

(12) PATENT
(19) AUSTRALIAN PATENT OFFICE

(11) Application No. AU 199658661 B2
(10) Patent No. 707820

(54) Title
Fiber reinforced aluminum matrix composite

(51)⁶ International Patent Classification(s)
B32B 005/02 B32B 015/20
B32B 015/02 C22C 001/09
B32B 015/14 H01B 001/02

(21) Application No: **199658661** (22) Application Date: **1996.05.21**

(87) WIPO No: **WO97/00976**

(30) Priority Data

(31) Number (32) Date (33) Country
08/492960 **1995.06.21** **US**

(43) Publication Date : **1997.01.22**
(43) Publication Journal Date : **1997.03.13**
(44) Accepted Journal Date : **1999.07.22**

(71) Applicant(s)
Minnesota Mining and Manufacturing Company

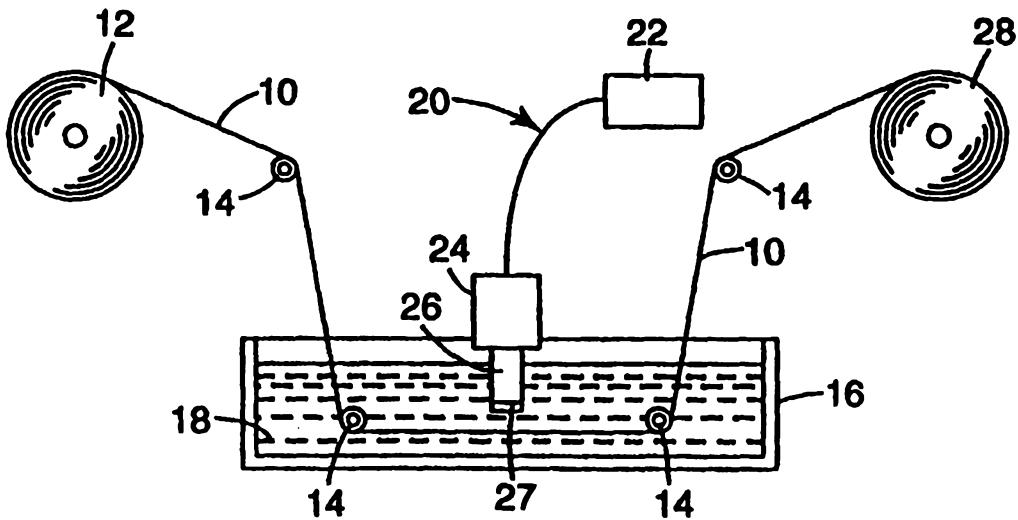
(72) Inventor(s)
Colin McCullough; Andreas Mortensen; Paul S. Werner; Herve E. Deve; Tracy L. Anderson

(74) Agent/Attorney
DAVIES COLLISON CAVE, GPO Box 3876, SYDNEY NSW 2001



(51) International Patent Classification⁶ : C22C 1/09, H01B 1/02		A1	(11) International Publication Number: WO 97/00976 (43) International Publication Date: 9 January 1997 (09.01.97)
(21) International Application Number: PCT/US96/07286			(81) Designated States: AU, CA, CN, JP, KR, NO, NZ, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).
(22) International Filing Date: 21 May 1996 (21.05.96)			
(30) Priority Data: 08/492,960 21 June 1995 (21.06.95) US			Published <i>With international search report.</i>
(71) Applicant: MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			
(72) Inventors: MCCULLOUGH, Colin; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). MORTENSEN, Andreas; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). WERNER, Paul, S.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). DEVE, Herve, E.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US). ANDERSON, Tracy, L.; P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			
(74) Agents: BERNSTEIN, David, B. et al.; Minnesota Mining and Manufacturing Company, Office of Intellectual Property Counsel, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).			

