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[54] METHOD FOR PREPARING METAL MOLDING COMPOSITIONS

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[57] **ABSTRACT**

Moldable pellets consisting essentially of a metal matrix having boron filaments positioned therein substantially in parallel and separated from each other by said metal; the method of producing the pellets, and the process of manufacturing shaped composite structures wherein said pellets are molded to the desired shape, e.g., by extrusion or transfer molding.

3 Claims, No Drawings

METHOD FOR PREPARING METAL MOLDING COMPOSITIONS

The invention described herein was made in the course of or under a contact or subcontract thereunder with the U. S. Department of Defense, Office of Naval Research.

BACKGROUND OF THE INVENTION

1 Field of the Invention

Composite structures having a metal matrix and discontinuous, boron fibers as reinforcement.

2 Background of the Invention

Use of refractory, discontinuous fibers as reinforcing agents in composites comprising a metal matrix is well known. Although it has been appreciated that parallel orientation of the fibers with respect to each other in a metal matrix contributes to high strength characteristics of the resulting composite, the means of realizing such orientation have been either cumbersome or ineffective. A frequently employed method has consisted of hand-packing the fibers in close proximity to each other in such a manner that the longitudinal axes of the fibers are parallel to each other, and then infiltrating the packing with a melt of the metal which is to serve as matrix. Hand-packing for orientation has many obvious disadvantages. Another method involves hand lay-up of the fibers in a molding with alternating layers of the powdered metal, and molding of the resulting structure by pressing and/or sintering. Whether the matrix metal be admixed with the reinforcing fiber in molten form or in dry, powder form, it is virtually impossible to keep the fibers from sliding and concentrating into pits or crevices of the mold, even before pressure is applied.

Upon application of pressure, of course, the loose fibers are readily broken or become disoriented. Still another method of orienting reinforcing fibers has involved exposing the fibers, while embedded in a mobile medium, to a magnetic field. Such a method, of course, is limited to magnetizable fibers and non-magnetizable matrices; moreover, the method does not provide the versatility of making various shapes.

Boron fibers or filaments are well known in the art to be valuable reinforcing agents for either organic polymer or metal matrices. Properties of boron filaments are described for example, by Harvey H. Herring in the Report to the National Aeronautics and Space Administration, which is entitled "Selected Mechanical and Physical Properties of Boron Filaments, " and identified as NASA–TN–D 3202, Jan. 1966, and also the report by Robert M. Witucki, entitled "Boron Filaments" and available from the Office of Technical Services, Arlington, Virginia, as publication CR–96. Boron is highly reactive and is susceptible to attack by numerous chemicals and to mechanical crushing as a result of extreme molding conditions. Moreover, it often is difficulty wetted by the matrix material, so that the usual hot-pressing powder metallurgy techniques do not provide for full realization of the potentially superior reinforcing property of boron fiber. Extrusion of random boron fiber/powdered metal mixture results in extensive fiber breakage. On the other hand, if the die casting method of forming unfilled metal, wherein a molten metal is forced into a die cavity, is modified in an attempt to include boron fibers suspended in the melt, surface shrinkage over thick areas frequently occurs, and breakage of fiber occurs during the compaction step and, in some case, again during cooling of the molding due to differences in thermal contraction of the components.

These and other problems relating to utilization of short boron fiber in composites having a metal matrix are solved by the present invention wherein there is provided a method which not only obviates the necessity of hand lay-up for obtaining parallel orientation, but also assures retention of the oriented positioning of the fibers during processing, and preservation of the intrinsically excellent reinforcing property of the boron fibers and also provides for very good adhesion of fiber to metal matrix.

SUMMARY OF THE INVENTION

The invention provides moldable pellets consisting essentially of a metal matrix having boron fibers positioned therein in parallel, said fibers being substantially separated from each other by the matrix metal. The invention also provides the method of producing said pellets and the method of manufacturing shaped composite structures wherein said pellets are molded to the desired shape using heat and pressure.

