g., a mold, to be reused in another MMC fabrication run.</p>		



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METHOD FOR MAKING A METAL MATRIX COMPOSITE BODY
COMPRISING A REINFORCEMENT PHASE PRODUCED IN SITU

5 TECHNICAL FIELD

The present invention relates to a new technique for producing a metal matrix composite body, and the body produced thereby. In particular, the present invention relates to thin metal matrix composite bodies, most or all of whose reinforcement phase is produced in situ.

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BACKGROUND ART

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Composite products comprising a metal matrix and a strengthening or reinforcing phase such as ceramic particulates, whiskers, fibers or the like, show great promise for a variety of applications because they combine some of the stiffness and wear resistance of the reinforcing phase with the ductility and toughness of the metal matrix. Generally, a metal matrix composite ("MMC") material will show an improvement in such properties as strength, stiffness, contact wear resistance, and elevated temperature strength retention relative to the matrix metal in monolithic form, but the degree to which any given property may be improved depends largely on the specific constituents, their volume or weight fraction, and how they are processed in forming the composite. In some instances, the composite also may be lighter in weight than the matrix metal per se. Aluminum matrix composites reinforced with ceramics such as silicon carbide in particulate, platelet, or whisker form, for example, are of interest because of their high stiffness, wear resistance and high temperature strength relative to aluminum.

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U.S. Patent No. 3,608,170 to Larson et al. discloses a technique for producing a metal composite article having a metallic substrate or backing. In particular, a porous preform of non-metallic material is placed into, but not completely filling, a casting mold. Molten metal, such as iron or copper, is poured into the mold and caused (e.g., by pressure or vacuum) to infiltrate the preform to produce a composite body having a homogeneous metal backing part.

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U.S. Patent No. 4,673,435 to Yamaguchi et al. discloses a method for producing an alumina composite body; particularly to a composite body comprising alumina, aluminum and silicon. In particular, a glass body comprising silica is immersed into

molten aluminum of at least 99 percent purity, under vacuum or inert atmosphere. The aluminum chemically converts the silica to alumina plus silicon. Thus a body comprising alumina and a solid solution of aluminum and silicon is produced. After that, the body is removed from the molten aluminum pool and heated under vacuum at a temperature of about 780°C to 950°C to remove excess aluminum on the surface of the body, react any residual silica remaining in the body and relieve stresses.

U.S. Patent No. 4,916,029 to Nagle et al. discloses a method for making a composite material comprising an in situ precipitate second phase in an intermetallic matrix. Specifically, the constituents or elements of the desired second phase material are mixed with a solvent matrix material comprising an intermetallic, or precursor thereof. The mixture is then heated to a temperature at which an exothermic reaction which forms the second phase is initiated. A solvent assisted reaction ensues, resulting in the extremely rapid formation and dispersion of finely divided particles of the second phase material in the matrix material.

What is common to each of the above-described patents is the requirement of pressing, molding or otherwise forming a permeable mass of filler material into some desired shape, followed by infiltration of the shaped mass with molten metal to produce a shaped composite body. The present invention addresses and overcomes many of the limitations associated with shaping filler materials.

DESCRIPTION OF COMMONLY OWNED PATENTS

Commonly owned U.S. Patent No. 4,935,055 to Aghajanian et al. teaches utilizing at least one of titanium diboride and/or a graphite coating such as graphite paper as a barrier means for making net or near net shaped metal matrix composite bodies. In particular, a permeable mass of ceramic filler material is formed such as to have at least one defined surface boundary and at least a portion of this surface boundary surrounded by or superimposed with a barrier means. Upon contact of the permeable mass with a molten matrix metal, under spontaneous infiltration conditions the molten matrix metal will infiltrate the filler material in a direction toward the barrier means. The barrier means prevents, inhibits or terminates migration of molten matrix metal beyond the surface boundary defined by the barrier means.

Commonly owned U.S. Patent No. 5,553,657 to Aghajanian et al. teaches the use of a gating means in combination with a spontaneous infiltration process to produce a metal matrix composite body. In particular, the gating means is provided to control or limit the areal contact between the supply of molten matrix metal and the filler material or preform to be infiltrated. The use of a gating means ameliorates the tendency of a formed metal matrix composite body to warp upon cooling from the processing temperature due to differences in thermal expansion coefficients between the metal matrix composite body and the attached residual carcass of uninfiltrated matrix metal. The gating means may also function to reduce the degree of bonding between the formed metal matrix composite body and the carcass of matrix metal, thereby facilitating separation of one from the other. Graphite is an example of an effective gating means for use in producing aluminum matrix composites, and is useful in graphite paper form and as a permeable particulate layer.

Commonly owned U.S. Patent No. 5,280,819 to Newkirk et al. teaches a number of different alternatives for making thin metal matrix composite bodies. In one embodiment of the invention, the filler material may be coated onto the body of matrix metal. Alternatively, the filler material may be shaped by a tape casting, slip casting, etc. to provide a thin preform. Still further, a plurality of thin metal matrix composite bodies may be produced simultaneously by stacking or laminating a plurality of thin preforms between alternating layers of barrier material and simultaneously infiltrating the set of preforms from a common source of matrix metal, by way of, for example, an exposed edge of each preform.

DISCLOSURE OF THE INVENTION

The present invention relates to a technique for producing a metal matrix composite (MMC) body. In particular, the present technique produces the reinforcement phase (e.g., filler material) for the MMC body in situ.

The present invention has as an objective the production of metal matrix composite bodies without the requirement of providing a permeable mass of filler material to the infiltration apparatus.

The present invention also has as an objective the fabrication of thin metal matrix composite bodies of net or near net shape.

The present invention furthermore has as an objective the ability to produce metal matrix composite bodies ranging in thickness from a fraction of a millimeter to several millimeters.

5 The present invention also has as an objective the recycling or reuse of the supporting surface or substrate against which the metal matrix composite body is formed without undue refurbishing of such surface.

10 According to the present improved process for making metal matrix composite bodies, a permeable infiltration enhancer material is applied by any suitable technique to a substrate or surface. A preferred substrate or surface is one which is substantially nonreactive with respect to the infiltration enhancer material under the processing conditions. In a preferred embodiment, the infiltration enhancer material is applied to the substrate or surface by a chemical vapor deposition (CVD) technique. For example, an infiltration enhancer precursor vapor may be chemically reacted with an infiltrating atmosphere to produce solid infiltration enhancer material on a surface. Next, at least a portion of the infiltration enhancer material is contacted to a molten matrix metal. Under the local processing conditions, the molten matrix metal is able to spontaneously infiltrate the permeable infiltration enhancer material. Concurrent with the infiltration at least a portion of the infiltration enhancer material is chemically reduced by the molten matrix metal to yield a ceramic material. The infiltration of molten matrix metal and chemical conversion of at least a portion of the infiltration enhancer material effectively results in a metal matrix composite material featuring the formed ceramic material embedded by matrix metal.

25 The metal matrix composite material having the reinforcement phase or filler material formed in situ exhibits great compositional flexibility. Specifically, the amount (e.g., volumetric loading) and morphology of the in situ formed filler material may be controlled by varying the processing conditions. Further, one or more additional filler materials may be incorporated into the metal matrix composite body by incorporating such ceramic material into the infiltration enhancer material or otherwise applying such ceramic material to the supporting surface prior to contact and infiltration by the molten matrix metal.

30 If desired, it is possible to increase the thickness of the present MMC bodies without resorting to additions of filler material to the infiltration apparatus or lay-up. In

particular, a fugitive material may be incorporated into the infiltration enhancer material or may be applied to the supporting surface prior to contact and infiltration by molten matrix metal. The fugitive material is one which can be caused to dissolve, volatilize, corrode or otherwise change form or disappear prior to completion of the MMC formation process. An example of a fugitive material in the context of the present invention is one which can be dissolved by the molten matrix metal, e.g., copper by molten aluminum.