(54) Title: FIBER REINFORCED ALUMINUM MATRIX COMPOSITE



(57) Abstract

A composite metal matrix formed of polycrystalline α -Al₂O₃ fibers encapsulated within a matrix of substantially pure elemental aluminum, or an alloy elemental aluminum and up to about 2 % copper is disclosed. The resulting materials are characterized by their high strength and low weight and are particularly well suited for applications in various industries including high voltage power transmission.

FIBER REINFORCED ALUMINUM MATRIX COMPOSITE

Government License Rights

5 The U.S. Government has a paid-up license in this invention and the right in limited circumstances to require the patent owner to license others on reasonable terms as provided for by the terms of contract No. MDA 972-90-C-0018 awarded by the Defense Advanced Research Projects Agency (DARPA).

Field of the Invention

10 The present invention pertains to composite materials of ceramic fibers within in an aluminum matrix. Such materials are well-suited for various applications in which high strength, low weight materials are required.

Background of the Invention

15 Continuous fiber reinforced aluminum matrix composites (CF-AMCs) offer exceptional specific properties when compared to conventional alloys and to particulate metal matrix composites. The longitudinal stiffness of such composite materials is typically three times that of conventional alloys, and the specific strength of such composites is typically twice that of high-strength steel or aluminum alloys. Furthermore, for many applications, CF-AMCs are particularly attractive when compared to graphite-polymer composites due to their more moderate anisotropy in properties, particularly their high strength in directions different than those of the fiber axes. Additionally, CF-AMCs offer substantial improvements in allowable service temperature ranges and do not suffer from 20 environmental problems typically encountered by polymeric matrix composites. Such problems include delamination and degradation in hot and humid environments, particularly when exposed to ultraviolet (UV) radiation.

25 Despite their numerous advantages, known CF-AMCs suffer drawbacks which have hampered their use in many engineering applications. CF-AMCs generally feature high modulus or high strength, but seldom combine both properties. This feature is taught in Table V of R.B. Bhagat, "Casting Fiber-

Reinforced Metal Matrix Composites", in Metal Matrix Composites: Processing and Interfaces, R.K. Everett and R.J. Arsenault Eds., Academic Press, 1991, pp. 43-82. In that reference, properties listed for cast CF-AMC only combine a strength in excess of 1 GPa with a modulus in excess of 160 GPa in high-strength carbon-reinforced aluminum, a composite which suffers from low transverse strength, low compressive strength, and poor corrosion resistance. At the present time, the most satisfactory approach for producing CF-AMCs in which high strength in all directions is combined with a high modulus in all directions is with fibers produced by chemical vapor deposition. The resulting fibers, typically boron, are very expensive, too large to be wound into preforms having a small-radius of curvature, and chemically reactive in molten aluminum. Each of these factors significantly reduces the processability and commercial desirability of the fiber.

Furthermore, composites such as aluminum oxide (alumina) fibers in aluminum alloy matrices suffer from additional drawbacks during their manufacture. In particular, during the production of such composite materials, it has been found to be difficult to cause the matrix material to completely infiltrate fiber bundles. Also, many composite metal materials known in the art suffer from insufficient long-term stability as a result of chemical interactions which can take place between the fibers and the surrounding matrix, resulting in fiber degradation over time. In still other instances, it has been found to be difficult to cause the matrix metal to completely wet the fibers. Although attempts have been made to overcome these problems (notably, providing the fibers with chemical coatings to increase wettability and limit chemical degradation, and using pressure differentials to assist matrix infiltration) such attempts have met with only limited success. For example, the resulting matrices have, in some instances, been shown to have decreased physical characteristics. Furthermore, fiber coating methods typically require the addition of several complicated process steps during the manufacturing process.

In view of the above, a need exists for ceramic fiber metal composite materials that offer improved strength and weight characteristics, are free of long term degradation, and which may be produced using a minimum of process steps.

3

In view of the above, a need exists for ceramic fibre metal composite materials that offer improved strength and weight characteristics, are free of long term degradation, and which may be produced using a minimum of process steps.