The reinforcing fiber may be entirely of boron or it may be a fiber or filament formed by vapor deposition of boron on a core of a high melting metal, e.g., a metal such as tungsten or chromium or other metals of say, Groups V to VIII of the Periodic Arrangement of Elements. The metal matrix of the pellets is a comparatively low melting metal, e.g., copper, aluminum, zinc, magnesium, silver, indium or any metal, generally, of Groups I to IV of said Periodic Arrangement of Elements. Two or more different such metals and or alloys thereof may comprise the matrix.

The pellets are preferably made by coating individual boron filaments with the matrix metal, bonding plurality of the coated filaments together using the same or a different matrix metal to form an elongated body, e.g., a rod, strand, ribbon or tape, and cutting the body into very short lengths, e.g., to from, say one-thirtysecond to one-half inch lengths. The elongated body may also be made by infiltration with the molten matrix metal of an assembly of the coated or uncoated filaments within a removable housing or upon a support whereby a fixed parallel arrangement of the filaments is made possible.

The pellets are useful molding compounds in the usual molding techniques, e.g., in extrusion, transfer, and compression molding processes.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

According to the invention the manufacture of shaped composites comprising a metal matrix of Groups I to IV of the Periodic Arrangement of Elements and discontinuous boron fiber oriented therein is facilitated by providing pellets of a molding composition wherein each of a plurality of the fibers is surrounded by said metal and bonded together substantially in parallel through said metal. Said pellets are prepared by cutting into small segments an elongated body, e.g., a rod, ribbon or tape consisting essentially of a multiplicity of continuous lengths of boron filament positioned in parallel within the metal matrix throughout the length of the body. By elongated body is meant any unit having a length which is say, at least ten times as much as any other dimension of the body.

The elongated body which is cut into the moldable pellets is produced by bonding together by means of said metal a plurality of continuous boron filaments in parallel arrangement. Advantageously, this is effected by passing the filaments individually through molten metal to coat them, combining into a bundle a plurality of the coated filaments as they emerge from the molten bath, and allowing the bundle to cool. Depending upon the combining means, the bundle may have a circular or rectangular cross-section, i.e., it may be a rod or strand or a ribbon or tape. Thus when the emerging coated filaments are passed through a circular aperture or a very narrow groove, they will be gathered together into a rod; on the other hand, when they are caused to pass through a narrow slit, they will form a ribbon or tape. Other means of forming the elongated body include separate coating and bundling steps. Thus the individual filament is first coated, either by passing through molten metal and cooling, or by electroplating, or by plasma spraying, or by electrodeless plating, or by vapor deposition. Then the coated filaments are formed into an integral body by passing a plurality of them in contiguous relationship through heated rolls to form a tape or by forcing them together at the softening temperature of the coating metal through a constricted orifice to form a rod or ribbon. The elongated body may also be formed, of course, by infiltrating with the molten metal a fixed, parallel arrangement of long lengths of the coated or uncoated filament within a housing or container. The bundles of filaments may also be made by co-extrusion or pultrusion. An easy laboratory method involves simply hand puddling of boron fiber in the molten metal to form a strand. For making the moldable pellets there may be used any elongated body consisting essentially of metal matrix having occluded therein the boron filament oriented in parallel along the length of the body; however, in order that each filament is substantially separated from each other by matrix metal, it is preferred to coat the individual filaments before bundling. Gathering the filaments while the coating is still hot enough to permit adhesion is simply a matter of convenience.

The thickness of metal coating upon the filament may vary widely, depending upon the filament-to-metal ratio desired in the final composite structure as well as upon the means of manufacturing the elongated body. When the latter is made by simply passing the individual filaments through molten metal and passing the coated filaments, before the metal coating has hardened, through a single orifice to form a bundle, the thickness of the coating will necessarily determine the filament/metal ratio. Of course, a quantity of metal must be present on the filament such that when several coated filaments are bundled together the filaments adhere to each other through the matrix metal. A substantially uniform coating on the metal is recommended in order to guard against voids and interstices in the bundle; however, uniformity of coating thickness is not critical, because subsequent pressing for production of the final product results in substantial homogeneity. Usually a substantially uniform coating of say, about 0.5 mil in thickness or even less, depending upon the fiber diameter, will suffice for adherence of the fibers to each other within a bundle. However, the coating thickness must be such as to give the desired filler/matrix ratio in the composite body formed by bundling the coated filaments together to cause cohesion of the semi-molten coating. Otherwise, infiltration or other techniques which permit introduction of additional metal must be used to form the rod-like bundles.