The supporting surface or substrate against which the infiltration enhancer material is applied preferably is substantially nonreactive with respect to the infiltration enhancer material. For the embodiment in which the infiltration enhancer material is formed by chemical vapor deposition, the substrate or surface material ideally is substantially nonreactive with the infiltration enhancer precursor material. Magnesium oxide and carbon are examples of materials which meet the above criteria under the local processing conditions. Further, at least one surface of the supporting surface or substrate should be capable of being formed to inversely replicate the desired exterior shape of the MMC body. Moreover, the supporting surface or substrate should possess barrier material characteristics, either intrinsically or upon further processing, such as application of a coating thereto. Still further, the supporting surface or substrate should be readily detachable or otherwise rendered removable from the adjacent formed MMC body.

A problem which must be addressed when producing metal matrix composite bodies by an infiltration process is removal of the residual carcass of matrix metal once the infiltration process is complete. The present invention contemplates that the matrix metal will be introduced or fed or contacted with the infiltration enhancer material from a small region of the latter such as an edge. In a preferred embodiment, the body of molten matrix metal is maintained at a lower elevation than most or all of the infiltration enhancer material thereby forcing the infiltration of molten matrix metal into the infiltration enhancer material to proceed against gravity. In this way the body of matrix metal contacts only a small segment or portion of the formed metal matrix composite body, thereby facilitating detachment and removal of the carcass of matrix metal from the formed metal matrix composite body once infiltration is substantially complete and the infiltrated matrix metal has solidified.

The present invention is particularly useful and desirable for producing thin metal matrix composite bodies. It has been discovered, however, that once a thin MMC body has been produced by the above described technique, the process may be substantially repeated to apply an additional layer of MMC material upon that which has been formed previously. Specifically, once a metal matrix composite layer has been formed onto the supporting substrate or surface, additional infiltration enhancer material may be applied to at least a portion of at least one surface of the formed MMC body (either directly or through deposition from the vapor phase). Contact of the newly applied infiltration enhancer layer with a source of molten matrix metal can lead to spontaneous infiltration of the molten matrix metal into the new infiltration enhancer layer to form additional MMC material.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1 is a cross-sectional schematic view illustrating the use of a fugitive material in connection with the present invention;

Figures 2A-2C are cross-sectional schematic views of a setup which illustrate the sequence of events which are believed to occur in producing thin, shaped MMC bodies in accordance with the present invention;

Figure 3 is a cross-sectional schematic view of a lay-up substantially in accordance with Example 1 for producing a metal matrix composite body having in situ produced reinforcement;

Figures 4A and 4B are optical photomicrographs at magnifications of about 425x and 1060x, respectively, of a polished cross-section of the thin-walled structure produced in accordance with Example 1;

Figure 5 is an approximately 400x magnification optical photomicrograph of a polished cross-section of a thin metal matrix composite structure produced in accordance with Example 2;

Figure 6 is a cross-sectional schematic view of a lay-up used to produce a near net shape metal matrix composite structure in accordance with Example 3; and

Figure 7 is a photograph of the near net shape metal matrix composite article produced in accordance with Example 3.

DEFINITIONS

“Aluminum”, as used herein, means and includes essentially pure metal (e.g., a relatively pure, commercially available unalloyed aluminum) or other grades of metal and metal alloys such as the commercially available metals having impurities and/or alloying constituents such as iron, silicon, copper, magnesium, manganese, chromium, zinc, etc., therein. An aluminum alloy for purposes of this definition is an alloy or intermetallic compound in which aluminum is the major constituent.

“Balance Non-Oxidizing Gas”, as used herein, means that any gas present in addition to the primary gas comprising the infiltrating atmosphere, is either an inert gas or a reducing gas which is substantially non-reactive with the matrix metal under the process conditions. Any oxidizing gas which may be present as an impurity in the gas(es) used should be insufficient to oxidize the matrix metal to any substantial extent under the process conditions.

“Barrier” or “Barrier Means”, as used herein, means any suitable means which interferes, inhibits, prevents or terminates the migration, movement, or the like, of molten matrix metal beyond a surface boundary of a permeable mass comprising infiltration enhancer material, where such surface boundary is defined by said barrier means. Suitable barrier means may be any such material, compound, element, composition, or the like, which, under the process conditions, maintains some integrity and is not substantially volatile (i.e., the barrier material does not volatilize to such an extent that it is rendered non-functional as a barrier).

Further, suitable “barrier means” includes materials which are substantially non-wettable by the migrating molten matrix metal under the process conditions employed. A barrier of this type appears to exhibit substantially little or no affinity for the molten matrix metal, and movement beyond the defined surface boundary of the permeable mass is prevented or inhibited by the barrier means. The barrier reduces any final machining or grinding that may be required and defines at least a portion of the surface of the resulting metal matrix composite product. The barrier may in certain cases be permeable or porous, or rendered permeable by, for example, drilling holes or puncturing the barrier, to permit gas to contact the molten matrix metal.

“Carcass” or “Carcass of Matrix Metal”, as used herein, refers to any of the original body of matrix metal remaining which has not been consumed during formation

of the metal matrix composite body, and typically, if allowed to cool, remains in at least partial contact with the metal matrix composite body which has been formed. It should be understood that the carcass may also include a second or foreign metal therein.

5 “Filler”, as used herein, refers to the reinforcement phase of a metal matrix composite, and is intended to include either single constituents or mixtures of constituents which are substantially non-reactive with and/or of limited solubility in the matrix metal and may be single or multi-phase.

10 “Infiltrating Atmosphere”, as used herein, means that atmosphere which is present which interacts with the matrix metal and/or infiltration enhancer precursor and/or infiltration enhancer and permits or enhances spontaneous infiltration of the matrix metal to occur.

15 “Infiltration Enhancer”, as used herein, means a material which promotes or assists in the spontaneous infiltration of a matrix metal. An infiltration enhancer may be formed from, for example, a reaction of an infiltration enhancer precursor with an infiltrating atmosphere to form a solid, liquid or gaseous species. Moreover, the infiltration enhancer may be supplied directly to at least one of the matrix metal and/or infiltrating atmosphere and functions in a substantially similar manner to an infiltration enhancer which has formed as a reaction between an infiltration enhancer precursor and another species. Ultimately, at least during the spontaneous infiltration, the infiltration enhancer should be located on at least a portion of the substantially non-reactive surface or substrate to achieve spontaneous infiltration.

20 “Infiltration Enhancer Precursor” or “Precursor to the Infiltration Enhancer”, as used herein, means a material which when used in combination with the matrix metal and/or infiltrating atmosphere forms an infiltration enhancer which induces or assists the matrix metal to spontaneously infiltrate the permeable material comprising the infiltration enhancer. Without wishing to be bound by any particular theory or explanation, it appears as though it may be necessary for the precursor to the infiltration enhancer to be capable of being positioned, located or transportable to a location which permits the infiltration enhancer precursor to interact with the infiltrating atmosphere and/or the matrix metal. An infiltration enhancer precursor may be supplied from a source alloyed with the matrix metal, or from a source external to the matrix metal.

“In-situ Filler” or “In-situ Reinforcement”, as used herein, refers to a filler material which instead of being provided to an infiltration apparatus or spontaneous infiltration system, forms during the infiltration process itself.

“Matrix Metal” or “Matrix Metal Alloy”, as used herein, means that metal which is utilized to form a metal matrix composite (e.g., before infiltration) and/or that metal which is intermingled with a reinforcement material to form a metal matrix composite body (e.g., after infiltration). When a specified metal is mentioned as the matrix metal, it should be understood that such matrix metal includes that metal as an essentially pure metal, a commercially available metal having impurities and/or alloying constituents therein, an intermetallic compound or an alloy in which that metal is the major or predominant constituent.

“Matrix Metal/Infiltration Enhancer Precursor/Infiltrating Atmosphere System” or “Spontaneous System”, as used herein, refers to that combination of materials which exhibit spontaneous infiltration into a permeable mass comprising an infiltration enhancer. It should be understood that whenever a “/” appears between an exemplary matrix metal, infiltration enhancer precursor and infiltrating atmosphere that the “/” is used to designate a system or combination of materials which, when combined in a particular manner, exhibits spontaneous infiltration into a permeable mass.