The present invention relates to continuous fibre aluminium matrix composites having wide industrial applicability. Embodiments of the present invention pertain to continuous fibre aluminium matrix composites having continuous high-strength, high-stiffness fibres contained within a matrix material that is free of contaminants likely to cause brittle intermetallic compounds or phases, or segregated domains of contaminant material at the matrix/fibre interface. The matrix material is selected to have a relatively low yield strength whereas the fibres are selected to have a relatively high tensile strength. Furthermore, the materials are selected such that the fibres are relatively chemically inert both in the molten and solid phases of the matrix.

According to one embodiment of the present invention there is provided a composite material comprising a plurality of continuous polycrystalline α -Al₂O₃ fibers contained within a matrix of aluminium, said matrix being substantially free of material phases or domains capable of enhancing the brittleness of said matrix, wherein said composite material has an average tensile strength greater than 1.17 Gpa.

According to another embodiment of the invention there is provided a composite material comprising a plurality of continuous polycrystalline α -Al₂O₃ fibers within a matrix selected from the group consisting of an aluminium matrix and a matrix of an alloy of aluminium and up to about 2% by weight copper, based on the total weight of said alloy matrix, wherein said matrices contain less than 0.05 percent by weight impurities, based on the total weight of said matrices, and wherein said composite material has an average tensile strength of at least 1.17 Gpa.

Such composite structures offer high strength and low weight, while at the same time avoid the potential for long term degradation. Such composites may also be made without the need for many of the process steps associated with prior art composite materials.

In one embodiment, the continuous fibre aluminium matrix composites of the present invention are formed into wires exhibiting desirable strength-to-weight characteristics and high electrical conductivity. Such wires are well-suited for use as core

3

materials in high voltage power transmission (HVPT) cables, as they provide electrical and physical characteristics which offer improvements over HVPT cables known in the prior art.

One wire according to the present invention comprises a composite material comprising a plurality of continuous polycrystalline α - Al_2O_3 fibers contained within a matrix of aluminium, said matrix being substantially free of material phases or domains capable of enhancing the brittleness of said matrix, wherein said composite material has an average tensile strength of greater than 1.17 GPa and said plurality of continuous polycrystalline α - Al_2O_3 fibers includes at least one tow of continuous polycrystalline α - Al_2O_3 .

Another wire according to the present invention comprises a composite material comprising a plurality of continuous polycrystalline α - Al_2O_3 fibers within a matrix selected from the group consisting of an aluminium matrix and a matrix of an alloy of aluminium and up to about 2% by weight copper, based on the total weight of said alloy matrix, wherein said matrices contain less than 0.05 percent by weight impurities, based on the total weight of said matrices, and wherein said composite material has an average tensile strength of at least 1.17 GPa and said plurality of continuous polycrystalline α - Al_2O_3 fibers includes at least one tow of continuous polycrystalline α - Al_2O_3 fibers.

According to a further embodiment of the invention there is provided a method of making a continuous composite wire, said method comprising the steps of: (a) melting a metallic matrix material selected from the group consisting of aluminium and an alloy of aluminium with up to 2% by weight copper, based on the total weight of said alloy matrix, wherein said matrix material contain less than 0.05 percent by weight impurities, based on the total weight of said matrix materials, to provide a contained volume of melted metallic matrix material; (b) imparting ultrasonic energy to cause vibration of the contained volume of melted metallic matrix material of step (a); (c) immersing a plurality of continuous polycrystalline α - Al_2O_3 fibers into said contained volume of melted metallic matrix material while maintaining said vibration to permit the melted metallic matrix material to infiltrate into and coat said plurality of fibers such that an infiltrated, coated plurality of fibers is

3

provided; and (d) withdrawing said infiltrated, coated plurality of fibers from said contained volume of melted metallic matrix material under conditions which permit the melted matrix material to solidify to provide a wire comprising composite materials according to the present invention wherein the plurality of continuous polycrystalline α -Al₂O₃ fibers include
8 at least one tow of continuous polycrystalline α -Al₂O₃ fibers.