The thickness of the elongated body will vary with the geometry of its cross-section. In the case of rods or strands the diameter will vary from, say about 10 to 100 mils. In the case of ribbons or tapes, the cross-section will have one dimension of from 10 to 100 mils and another dimension which will correspond to the thickness of from 1 to about 5 coated wires, say, a dimension of from about 4 to 20 mils. Generally, pellets cut from elongated bodies of either circular or rectangular cross-section will have a length of from about 0.05 to 0.5 inch and the next greater dimension of from about 0.01 to 0.25 inch with the dimension lengthwise the body being greater than any other dimension of the pellet.

The size of the pellets into which the elongated body is cut will depend somewhat upon the procedure to be used for making the finished composite. For extrusion and transfer or flow molding it is advantageous to cut the rod, ribbon, or tape into lengths which will vary in length from, say, .03 to 0.05 inch and preferably from about 0.06 to 0.25 inch. The pellets may contain substantially any number of boron filaments. Generally, depending upon the thickness of the filament, there will be from, say, about 5 to about 50 filaments longitudinally disposed parallelwise in the bundle or grouping which makes up the elongated body. Cutting the body into the pellet-size lengths, results, of course, in pellets containing the same number of filaments in the same orientation.

The pellets are useful molding compositions of generally employed molding techniques. Thus, they may be extruded, i.e., forced through a constructed orifice of substantially any geometrical configuration or size to give shaped extrudates having discontinuous boron fibers occluded therein in substantially parallel, overlapping array, Separation of the fibers from each other in the pellet-size molding compound prevents the fiber from being crushed or grossly deformed, which may occur when the fibers are randomly positioned as, for instance, in a molding mix of the dry components or in fragments of preformed mats formed by mixing short fiber with the molten metal and cooling. The presently provided pellets are likewise useful in conventional transfer molding and plunger molding processes as well as in conventional compression molding. The small pellets may actually be considered to be preforms; hence, they are particularly useful in transfer and flow molding processes in that they provide for reduced bulk factor and uniform density, thus assuring uniform preheating and controlled mobility of the molding compound from the receptacle through the gate. Alignment of the fibers in the pellets serves to reduce breakage thereof, and such alignment is retained in the finished molding.

Flow molding or extrusion of the pellets may be conducted at even below the melting point of the matrix metal without substantial fiber breakage. Indeed, use of temperatures below the melting point of the matrix is beneficial in that it permits controlled flow rate and assures that fiber and matrix flow together. Such controlled flow from the heated receptacle through the similarly heated runners and gates and into the forming die thereby permits controlled fiber orientation and distribution, so that either rod or sheet of unidirectionally oriented, overlapped fibers or complex, contoured shapes having locally controlled fiber orientation can be produced.

Advantageously, the temperature employed for either extrusion, transfer or flow molding or compression molding is below the melting point of the pure metal, or below the liquids temperature of the alloy (for control of fiber flow). Also at such temperatures the possibility of any deleterious fiber-matrix interaction is minimized. However, the temperature should be high enough to assure flow of the molding compound during molding. Preferably, the pellets should be heated in the receptacle to a temperature of about from 5° to 30° C. below the matrix melting or liquidus temperature. Lower temperatures may be used, provided the equipment can deliver enough force or pressure to do the job. Of course, other factors may enter in at lower temperatures, such as the surface condition of the molded part. Heating in the receptacle should be conducted for a period which is long enough to insure thorough heating of the molding compound, which period is preferably less than one hour. Molding equipment, e.g., orifice, runner, gate and the platens should be maintained at about the same temperature. The pressure used in flow molding or extrusion techniques is determined by several factors, including runner and gate dimensions, matrix and fiber properties, temperature, reduction ratio, lubrication, etc. The pressure conditions to be used with a particular molding composition and with a particular technique and equipment is thus commonly and necessarily arrived at by routine experimentation which is well within the purview of those skilled in the art.