“Metal Matrix Composite” or “MMC”, as used herein, means a material comprising a two- or three-dimensionally interconnected alloy or matrix metal which has embedded a reinforcement material. The matrix metal may include various alloying elements to provide specifically desired mechanical and physical properties in the resulting composite.

A Metal “Different” from the Matrix Metal means a metal which does not contain, as a primary constituent, the same metal as the matrix metal (e.g., if the primary constituent of the matrix metal is aluminum, the “different” metal could have a primary constituent of, for example, nickel).

“Reservoir”, as used herein, means a separate body of matrix metal positioned relative to a permeable mass so that, when the metal is molten, it may flow to replenish, or in some cases to initially provide and subsequently replenish, that portion, segment or source of matrix metal which is in contact with a filler material or infiltration enhancer.

“Spontaneous Infiltration”, as used herein, means the infiltration of matrix metal into the permeable mass of infiltration enhancer, reinforcement material or the like occurs without requirement for the application of pressure or vacuum (whether externally applied or internally created).

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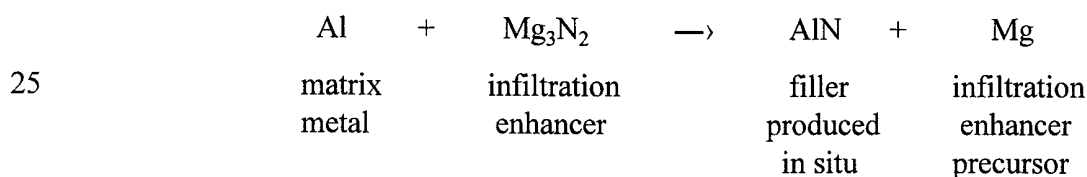
MODES FOR CARRYING OUT THE INVENTION

According to the present improved process for making metal matrix composite bodies, an infiltration enhancer material is applied by any suitable technique to a substrate or surface. A preferred substrate or surface is one which is substantially nonreactive with respect to the infiltration enhancer material under the processing conditions. In a preferred embodiment, the infiltration enhancer material is applied to the substrate or surface by a chemical vapor deposition (CVD) technique. For example, an infiltration enhancer precursor vapor may be chemically reacted with an infiltrating atmosphere to produce solid infiltration enhancer material on a surface. Next, at least a portion of the infiltration enhancer material is contacted to a molten matrix metal. Under the local processing conditions, the molten matrix metal is able to spontaneously infiltrate the permeable infiltration enhancer material. Concurrent (or at least contemporaneous) with the infiltration at least a portion of the infiltration enhancer material chemically reacts with the molten matrix metal to yield a ceramic material. The infiltration of molten matrix metal and chemical conversion of at least a portion of the infiltration enhancer material effectively results in a metal matrix composite material featuring the formed ceramic material embedded by matrix metal.

The metal matrix composite material having the reinforcement phase or filler material formed in situ exhibits great compositional flexibility. Specifically, the amount (e.g., volumetric loading) and morphology of the in situ formed filler material may be controlled according to the way in which the infiltration enhancer material is deposited or applied to its supporting surface. Further, one or more additional filler materials may be incorporated into the metal matrix composite body by incorporating such ceramic material into the infiltration enhancer material or otherwise applying such ceramic material to the supporting surface prior to contact and infiltration by the molten matrix metal. In this way, the applied filler material can tailor the composition of the MMC body, as well as increase the thickness of MMC material.

Still further, it is possible to increase the thickness of the MMC body without resorting to a material which becomes part of the reinforcement phase of the MMC body so produced. In particular, a fugitive material may be incorporated into the infiltration enhancer material or may be applied to the supporting surface prior to contact and infiltration by molten matrix metal. The fugitive material is one which can be cause to dissolve, volatilize, corrode or otherwise change form or disappear prior to completion of the MMC formation process. An example of a fugitive material in the context of the present invention is the metal copper.

Specifically, and with reference to Figure 1, a fugitive material 11 is applied to the supporting surface or substrate 13 to a desired thickness. In a preferred embodiment, the fugitive material comprises a metal in particulate form. In a particularly preferred embodiment the fugitive metal comprises a metal having a melting point higher than the melting point of the infiltrating matrix metal. For example, if the matrix metal comprises aluminum, the fugitive metal could comprise particulate copper. Under spontaneous infiltration conditions (e.g., in the presence of an infiltration enhancer or infiltrating atmosphere/infiltration enhancer precursor combination), infiltration enhancer 15 is deposited on at least a portion of the surfaces presented by the fugitive metal 11. The molten matrix metal 17 comprising aluminum infiltrates through the fugitive metal layer 11 comprising particulate copper. Simultaneous or at least contemporaneous with the infiltration, the matrix metal chemically reduces the infiltration enhancer to infiltration enhancer precursor 19, and some matrix metal is oxidized to form aluminum nitride. This redox reaction can be written as:



The copper particulate alloys with the infiltrated matrix metal. What is formed is a metal matrix composite surface layer adjoining the supporting surface or substrate. The MMC material comprises aluminum, copper, magnesium as the matrix phase and aluminum nitride as the filler material reinforcement 16. Using such particulate metals as fugitive materials, MMC surface layers up to about 3 millimeters have been produced. Further,

when the fugitive metal comprises a metal having a chemical composition which is different from that of the matrix metal, the possibility of "metal phase tailoring" exists. Such metal phase tailoring affords the opportunity of further modifying or engineering the properties of at least the surface layer through control of the extent of alloying or diffusion of the fugitive metal into the matrix metal, or of the extent of reaction of the fugitive metal with the matrix metal (e.g., to form intermetallics). Commonly owned U.S. Patents 5,518,061 and 5,287,911 provide a detailed discussion of metal phase tailoring, particularly as applied to metal matrix composites. The entire disclosures of these two commonly owned U.S. patents are incorporated herein by reference.

The supporting surface or substrate against which the infiltration enhancer material is applied (or formed) preferably is substantially nonreactive with respect to the infiltration enhancer material and the matrix metal. For the embodiment in which the infiltration enhancer material is formed by chemical vapor deposition, the substrate or surface material ideally is substantially nonreactive also with the infiltration enhancer precursor material. Magnesium oxide and carbon are examples of materials which meet the above criteria under the local processing conditions. In a particularly preferred embodiment, the surface or substrate comprises a barrier material, or at least functions as a barrier. In other words, the matrix metal is not permitted to infiltrate into the supporting surface or substrate. Further, the preferred supporting surface or substrate is one which is readily detachable or otherwise removable from the adjacent formed MMC body. Still further, the ideal supporting surface or substrate is one which is capable of being re-used repeatedly in subsequent runs.

Suitable barriers particularly useful for aluminum matrix alloys are those containing carbon, especially the crystalline allotropic form of carbon known as graphite. Graphite is essentially non-wettable by the molten aluminum alloy under the described process conditions. A particular preferred graphite is a graphite foil product that is sold under the trademark Grafoil®, registered to Union Carbide. This graphite foil is resistant to heat and is chemically inert. Grafoil® graphite foil is flexible, compatible, conformable and resilient.

Another form of graphite which functions as a barrier material and which has particular utility in the context of the present application as a substrate or mold surface is bulk graphite. This form of graphite is readily machinable, self-supporting, and

maintains dimensional stability. Furthermore, it is contemplated that bulk graphite, when used as a mold surface, may be reusable for a number of MMC fabrication runs before having to be discarded. Bulk graphite is commercially available in a great number of grades depending upon purity, grain size and porosity. Although some of the grades are fairly porous, it is not expected that under the process conditions that substantial quantities of infiltration atmosphere can permeate the wall thicknesses of the bulk graphite material which are contemplated. Accordingly, where a bulk graphite mold surface completely encloses the source of matrix metal and/or infiltration enhancer precursor material, it may be necessary to provide one or more vent holes in a bulk graphite mold surface to permit ingress/egress of infiltrating atmosphere to the otherwise enclosed interior space.