Brief Description of the Drawings

Figure 1: is a schematic representation of an apparatus for producing composite metal matrix wires using ultrasonic energy.

13 Figures 2a and 2b: are schematic, cross-sections of two embodiments of overhead high voltage transmission cables having composite metal matrix cores.

Figure 3: is a chart comparing strength-to-weight ratios for materials of the present invention with other materials.

18 Figures 4a and 4b: are graphs comparing projected sag as a function of span length for various cables.

Figure 5: is a graph showing the coefficient of thermal expansion as a function of temperature for a CF-AMC wire.

Detailed Description

23 The fiber reinforced aluminium matrix composites of the present invention comprise continuous fibres of polycrystalline α -Al₂O₃ fibres encapsulated within either a matrix of substantially pure elemental aluminium or an alloy of pure aluminium with up to 2% by weight copper, based on the total weight of the matrix. The preferred fibres comprise equiaxed grains of less than 100 nm in size and a fibre diameter in the range of 1 to 50 micrometres. A fibre diameter in the range of 5 to 25 micrometeres is preferred with a ramp of 5 to 15 micrometers being most preferred. Preferred composite materials according to the present invention have a fibre density of between 3.90 to 3.95 grams per cubic centimetre. Among the preferred fibres are those described in US Patent No 4954462
28 (Wood *et al.*, assigned to Minnesota Mining and Manufacturing

33

Company, St Paul, MN). Such fibres are available commercially under the designation NEXTEL 610 Ceramic Fibres from the Minnesota Mining and Manufacturing Company, St. Paul, MN. The encapsulating matrix is selected to be such that it does not react chemically with the fibre material (that is, is relatively chemically inert with respect to the fibre material), thereby eliminating the need to provide a protective coating on the fibre exterior.

As used herein, the term "polycrystalline" means a material having predominantly a plurality of crystalline grains in which the grain size is less than the diameter of the fibre in which the grains are present. The term "continuous" is intended to mean a fibre having a length which is relatively infinite when compared to the fibre diameter. In practical terms, such fibres have a length on the order of 15 centimetres or at least several metres, and may even have lengths on the order of kilometres or more.

In the preferred embodiments, the use of a matrix comprising either substantially pure elemental aluminium, or an alloy of elemental aluminium with up to 2 % by weight copper, based on the total weight of the matrix, has been shown to produce successful composites. As used herein the terms "substantially pure elemental aluminium", "pure aluminium" and "elemental aluminium" are interchangeable and are intended to mean aluminium containing less than 0.05 % by weight impurities. Such impurities typically comprise first row transition metals (titanium, vanadium, chromium, manganese, iron, cobalt, nickel and zinc) as well as second and third row metals and elements in the lanthanide series. In one preferred embodiment, the terms are intended to mean aluminium having less than 0.03 % by weight iron, with less than 0.01 % by weight iron being most preferred. Minimizing the iron content is desirable because iron is a common contaminant of aluminium, and further, because iron and aluminium combine to form brittle intermetallic compounds (for example, Al_3Fe , Al_2Fe , etc). It is also particularly desirable to avoid contamination by silicon such as from SiO_2 , which can be reduced to free silicon in the presence of molten aluminium) because silicon, like iron, forms a brittle phase, and because silicon can react with the aluminium (and any iron which may be present) to form brittle Al-Fe-Si intermetallic compounds. The presence of brittle phases in the composite is undesirable, as such phases tend to promote fracture in the composite when subjected to stress. In particular, such brittle phases may cause the matrix to fracture even before the

reinforcing ceramic fibres fracture, resulting in composite failure. Generally, it is desirable to avoid substantial amounts of any transition metal, (that is, Groups IB through VIIIB of the periodic table), that form brittle intermetallic compounds. Iron and silicon have been particularly specified herein as a result of their commonality as impurities in metallurgical processes.