Substantially any metal may be used as the matrix; however, because the readily softened metals are more useful in conventional hot molding processes, the pellets are most advantageously prepared from such metals as, say, aluminum, copper, zinc, silver, magnesium and lead and other metals of e.g., Groups I to IV of the Periodic Arrangement of the Elements. Alloys of such metals are generally useful. Instead of a single metal, two or more may form a matrix. Thus, the long boron filament may be coated first with one metal and then with one or more successive coats of another metal previous to bundling. In cases where there is deleterious reaction between the boron filament and the molten metal, the filament may first be coated with a thin layer of unreactive silicon carbide before it is coated with metal. Also, filaments which have been metal coated may be assembled parallel to each other within a container and the resulting assembly infiltrated with another metal. Although boron fibers or filaments are well known in the art to be valuable reinforcing agents in composite structures having a matrix of the commercially available polymers, boron is susceptible to attack by numerous chemicals, and, it has often been necessary to provide for separating boron fiber from the polymeric matrix, e.g., by employing an intermediate layer of an inert material between the fiber and the matrix. In the present instance, there appears to be no degradation of the boron fiber or filament during fabrication of the pellets or of the composite structures molded therefrom; and, although interaction of a kind may occur between boron and the matrix metal, such interaction if any, results in a beneficial effect, possibly owing to formation of some metal boride linkages between filament and matrix which result in enhancement of mechanical and thermal properties. For some purposes, however, e.g., to obtain greater thermal stability or better adhesion, it may be desirable to coat the fiber with a comparatively non-reactive high-melting metal, e.g., a metal of Groups V to VIII of the Periodic Arrangement of Elements, e.g., nickel, tungsten or molybdenum or with silicon carbide, as previously mentioned. Such coated boron fiber will also be referred to herein and in the claims as boron fiber or filament.

The presently employed reinforcing fiber may be entirely of boron or it may be a fiber or filament formed by vapor phase deposition of boron on a core of a high melting metal. Vapor phase deposition of boron on a filament of metal such as tungsten, tantalum, molybdenum or titanium or another metal of Groups V to VIII of the Periodic Arrangement of Elements, is generally employed in the manufacture of boron filaments. Irrespective of the nature of the core, the art usually refers to the filaments as boron filament, and this terminology is meant to be so understood in the present specification and the appended claims. Presently, the most readily available of the boron fibers or filaments or reinforcing grade are those which have been produced by vapor deposition of boron on tungsten. Generally, the presently useful filaments will have a diameter of from, say, about 1 mil to about 6 mils. The cored filaments will have similar over-all diameters, with the core ranging from, say, 0.3 mil to 0.8 mil in diameter. Thus a commonly used boron filament has a tungsten core diameter of 0.5 mil and a 1.8 mil coating of boron (total diameter, 4.1 mil). Generally, the quantity of boron present is greater than that of the metal core. A Group V to VIII metal may also be used to coat the cored boron filament.

The amount of boron filament in the molding pellets and hence in the shaped composites molded therefrom will vary greatly, depending upon the properties desired; however, in order to impart significant improvements as compared to the un-reinforced metal, the filament should be present in a quantity of at least 5 per cent by volume of the pellet or molding. Boron filament loadings of as high as about 80 per cent by volume are attainable; but, to obtain optimum modulus and strength characteristics, it is preferred to employ the boron filament in a quantity which is from, say, about 15 per cent to about 60 per cent by volume of the pellet or finished composite.

For processing convenience in extrusion or transfer molding, it may be desirable to introduce the fibrous reinforcing agent in the form of very highly loaded pellets containing from 40 to 80 per cent of fiber and subsequently, during processing, to blend these pellets with granules or a melt of pure metal to achieve the finally desired loading of reinforcing agent uniformly distributed and oriented in the molding.