Graphite in the form of fine particles also possesses desirable barrier properties. This form of graphite is particularly attractive because it can be mixed with a liquid vehicle to form a slurry or suspension, which can be painted or sprayed or otherwise coated onto a surface of a refractory material to provide barrier properties. A particularly preferred form of such a graphite slurry is that of colloidal graphite which is available commercially under the tradenames "Dylon" or "Dag", belonging to Dylon Industries and Acheson Colloids Company, respectively. An additional attribute of such particulate graphite is its property of acting as a release layer. Specifically, a mold surface which may function as a barrier material but which may or may not possess release properties may be coated with particulate graphite (which may be provided in slurry or suspension form), and dried. Upon formation of a metal matrix composite material adjacent to this coated surface, the formed MMC body may be removed from the barrier or mold surface with a minimum of effort. Then, any remaining residual graphite material adhered to the MMC body or to the barrier material may be physically removed as, for example, by grit blasting. Seldom are abrasive grits required to remove such residual graphite release material. Instead, frequently it is entirely satisfactory to impact such a surface with walnut shells using forced air or low-pressure compressed air to dislodge the adhered graphite particles.

A problem which must be addressed when producing metal matrix composite bodies by an infiltration process is removal of the residual carcass of matrix metal once the infiltration process is complete. While not mandatory, the present invention

contemplates that the matrix metal is introduced or contacted with the infiltration enhancer material from a small region of the latter such as an edge of a coating layer. In a preferred embodiment, the body of molten matrix metal is maintained at a lower elevation than most or all of the infiltration enhancer material, thereby forcing the infiltration of molten matrix metal into the infiltration enhancer material to proceed against gravity. In this "countergravitational" way the body of matrix metal contacts only a small segment or portion of the formed metal matrix composite body, thereby facilitating detachment and removal of the carcass of matrix metal from the formed metal matrix composite body once infiltration is substantially complete and the infiltrated matrix metal has solidified.

Figures 2A-2C illustrate a sequence of events which may occur in producing a thin MMC body have in-situ produced reinforcement. Specifically, in Figure 2A refractory boat 22 supports substrate material 24 and houses matrix metal 26. Matrix metal contains a source for infiltration enhancer precursor. In Figure 2B matrix metal 26 is molten and in the presence of infiltrating atmosphere 28 causes infiltration enhancer 23 to be deposited on the exposed surfaces of refractory boat 22 and substrate 24 as a powdery, porous coating layer. Molten matrix metal spontaneously infiltrates infiltration enhancer layer 23 and chemically reacts with the infiltration enhancer material to form a solid reinforcement phase, thereby producing a metal matrix composite body 25. With reference to Figure 2C, upon solidification of molten matrix metal 26, the setup may be disassembled and the thin metal matrix composite body 25 recovered. Note that some residual metal matrix composite material 25 remains adhered to the surface of refractory boat 22.

The present invention is particularly useful and desirable for producing thin metal matrix composite bodies. It has been discovered, however, that once a thin MMC body has been produced by the above described technique, the process may be substantially repeated to apply an additional layer of MMC material upon the previously formed layer. Specifically, once a metal matrix composite layer has been formed onto the supporting substrate or surface, additional infiltration enhancer material may be applied to at least a portion of at least one surface of the formed MMC body (either directly or through deposition from the vapor phase). Optionally, one or more filler materials and/or fugitive materials may also be supplied. Contact of the newly applied infiltration enhancer layer

with a source of molten matrix metal causes spontaneous infiltration of the molten matrix metal into the new infiltration enhancer layer to form additional MMC material.

The source of the molten matrix metal is largely immaterial in forming the new MMC layer. The source of matrix metal for the new MMC body can be freshly supplied
5 matrix metal, the residual uninfiltrated matrix metal from the previous run, or it can be that matrix metal which already has infiltrated a previous layer of infiltration enhancer material. In the event of the latter, porosity or voids may form in the existing MMC body unless a source of molten matrix metal is in fluid communication with this previously formed MMC body as a reservoir to replace that matrix metal which is lost
10 from this MMC body in infiltrating the newly applied infiltration enhancer material to form the new, additional MMC body.

Although the present patent disclosure predominantly addresses the embodiment in which the infiltration enhancer material is deposited onto a supporting surface or substrate by the chemical reaction of two vapor species (referred to herein as a "chemical
15 vapor deposition process"), many other techniques for applying a permeable layer of solid infiltration enhancer material to this substrate is encompassed by the present disclosure. For example, a particulate infiltration enhancer material may be applied to the substrate by painting, dip coating or spray coating the particulate in slurry form onto the substrate, followed by drying or otherwise volatilizing the liquid carrier material. In
20 another embodiment, solid infiltration enhancer material is deposited onto the substrate from the vapor phase, e.g. by physical vapor deposition. Alternatively, a permeable layer of solid infiltration enhancer precursor material may be applied to a substrate (again, for example, in slurry form), followed by chemical conversion to an infiltration enhancer by chemical reaction with an infiltrating atmosphere, or a reactive constituent thereof. Thus,
25 for example, if the infiltration enhancer precursor were magnesium particulate, a slurry could be made using an organic solvent, and the magnesium particulate slurry coated onto a mold surface. After removing the organic solvent, the magnesium particulate layer could be reacted with a nitrogen-containing gas (e.g., molecular nitrogen or ammonia) to convert at least some of the magnesium particulate to magnesium nitride.

30 For the embodiment in which the infiltration enhancer material is formed by chemical vapor deposition from infiltrating atmosphere and infiltration enhancer precursor reactants, it may be desirable that the supporting surface or substrate take the

form of a shell or mold or housing which is capable of enclosing or otherwise substantially confining the infiltration enhancer precursor vapor. The infiltration enhancer precursor vapor thus is used more efficiently and economically, with the result that the infiltration enhancer layer can be deposited relatively uniformly and quickly. In this way a porous magnesium nitride infiltration enhancer layer, for example, has been deposited by chemical vapor deposition to a thickness on the order of 1 millimeter in the span of a few hours.

Chemical vapor deposition techniques are frequently employed to deposit dense coatings onto substrates. The chemical vapor deposition parameters of the present invention are such that the infiltration enhancer material which is deposited on a substrate is not a dense layer, but instead exhibits considerable porosity, much of which is interconnected. Permeability to the molten matrix metal is believed to be important in yielding a material which can be considered to be a metal matrix composite material.

Deposition of a permeable magnesium nitride infiltration enhancer layer can be accomplished, for example, at a temperature of about 900°C under atmospheric pressure by reacting commercially pure nitrogen with a source of magnesium. The magnesium source may be provided separately or may be alloyed in the matrix metal. Magnesium metal possesses a fairly high vapor pressure, with the result that useful quantities of magnesium nitride can be produced at temperatures of around 600°C or possibly somewhat even lower. An artisan of ordinary skill will readily appreciate that desirable permeable magnesium nitride infiltration enhancer deposits can be produced over a considerable range of temperature and pressure, at least when the nitrogen source is molecular nitrogen gas.

Without wishing to be bound by any particular theory or explanation, when an infiltration enhancer precursor is utilized in combination with at least one of the matrix metal, and/or infiltrating atmosphere, the infiltration enhancer precursor may react to form an infiltration enhancer which induces or assists molten matrix metal to spontaneously infiltrate the thus formed infiltration enhancer material. Moreover, it appears as though it may be necessary for the precursor to the infiltration enhancer to be capable of being positioned, located or transportable to a location which permits the infiltration enhancer precursor to interact with at least one of the infiltrating atmosphere and/or molten matrix metal. For example, in some matrix metal/infiltration enhancer

precursor/infiltrating atmosphere systems, it is desirable for the infiltration enhancer precursor to volatilize at, near, or in some cases, even somewhat above the temperature at which the matrix metal becomes molten. Such volatilization may lead to a reaction of the infiltration enhancer precursor with the infiltrating atmosphere to form a solid, liquid or gaseous infiltration enhancer on at least a portion of the mold surface or barrier material, thereby enhancing wetting by the matrix metal.