Each of the first row transition metals described above is relatively soluble in molten alumina and, as noted, can react with the aluminium to form is brittle intermetallic compounds. In contrast, metal impurities such as tin, lead, bismuth, antimony and the like do not form compounds with aluminium, and are virtually insoluble in molten aluminium.

As a result, those impurities tend to segregate to the fibre/matrix interface, thereby weakening the composite strength at the interface. Although such segregation may aid longitudinal stem of the ultimate composite by contributing to a global load sharing domain (discussed below), the presence of the impurities ultimately results in a substantial reduction in the transverse strength of the composite due to decohesion at the fibre/matrix interface.

Elements from Groups IA and IIA of the periodic table tend to react with the fibre and drastically decrease the strength of the fibre in the composite. Magnesium and lithium are particularly undesirable elements in this regard, due, in part, to the length of time the fibres and the metal must be maintained at high temperatures during processing or in use.

It should be understood that referents to "substantially pure elemental aluminium", "pure aluminium", and "elemental aluminium" as used herein, are intended to apply to the matrix material rather than to the reinforcing fibre, since the fibres will likely include domains of iron (and possible other) compounds within their grain structure. Such domains typically are remnants of the fibre manufacturing process and have, at most, negligible effect on the overall characteristics of the resulting composite material, since they tend to be relatively small and fully encapsulated within the grains of the fibre. As such, they do not interact with the composite matrix, and thereby avoid the drawbacks associated with matrix contamination.

The metal matrix used in the composite of the present invention is selected to have a low yield strength relative to the reinforcing fibres. All this context, yield strength is defined as the stress at 0.2% of offset strain in a standardized tensile test (described in ASTM tensile standard E345-93) of the unreinforced metal or alloy. Generally, two classes

of aluminium matrix composites can be broadly distinguished based on the matrix yield strength. Composites in which the matrix has a relatively low yield strength have a high longitudinal tensile strength governed primarily by the strength of the reinforcing fibres.

As used herein, low yield strength aluminium matrices in aluminium matrix composites are

5 defined as matrices with a yield strength of less than 150 MPa. The matrix yield

strength is preferably measured on a sample of matrix material having the same composition and which has been fabricated in the same manner as the material used to form the composite matrix. Thus, for example, the yield strength of a substantially pure elemental aluminium matrix material used in a composite material would be determined by testing the

10 yield strength of substantially pure elemental aluminium without a fibre reinforcement. In

composites with low yield-strength matrices, matrix shearing in the vicinity of the matrix-fibre interface reduces the stress concentrations near broken fibres and allows for global stress redistribution. In this regime, the composite reaches "rule-of-mixtures" strength.

Pure aluminium has a yield strength of less than 13.8 MPa (2 ksi) and Al-2

15 wt% copper has a yield strength less than 96.5 MPa (14 ksi).

The low yield strength matrix composites described above may be contrasted with high yield strength matrices which typically exhibit lower composite longitudinal strength than the predicted "rule-of-mixtures" strength. In composites having high strength matrices,

the characteristic failure mode is a catastrophic crack propagation. In composite materials, high yield strength matrices typically resist shearing from broken fibres, thereby producing a high stress concentration near any fibre breaks. The high stress concentration allows

cracks to propagate, leading to failure of the nearest fibre and catastrophic failure of the composite well before the "rule-of-mixtures" strength is reached. Failure modes in this regime are said to result from "local load sharing". For a metal matrix composite with

25 50 volume per cent fibre, a low yield strength matrix produces a strong (that is, greater than 1.17 GPa (170 ksi)) composite when combined with alumina fibres having, strengths of greater than 2.8 GPa (400 ksi). Thus, it is believed that for the same fibre loading, the composite strength will increase with fibre strength.