The filament is present in the elongated body and in the pellets in continuous form; in the molded structures, it is present in discontinuous form. By "continuous form" is meant the positioning of the filament along one dimension of the reinforced unit. By "discontinuous form" is meant use of very small lengths of the filament, say, pieces which may vary from about one-thirtysecond inch or less to about one-fourth inch, which pieces are smaller than any one dimension of the reinforced unit. Presence of continuous lengths of the filament in the elongated body and in the pellet generally provides for uniaxial positioning of the filament; the orientation thus obtained, plus the presence of matrix metal between the filaments eliminates contact of the filaments with each other so that during molding there is eliminated the filament damage which occurs when filaments are forced against each other during pressing.

The invention thus provides a new and valuable method by which a metal/boron fiber mixture can be flow molded or pressure formed into complex three dimensional shapes or extruded without extensive fiber breakage, thereby retaining the high reinforcement efficiency of boron fiber.

The invention is further illustrated by, but not limited to, the following examples:

EXAMPLE 1

This example shows the manufacture and molding of pellets consisting of indium as the matrix metal and boron filament (United Aircraft) having a diameter of 1.4 mil, said filament having an approximately 0.5 mil tungsten core and an approximately 0.45 mil thick coating of boron. A graphite block was clamped on each side of the ceramic cover of a hot plate. About 40 of such boron filaments were respectively threaded through separate small grooves in the first graphite block, passed through a pool of indium on the ceramic surface of the hot plate and then converged through a single groove in the second graphite block to form a boron bundle. The molten indium on the ceramic hot plate coated the boron fibers individually during their continuous passage over the hot plate. There resulted a uniformly coated boron bundle, about one-thirtysecond inch in diameter, wherein each filament of the bundle was bonded to another filament through a layer of indium. The indium-coated boron bundle was taken up on a reel, as formed. Subsequently, lengths of the reeled material were cut into one-fourth inch length grains and the grains were extruded at 150° C. from a 1 inch extrusion chamber through a one-eighth inch diameter tapered (4:1 area reduction) orifice. The fibers in the extrudate thus obtained were well collimated and essentially unbroken. The extrudate, having 9 volume per cent fiber loading, had a flexural strength of 1,100 psi, and a flexural modulus of 3,050,000 psi as compared to 250 psi and 1,050,000 psi, the flexural and modulus values for cast indium.

Transfer molding, instead of extrusion, of the one-fourth inch length grains, demonstrated similarly marked improvement in the mechanical characteristics of the resulting composite as compared to indium, alone.

EXAMPLE 2

Employing the procedure described in Example 1, continuous boron filament having an 0.5 mil tungsten core and a total diameter of 4 mil was coated with indium and bundled together as in that Example. The resulting rod was cut into lengths of from about **one-eighth to one-fourth inch and the pellets thus obtained were extruded at** 150° C. and 200 lbs. oil pressure to give several feet of a smooth extrudate having a diameter of one-fourth inch. It was sectioned lengthwise and etched in concentrated nitric acid to reveal the fibers. Microscopic examination of the etched surface showed parallel, overlapping arrangement of long, unbroken fibers.

EXAMPLE 3

Continuous boron filament (0.5 mil tungsten core, with total diameter of 1.4 mil) was electrodelessly coated with nickel, and the resulting nickel-coated filament was cut into one-fourth inch lengths and sprinkled on to the surface of a molten indium pool resting on the flat surface of a ceramic hot plate. The cut fiber was worked into the indium and then scraped to the edge of the ceramic plate to form a rope. Upon cooling the rope, visual and microscopic examination showed that the boron fibers were all individually encased, substantially in parallel, by the indium.

The ropes were cut into one-half inch lengths and extruded at 130° C. and 170 pounds oil pressure through a molybdenum sulfide-coated one-eighth inch diameter steel orifice. The extruded rod possessed a very smooth surface. Evaluation of the ultimate tensile strength of this extrudate gave a valve of 1,095 psi, as compared to 258 psi, the similarly obtained value for indium, alone.

EXAMPLE 4.

Boron filament, 4 mil in diameter (0.5 mil tungsten core), was coated with lead-tin (50/50) solder by the procedure used in Example 1 except that the filaments passed through a pool of the molten solder on the ceramic hot plate. There were used nine freshly coated filaments to form a rod, which was subsequently cut into one-fourth inch lengths to give pellets. Transfer molding of the pellets at about 10° C. below the liquidus point of the solder gave a smooth, integral molded unit.