Thus, for example, if an infiltration enhancer precursor was included or combined with, at least at some point during the process, molten matrix metal, it is possible that the infiltration enhancer precursor could volatilize from the molten matrix metal and react with the infiltrating atmosphere. Such reaction could result in the formation of a solid infiltration enhancer species, if such solid species was stable at the infiltration temperature, said solid species being capable of being deposited on at least a portion of a mold surface or a barrier material, as, for example, a coating. If such a solid species of infiltration enhancer were formed, molten matrix metal may have a tendency to react (e.g., the molten matrix metal may reduce the formed infiltration enhancer) such that infiltration enhancer precursor is again produced. This newly re-formed infiltration enhancer precursor then may become associated with (e.g., dissolved in or alloyed with) the molten matrix metal. Accordingly, additional infiltration enhancer precursor may then be available to volatilize and react with another species (e.g., the infiltrating atmosphere) and again form a similar solid species.

In a preferred embodiment of the invention, it is possible that the infiltration enhancer precursor can be at least partially reacted with the infiltrating atmosphere such that the infiltration enhancer can be formed on at least a portion of the mold surface prior to or substantially contiguous with contacting the mold surface with the matrix metal (e.g., if magnesium was the infiltration enhancer precursor and nitrogen was the infiltrating atmosphere, the infiltration enhancer could be magnesium nitride which would be located on at least a portion of the mold whose shape or surface is to be replicated).

This patent disclosure primarily discusses the aluminum/magnesium/nitrogen spontaneous infiltration system. Additionally, spontaneous infiltration has been observed in the aluminum/strontium/nitrogen system, the aluminum/calcium/nitrogen system, and the aluminum/ zinc/oxygen system. Accordingly, it should be understood

that other matrix metal/infiltration enhancer precursor/infiltrating atmosphere systems may exist and may behave in a similar manner.

For aluminum infiltration according to the present invention, a certain amount of aluminum nitride forms in the resulting MMC body above and beyond that which is due to chemical reaction of the infiltration enhancer material, due to direct nitridation of the aluminum matrix metal. At low processing temperatures, a negligible amount of this direct nitridation occurs. However, as the upper end of the temperature range is approached, direct nitridation of the matrix metal is more likely to occur. Thus, the amount of the nitride phase in the metal matrix can be controlled by varying the processing temperature at which infiltration occurs. The specific process temperature at which nitride formation becomes more pronounced also varies with such factors as the matrix aluminum alloy used and the nitrogen concentration of the infiltrating atmosphere.

Further, the constituency of the matrix metal within the metal matrix composite and the number and size of defects, for example, porosity, may be modified by controlling the cooling rate of the metal matrix composite. For example, the metal matrix composite may be directionally solidified by any number of techniques including: placing the container holding the metal matrix composite upon a chill plate; and/or selectively placing insulating materials about the container. Further, the constituency of the metal matrix may be modified after formation of the metal matrix composite. For example, exposure of the formed metal matrix composite to a heat treatment may improve the tensile strength of the metal matrix composite. (The standard test for tensile strength is ASTM-D3552-77 (reapproved 1982).)

For example, a desirable heat treatment for a metal matrix composite containing a 520.0 aluminum alloy as the matrix metal may comprise heating the metal matrix composite to an elevated temperature, for example, to about 430°C, which is maintained for an extended period (e.g., 18-20 hours). The metal matrix may then be quenched in boiling water at about 100°C for about 20 seconds (i.e., a T-4 heat treatment) which can temper or improve the ability of the composite to withstand tensile stresses.

Moreover, it is possible to use a reservoir of matrix metal to assure complete infiltration and conversion of the infiltration enhancer and/or to supply a second metal which has a different composition from the first source of matrix metal. Specifically, in

some cases it may be desirable to utilize a matrix metal in the reservoir which differs in composition from the first source of matrix metal. For example, if an aluminum alloy is used as the first source of matrix metal, then virtually any other metal or metal alloy which was molten at the processing temperature could be used as the reservoir metal.

5 Molten metals frequently are very miscible with each other which would result in the reservoir metal mixing with the first source of matrix metal so long as an adequate amount of time is given for the mixing to occur. Thus, by using a reservoir metal which is different in composition from the first source of matrix metal, it is possible to tailor the properties of the metal matrix to meet various operating requirements and thus tailor the
10 properties of the metal matrix composite.

The following examples further illustrate the present invention.

Example 1

This example demonstrates the production of a thin metal matrix composite structure. Moreover, this example demonstrates the production of a composite material
15 comprising aluminum nitride reinforcing an aluminum alloy matrix wherein the aluminum nitride phase is formed in situ.

Referring to Figure 3, a graphite boat 31 having an internal cavity measuring about 6 inches (152 mm) square by about 2 inches (51 mm) deep was filled to a depth of about 1 inch (25 mm) with a particulate bedding material admixture 33 comprising by
20 weight about 15% grade F-69 glass frit (Fusion Ceramics, Inc., Carrollton, Ohio) and the balance 90 grit (216 microns average particle diameter), 38 Alundum® aluminum oxide particulate (Norton-St. Gobain, Worcester, Massachusetts). A body of matrix metal 35 measuring about 1 inch x 2 inches x 3 inches (25 mm x 51 mm x 76 mm) and having a mass of about 353 grams and comprising by weight about 10% magnesium, balance
25 aluminum, was centered over the particulate admixture bedding with the long end of the ingot oriented vertically. The bedding material comprising this glass frit is substantially non-infiltratable by molten matrix metal under the present processing conditions.

Next a colloidal graphite release layer 37 was coated onto the interior surface of a graphite crucible 39 (grade ATJ, Poco Graphite Inc., Decatur, Texas) measuring about 4
30 inches (102 mm) in diameter by about 3.5 inches (89 mm) deep and having a wall thickness of about 3/16 inch (4.8 mm). This bulk graphite crucible 39 also featured a 1/16 inch (1.6 mm) diameter vent hole 36. Specifically, the interior was painted with

Dylon CW colloidal graphite (Dylon Industries Inc., Cleveland, Ohio) followed by drying at a temperature of about 80°C. Then this coated surface was then further painted with Dag 154 colloidal graphite (Acheson Colloids Company, Port Huron, Michigan). After drying this second coating at about 80°C, the coated crucible was then centered
5 over the ingot of matrix metal to complete the lay-up.

The lay-up was then placed into the retort of a resistance-heated controlled atmosphere furnace at substantially ambient temperature and pressure. After sealing the retort, the heating chamber within the retort was evacuated and backfilled with commercially pure nitrogen gas, after which a steady flow rate of the nitrogen gas of
10 about 20 liters per minute (at atmospheric pressure) was maintained. The contents of the retort were then heated to a temperature of about 900°C at a rate of about 200°C per hour. After maintaining a temperature of about 900°C for about one hour, the temperature was permitted to fall at the furnace's natural or intrinsic cooling rate to a temperature of about 800°C. After maintaining a temperature of about 800°C for about
15 one hour, the retort was cooled below 50°C at the furnace cooling rate.

Disassembly of the lay-up showed at least the onset of formation of a thin-walled structure. Specifically, a small amount of a thin metallic-appearing layer was observed to have "grown" partway up the wall of the coated mold on one side of the lay-up. Further, a yellowish material (suggesting magnesium nitride formation) was observed
20 extending a slight ways up the mold wall above the metal-containing growth film.

The run was repeated using new colloidal graphite coatings on the bulk graphite crucible, a 352 gram matrix metal ingot, and a temperature soak of about 1.5 hours at 900°C. This time, the thin-walled structure was more extensive, but still not contacting all internal surfaces of the mold. Specifically, a thin-walled structure extended about 2-3
25 inches (51-76 mm) up from the outer edge of the solidified carcass of matrix metal. The formed thin-walled structure fairly accurately replicated those surfaces of the crucible which it adjoined.

Figures 4A and 4B are optical photomicrographs of a polished cross-section of the formed material taken at a magnification of about 425x and about 1,060x,
30 respectively. The cross-section shows a total wall thickness of about 120 microns with an approximately 14-15 micron thick zone of metal matrix composite material 43 sandwiched between layers of substantially unreinforced matrix metal 45. The metal

matrix composite layer comprises what appears to be a ceramic phase embedded by matrix metal, the ceramic phase being produced in situ. The ceramic phase was reasoned to be aluminum nitride. The in-situ produced ceramic phase features at least an acicular and possibly also an equiaxed morphology with the acicular needles being on the order of up to 6 microns in length by about 1 micron in cross-section.