The strength of the composite may be further improved by infiltrating the polycrystalline α -Al₂O₃ fibre tows with small or whiskers or short (chopped) fibres, of alumina. Such particles, whiskers or fibres typically on the order of less than 20

al., assigned to Kabushiki Kaisha Toyota Chuo Kenkyusho and Ube Industries, Ltd., both of Japan).

As noted above, one of the significant obstacles in forming composite materials relates to the difficulty in sufficiently wetting reinforcing fibers with the surrounding matrix material. Likewise, infiltration of the fiber tows with the matrix material is also a significant problem in the production of composite metal matrix wires, since the continuous wiring forming process typically takes place at or near atmospheric pressure. This problem also exists for composite materials formed in batch processes at or near atmospheric pressure.

The problem of incomplete matrix infiltration of the fiber tow can be overcome through the use of a source of ultrasonic energy as a matrix infiltration aid. For example, U.S. Patent No. 4,779,563 (Ishikawa et al., assigned to Agency of Industrial Science and Technology, Tokyo, Japan), describes the use of ultrasonic wave vibration apparatus for use in the production of preform wires, sheets, or tapes from silicon carbide fiber reinforced metal composites. The ultrasonic wave energy is provided to the fibers via a vibrator having a transducer and an ultrasonic "horn" immersed in the molten matrix material in the vicinity of the fibers. The horn is preferably fabricated of a material having little, if any, solubility in the molten matrix to thereby prevent the introduction of contaminants into the matrix. In the present case, horns of commercially pure niobium, or alloys of 95% niobium and 5% molybdenum have been found to yield satisfactory results. The transducer used therewith typically comprises titanium.

One embodiment of a metal matrix fabrication system employing an ultrasonic horn is presented in FIG. 1. In that Figure, a polycrystalline α -Al₂O₃ fiber tow 10 is unwound from a supply roll 12 and drawn, by rollers 14, through a vessel 16 containing the matrix metal 18 in molten form. While immersed in the molten matrix metal 18, the fiber tow 10 is subjected to ultrasonic energy provided by an ultrasonic energy source 20 which is immersed in the molten matrix metal 18 in the vicinity of a section of the tow 10. The ultrasonic energy source 20 comprises an oscillator 22 and a vibrator 24 having a transducer 26 and a horn 27. The horn 27 vibrates the molten matrix metal 18 at a frequency produced by the

oscillator 22 and transmitted to the vibrator 24 and transducer 26. In so doing, the matrix material is caused to thoroughly infiltrate the fiber tow. The infiltrated tow is drawn from the molten matrix and stored on a take-up roll 28.

The process of making a metal matrix composite often involves forming fibers into a "preform". Typically, fibers are wound into arrays and stacked. Fine diameter alumina fibers are wound so that fibers in a tow stay parallel to one another. The stacking is done in any fashion to obtain a desired fiber density in the final composite. Fibers can be made into simple preforms by winding around a rectangular drum, a wheel or a hoop. Alternatively, they can be wrapped onto a cylinder. The multiple layers of fibers wound or wrapped in this fashion are cut off and stacked or bundled together to form a desired shape. Handling the fiber arrays is aided by using water either straight or mixed with an organic binder to hold the fibers together in a mat.

One method of making a composite part is to position the fibers in a mold, filling the mold with molten metal, and then subjecting the filled mold to elevated pressure. Such a process is disclosed in U.S. Patent No. 3,547,180 entitled "Production of Reinforced Composites". The mold should not be a source of contamination to the matrix metal. In one embodiment, the molds can be formed of graphite, alumina, or alumina-coated steel. The fibers can be stacked in the mold in a desired configuration; e.g., parallel to the walls of the mold, or in layers arrayed perpendicular to one another, as is known in the art. The shape of the composite material can be any shape into which a mold can be made. As such, fiber structures can be fabricated using numerous preforms, including, but not limited to, rectangular drums, wheel or hoop shapes, cylindrical shapes, or various molded shapes resulting from stacking or otherwise loading fibers in a mold cavity. Each of the preforms described above relates to a batch process for making a composite device. Continuous processes for the formation of substantially continuous wires, tapes, cables and the like may be employed as well. Typically, only minor machining of the surface of a finished part is necessary. It is possible also to machine any shape from a block of the composite material by using diamond tooling. Thus, it becomes possible to produce many complex shapes.