EXAMPLE 5

The 4 mil diameter boron filament described in Example 2 was coated with galvanizing grade zinc by the procedure used in Example 1, except that the fibers passed through a pool of molten zinc on the ceramic hot plate. A rod was formed from 16 of the freshly coated fibers and cut into one-fourth inch lengths. Extrusion of the resulting pellets at about 400° C. gave an extrudate containing the fibers in parallel, overlapping array and characterized by mechanical properties which were significantly superior to those of extruded zinc, alone.

EXAMPLE 6

The 4 mil boron filament described in Example 2 was coated with aluminum by the procedure used in Example 1, except that the fibers passed through a pool of molten aluminum on the ceramic hot plate. A rod prepared from 9 of the freshly aluminum-coated filaments was cut into one-fourth inch pellets. The pellets were charged to a receptacle where they were uniformly heated to just below the melting point of aluminum, and maintained at said temperature as they passed through a runner and gate into a likewise heated cavity for back transfer molding to give a cylindrical tube.

EXAMPLE 7

The 4 mil diameter boron filament described in Example 2 was electrolessly nickel plated to provide a thick nickel coating (plated first for 45 minutes, then replated for 30 minutes) and several of the plated filaments were positioned lengthwise, uniaxially, in 12 mm I.D. capillary tube. The tube with its contents was immersed in molten lead-tin (50:50 solder) and vacuum infiltrated. After solidification, the glass tube was broken away from the rod of solder plus fiber. The rod was cut to give one-fourth inch long pellets having parallel orientation of the fiber along the length of the pellet. Extrusion of the pellets at 170° C. gave a smooth, cylindrical extrudate wherein the reinforcing filament was positioned in parallel within the matrix metal.

EXAMPLE 8

The nickel-plated boron filament of Example 7 was positioned in a capillary tube as in that Example and the tube and its contents was infiltrated with molten magnesium, the said infiltration being conducted under a blanket of sulfur dioxide in order to avoid any hazards incident to use of this molten metal and to maintain cleanliness of the melt. The glass tube was readily removed from the rod which resulted upon solidification, and the rod was cut into three-eighth inch pellets. Extrusion of the pellets at about 620° C through a rectangular orifice gave a well-formed extrudate wherein the reinforcing boron fibers were aligned in parallel along the length of the extrudate.

Operating as above described, boron filament is readily incorporated into other metal matrices, e.g., copper, silver, gallium, titanium; etc., to provide molding compounds for speedy, potentially low cost forming of boron-reinforced metal structures. The presently provided molding compounds of pre-encapsulated, collimated boron fibers thus constitute a notable improvement in the art.

Employing the pellets and the fabrication method made possible thereby, there are readily produced extremely tough, shaped composite structures which, depending upon the configuration of the mold, are useful in numerous industrial and space applications wherein high-strength components are required; e.g., bearings, gaskets and machine tools of all kinds.

It is to be understood that although the invention has been described with specific reference to particular embodiments thereof, it is not to be so limited since changes and alterations therein may be made which are within the full intended scope of this invention.

What we claim is:

1. The method of manufacturing a shaped composite structure wherein moldable pellets consisting essentially of
   a. a metal of Groups I to IV of the Periodic Arrangement of Elements and
   b. boron filaments having a length of about 0.06 to about 0.25 inch, said filaments being positioned substantially in parallel in the metal and

   separated from one another by said metal, are extruded through a restricted orifice at a temperature which is below the melting point of the matrix but at a temperature which is sufficient to effect flow of the metal at the pressure of extrusion.

2. The method defined in claim 1 further limited in that the temperature is from 5° to 30° C. below the melting point of the metal.

3. The method defined in claim 1 wherein said moldable pellets are transfer molded by charging the pellets to a receptacle; heating the pellets to a temperature below the melting point of the matrix metal but sufficiently high to permit flow of the pellets; and applying pressure to force the pellets to flow through a runner and gate into a mold while maintaining the pellets at said temperature.

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