Thus, this example demonstrates that a thin-walled metal matrix composite structure can be produced which replicates at least a portion of an interior surface of a mold and which features the ceramic reinforcement of the metal matrix composite material being produced in situ.

Example 2

This example also demonstrates the production of a thin metal matrix composite body whose reinforcement is produced in situ.

The lay-up for the present example was similar to the lay-up of Example 1, except that a graphite foil box was used in place of the coated graphite mold. Specifically, a single sheet of Grafoil® graphite foil material (Union Carbide Company, Carbon Products Division, Cleveland, Ohio) measuring about 15 mils (0.4 mm) thick was formed into a five-sided box measuring about 4 inches (102 mm) square by about 2 inches (51 mm) deep by making strategically placed cuts and folds into a single sheet of the graphite foil material and stapling the box as needed to render the box self-supporting. The open end of the box was then placed over the body of matrix metal onto the surface of the particulate bedding material admixture to form the lay-up.

The lay-up was then thermally processed in substantially the same manner as the lay-up in Example 1, except that the lay-up was maintained at a temperature of about 900°C for about one hour, and maintained at the temperature of about 800°C for about two hours. It was not necessary to intentionally place a vent hole into the graphite foil box because the junctions and seams in the graphite foil box were sufficiently "leaky" as to permit the commercially pure nitrogen atmosphere to gain access to the interior of the box.

Disassembly of the lay-up following the run (removal of the graphite foiled box) revealed that a metal matrix composite body had formed on at least some of the interior surfaces of the graphite foil box.

A section of the wall of the formed body was mounted, polished and examined in the optical microscope. The cross-section presented by a 50x magnification photomicrograph was used to determine the 200 to 250 micron wall thickness of the formed MMC body. Figure 5 is an approximately 400x magnification photomicrograph of the polished cross-section of the wall, and reveals an acicular ceramic phase 53 embedded by the matrix metal 55. The acicular phase ranges in length up to about 70 microns, has a cross-section on the order of 1 micron, and displays a slight curvature. This phase was inferred as comprising aluminum nitride.

Quantitative image analysis was performed on the sample. A total of about 68 frames corresponding to a total area analyzed of about 1.2 square millimeters revealed the following areal fractions of material: ceramic, $27 \pm 7\%$; metal, $73 \pm 7\%$; pores, $0.1 \pm 0.4\%$. The volume fractions of the various phases should be about the same as the areal fractions.

Example 3

This example further demonstrates the production of a thin metal matrix composite body whose reinforcement is produced in situ. This example also demonstrates near net shape replication.

The production conditions of the present example were similar to those in Example 2; however, the matrix metal body 65 of the present example comprised by weight about 8 percent magnesium and the balance aluminum. Also, the body of matrix metal was housed in its own five-sided graphite foil box 67, as show in Figure 6, instead of directly contacting the bedding material admixture 33. Furthermore, the lay-up was thermally processed differently, specifically, by heating from about 20°C to a temperature of about 200°C at a rate of about 100°C per hour. After maintaining a temperature of about 200°C for about 5 hours, the temperature of the furnace and its contents was increased to a temperature of about 900°C at a rate of about 100°C per hour. After maintaining a temperature of about 900°C for about 6 hours, the temperature was decreased to a temperature of about 700°C at a rate of about 100°C per hour. Upon cooling to about 700°C, the furnace was opened and the lay-up was removed and permitted to cool naturally in air.

Disassembly of the lay-up following the run, and specifically removal of the inverted graphite foil box 69, revealed that a metal matrix composite body had formed on the box interior and had substantially replicated all surfaces thereof. Sectioning of the metal matrix composite body revealed it to be hollow and bound on the bottom surface by the carcass of matrix metal. Significantly, the formed MMC structure accurately replicated the interior surfaces of the graphite foil box, as suggested by Figure 7. The formed structure had a wall thickness of about 1-2 mm.

The preceding examples illustrate certain embodiments and features of the present invention. An artisan of ordinary skill will appreciate that numerous modifications may be made to the above-described examples without departing from the present invention as defined in the appended claims.

WHAT IS CLAIMED IS:

1. A method for producing a metal matrix composite body, comprising:
applying a permeable material consisting essentially of an infiltration
5 enhancer to a substantially non-reactive surface;
contacting a molten matrix metal to at least a portion of said permeable
material; and
spontaneously infiltrating said molten matrix metal into said permeable
material up to said substantially non-reactive surface.
10
2. The method of claim 1, wherein said infiltration enhancer comprises
magnesium nitride and said matrix metal comprises aluminum, and further wherein at
least a portion of said aluminum matrix metal chemically reacts with at least a portion of
said magnesium nitride infiltration enhancer to produce aluminum nitride.
15
3. The method of claim 1, wherein said applying comprises depositing said
infiltration enhancer by means of a chemical vapor deposition process.
4. The method of claim 3, wherein said chemical vapor deposition process
20 comprises chemically reacting vapor phase infiltration enhancer precursor with an
infiltrating atmosphere.
5. The method of claim 4, wherein said vapor phase infiltration enhancer
precursor material comprises magnesium and said infiltrating atmosphere comprises
25 nitrogen.
6. The method of claim 1, wherein said formed metal matrix composite body
comprises a geometry which substantially inversely replicates a shape of said
substantially non-reactive surface.
- 30 7. The method of claim 1, wherein said substantially non-reactive surface
comprises a barrier material.

8. The method of claim 1, further comprising applying a barrier material layer or coating to said substantially non-reactive surface.

5 9. The method of claim 1, wherein said substantially non-reactive surface is readily separable or detachable from said formed metal matrix composite body.

10. The method of claim 1, wherein said infiltration enhancer is applied to at least a portion of said substantially non-reactive surface as a slurry.

10 11. The method of claim 1, wherein said matrix metal is contacted to a zone of said permeable material which is small relative to a total area presented by said infiltration enhancer layer or coating.

15 12. The method of claim 1, wherein said infiltrating occurs predominantly in a direction which is countergravitational.

13. The method of claim 1, wherein said infiltration enhancer comprises a nitride of an element selected from the group consisting of magnesium, calcium and strontium.

20

14. The method of claim 1, wherein said infiltration enhancer comprises zinc oxide.

25 15. The method of claim 1, wherein said substantially non-reactive surface is reusable.

16. A method for producing a metal matrix composite body, comprising:
applying a permeable material comprising an infiltration enhancer and a fugitive material to a substantially non-reactive surface;
30 contacting a molten matrix metal to at least a portion of said permeable material; and

spontaneously infiltrating said molten matrix metal into said permeable material up to said substantially non-reactive surface.

17. A method for producing a metal matrix composite body, comprising:
- 5 applying a permeable material comprising an infiltration enhancer to a substantially non-reactive surface;
- contacting a molten matrix metal to at least a portion of said permeable material;
- spontaneously infiltrating said molten matrix metal into said permeable
- 10 material up to said substantially non-reactive surface to form said metal matrix composite body;
- reacting at least a portion of said matrix metal with at least a portion of said permeable material to form a filler material for said metal matrix composite body;
- solidifying said molten matrix metal; and
- 15 recovering said substantially non-reactive surface.

18. The method of claim 17, wherein said applying comprises supplying an infiltration enhancer precursor material to said substantially non-reactive surface in solid form, and then reacting at least a portion of said infiltration enhancer precursor material
- 20 with an infiltrating atmosphere to produce said infiltration enhancer.

19. The method of claim 5, wherein said infiltration enhancer precursor material is heated to a temperature of at least 600°C, and said infiltrating atmosphere is provided at about atmospheric pressure.

Fig. 1.