A wire shape can be formed by infiltrating bundles or tows of alumina fibre with molten aluminium. This can be done by feeding tows of fibres into a bath of molten aluminium. To obtain wetting of the fibres, an ultrasonic horn is used to agitate the bath while the fibres pass through it.

5 Fibre reinforced metal matrix composites are important for applications wherein lightweight, strong, high-temperature-resistant (at least 300°C) materials are needed. For example, the composites can be used for gas turbine compressor blades in jet engines, structural tubes, actuator rods, I-beams, automotive connecting rods, missile fins, fly wheel rotors, sports equipment (for example, golf clubs) and power transmission cable support 10 cores. Metal matrix composites are superior to unreinforced metals in stiffness, strength, fatigue resistance, and wear characteristics.

In one preferred embodiment of the present invention, the composite material comprises between 15 30% to 70% by volume polycrystalline α -Al₂O₃ fibres, based on the total volume of the composite material within a substantially elemental aluminium matrix. It is preferred that the matrix contains less than 0.03% by weight iron, and most preferably less than 0.01% by weight iron, based on the total weight of the 20 matrix. A fibre content of between 40% to 60% by volume polycrystalline α -Al₂O₃ fibres is preferred. Such composites, formed with a matrix having a yield strength of less than 20 MPa and fibres having a longitudinal tensile strength of at least 2.8 GPa have been found to have excellent strength characteristics.

The matrix may also be formed from an alloy of elemental aluminium with up to 25 2% by weight copper, based on the total weight of the matrix. As in the embodiment in which a substantially pure elemental aluminium matrix is used, composites having an aluminium/copper alloy matrix preferably comprise between 30% to 70% by volume polycrystalline α -Al₂O₃ fibres by weight, and more preferably therefore 40% to 60% by volume polycrystalline α -Al₂O₃ fibres based on the total volume of the composite. In addition, the matrix preferably contains less than 0.03% by weight iron and most preferably less than 0.01% by weight iron, based on the total weight of the composite. The aluminium/copper matrix preferably has a yield strength of less than 30 90 MPa, and, as above, the polycrystalline α -Al₂O₃ fibres have a longitudinal tensile strength of at least 2.8 GPa. The properties of two composites, a first with an elemental aluminium

matrix, and a second with a matrix of the specified aluminium/copper alloy, each having between 55 to 65 volume per cent polycrystalline α -Al₂O₃ fibres are presented in Table I below:

100 100 100 100 100

Table I: SUMMARY OF COMPOSITE PROPERTIES ⁽¹⁾

	Pure Al 55-65 vol% Al₂O₃	Al-2wt%Cu 55-65 vol% Al₂O₃
Longitudinal Young's Modulus, E ₁₁ ⁽²⁾	220 - 260 GPa (32 - 38 Msi)	220 - 260 GPa (32 - 38 Msi)
Transverse Young's Modulus, E ₂₂	120 - 140 GPa (17.5 - 20 Msi)	150 - 160 GPa (22 - 23 Msi)
Shear Modulus, G ₁₂	48 - 50 GPa (6.5 - 7.3 Msi)	45 - 47 GPa (6.5 - 6.8 Msi)
Shear Modulus, G ₂₁	54 - 57 GPa (7.8 - 8.3 Msi)	55 - 56 GPa (8 - 8.2 Msi)
Long. tensile strength S _{11, T}	1500 - 1900 MPa (220 - 275 ksi)	1500 - 1800 MPa (220 - 260 ksi)
Long. compressive strength, S _{11, c}	1700 - 1800 MPa (245 - 260 ksi)	3500 - 3700 MPa (500 - 540 ksi)
Shear Strength S ₂₁ - S ₁₂ at 2% strain	70 MPa (10 ksi)	140 MPa (20 ksi)
Trans. strength S ₂₂ at 1% strain	110 - 130 MPa (16 - 19 ksi)	270 - 320 MPa (39 - 46 ksi)