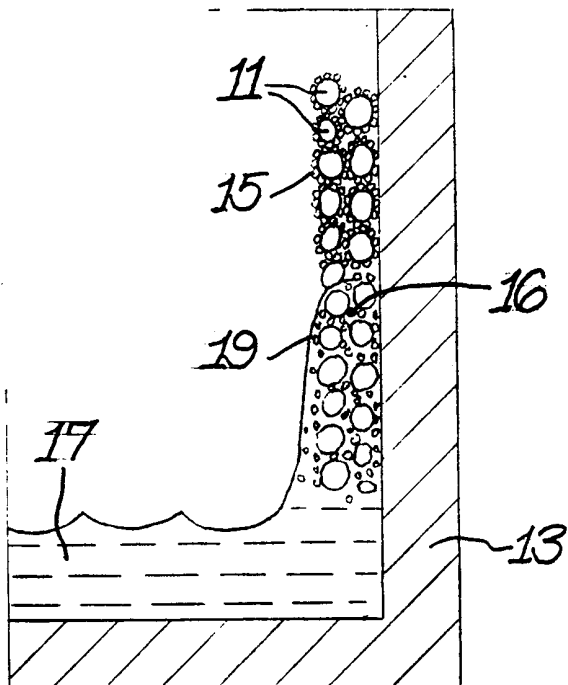


Fig. 2A.

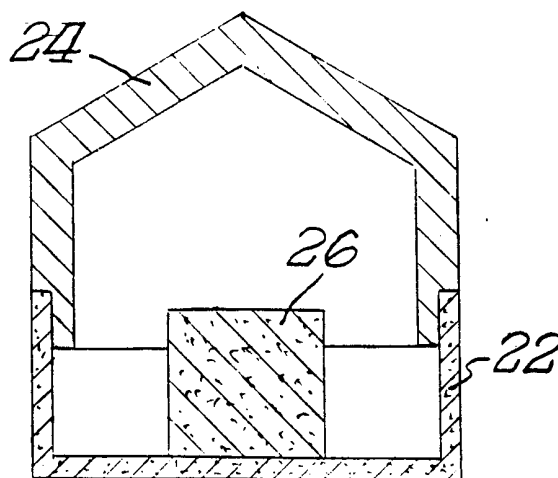


Fig. 2C.

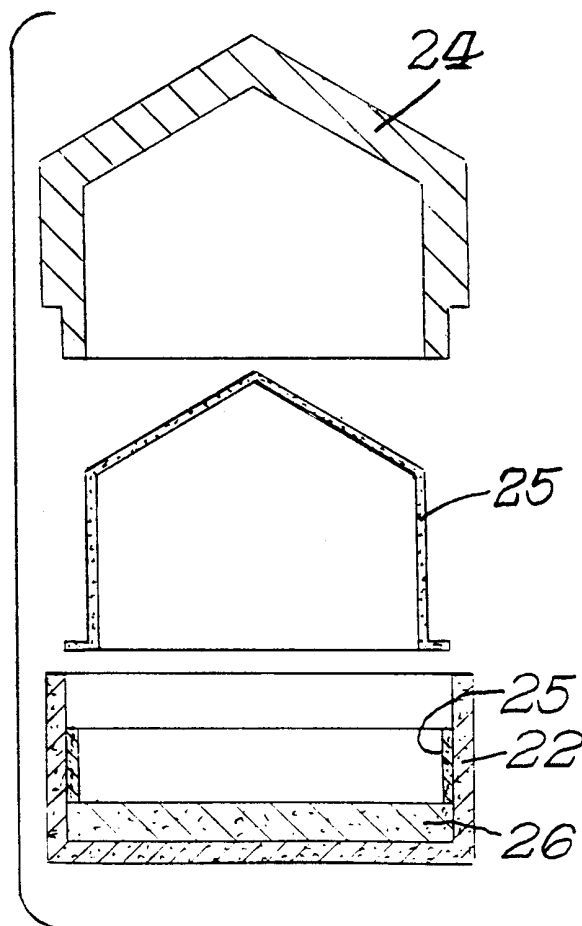


Fig. 2B.

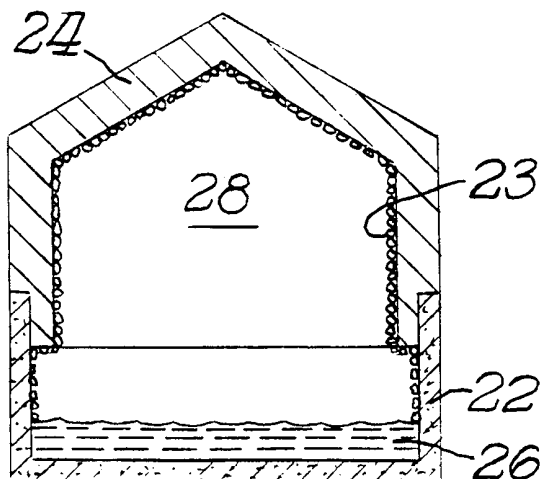


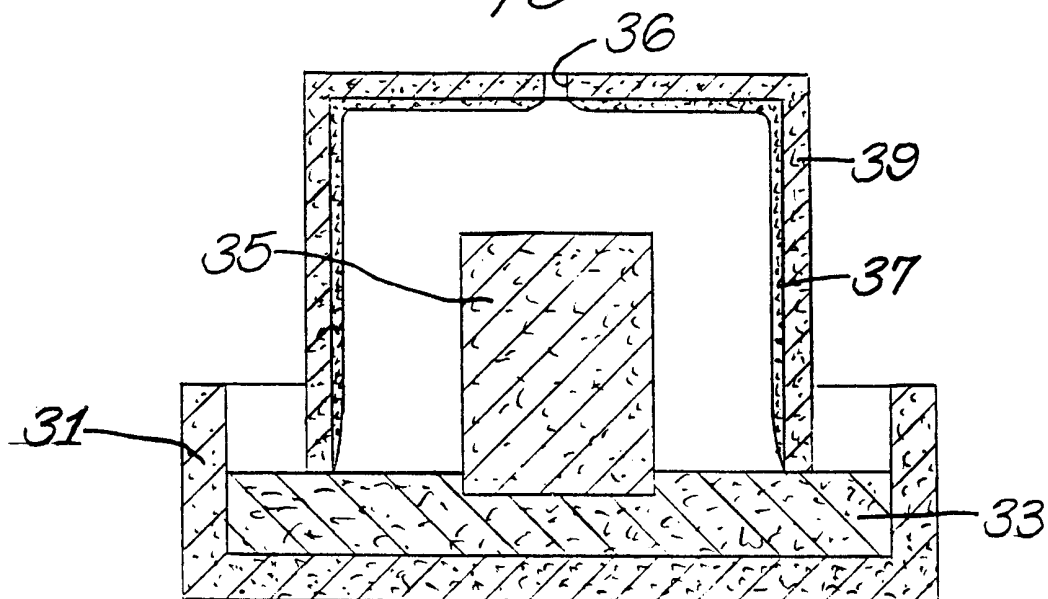
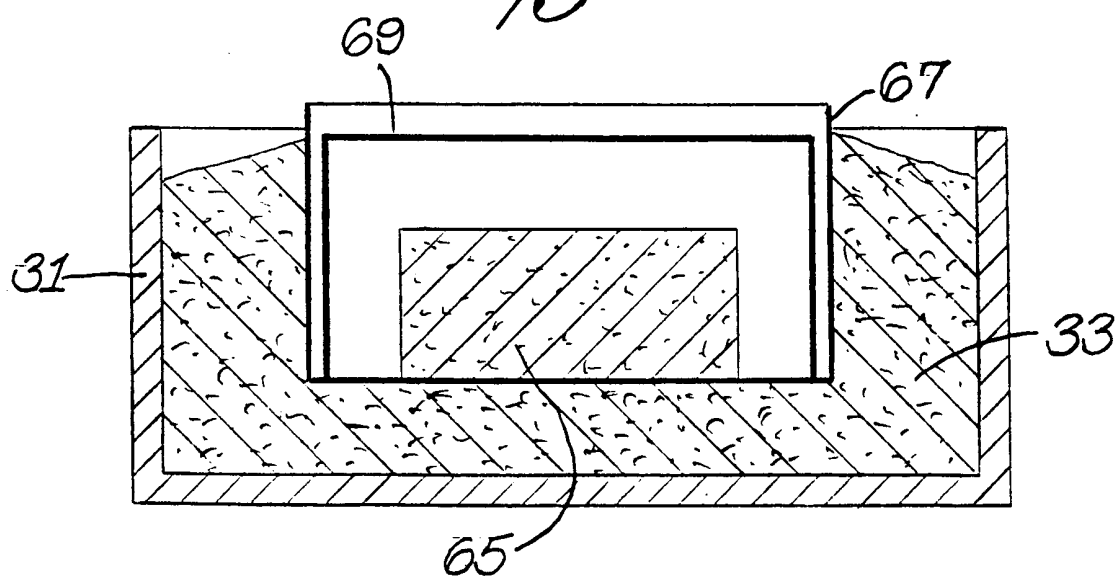
Fig. 3.*Fig. 6.*

Fig. 4A.

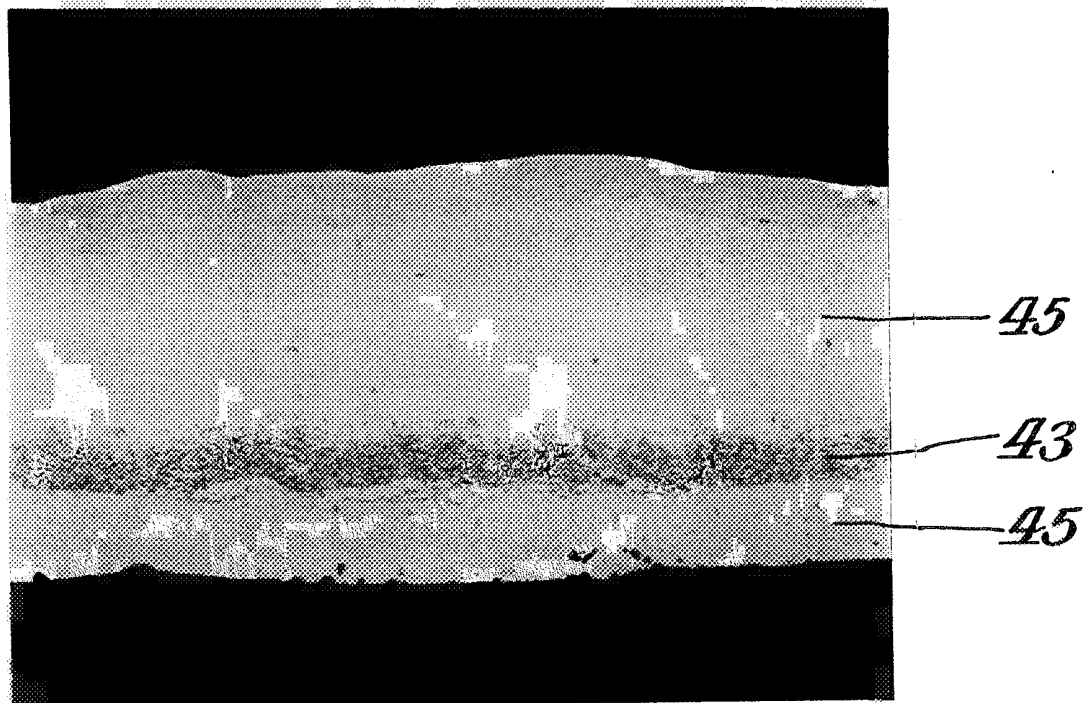
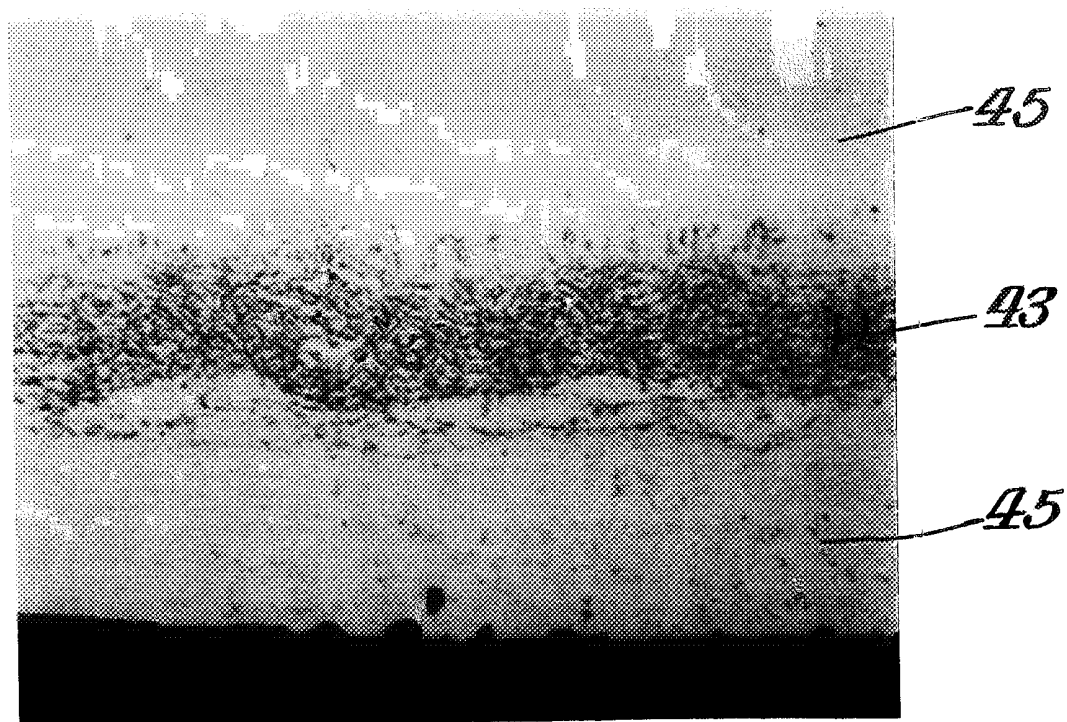


Fig. 4B.



4/4

Fig. 5.

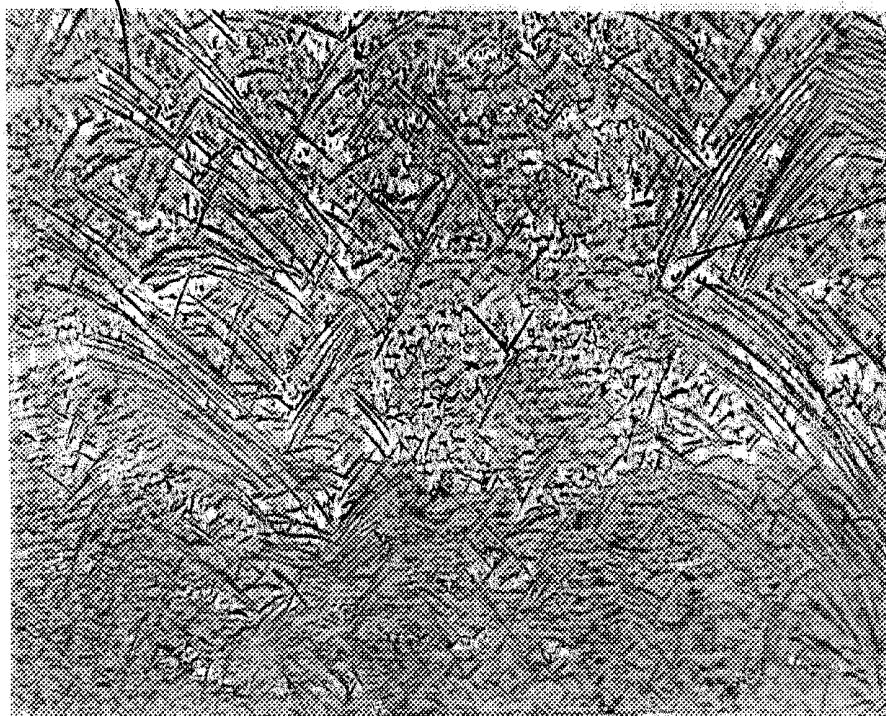


Fig. 7.