⁽¹⁾ The properties listed in this table represent a range of mechanical performance measured on composites containing 55-65 vol% NEXTELTM 610 ceramic fibers. The range is not representative of the statistical scatter.

⁽²⁾ Index Notation

1 = Fiber direction; 2 = Transverse direction; ij: i direction normal to the plane in which the stress is acting, j = stress direction, S = Ultimate strength unless specified.

Although suitable for a wide variety of uses, in one embodiment, the composites of the present invention have applicability in the formation of composite matrix wire. Such wires are formed from substantially continuous polycrystalline α -Al₂O₃ fibres contained within the substantially pure elemental aluminium matrix or the matrix formed from the alloy of elemental aluminium and up to 2% copper by weight described above. Such wires are made by a process in which a spool of substantially continuous polycrystalline α -Al₂O₃ fibres, arranged in a fibre tow, is pulled through a bath of molten matrix material. The resulting segment is then solidified, thereby providing fibres encapsulated within the matrix. It is preferred that an ultrasonic horn, as described above, is lowered into the molten matrix bath and used to aid the infiltration of the matrix into the fibre tows.

Composite metal matrix wires, such as those described above, are useful in numerous applications. Such wires are believed to be particularly desirable for use in overhead high voltage power transmission cables due to their combination of low weight, high strength, good electrical conductivity, low coefficient of thermal expansion, high use temperatures, and resistance to corrosion. The competitiveness of composite metal matrix wires, such as those described above for use in overhead high voltage power transmission, is a result of the significant effect cable performance has on the entire electricity transport system. Cable having lower weight per unit strength, coupled with increased conductivity and lower thermal expansion, provides the ability to install greater cable spans and/or lower tower heights. As a result, the costs of constructing electrical towers for a given electricity transport system can be significantly reduced. Additionally, improvements in the electrical properties of a conductor can reduce electrical properties of a conductor can reduce electrical losses in the transmission system, thereby reducing the need for additional power generation to compensate for such losses.

As noted above, the composite metal matrix wires of the present invention are believed to be particularly well-suited for use in overhead high voltage power transmission cables. In one embodiment, an overhead high voltage power transmission cable can include an electrically conductive core formed by at least one composite metal matrix wire according to the present invention. The core is surrounded by at least one conductive jacket formed by a plurality of aluminium or aluminium alloy wires. Numerous cable core and jacket configurations are known in the cable art. For example, as shown in Figure 2a,

3

cross-section of one overhead high voltage power transmission cable 30 may be a core 32 of nineteen individual composite metal matrix wires 34 surrounded by a jacket 36 of thirty individual aluminium or aluminium alloy wires 38. Likewise, as shown in Figure 2b, as one of many alternatives, the cross-section of a difference overhead high voltage power transmission cable 30' may be a core 32' of thirty seven individual composite metal matrix wires 34' surrounded by a jacket 36' of twenty one individual aluminium or aluminium alloy wires 38'.

8 The weight percentage of composite metal matrix wires within the cable will depend upon the design of the transmission line. In that cable, the aluminium or aluminium 13 alloy wires used in the conductive jackets are any of the various materials known in the art of overhead high voltage power transmission, including, but not limited to, 1350 Al or 6201 Al.

18 In another embodiment, an overhead high voltage power transmission cable can be constructed entirely of a plurality of continuous fibre aluminium matrix composite wires (CF-AMCs). As is discussed below, such a construction is well-suited for long cable spans in which the strength-to-weight ratio and the coefficient of thermal expansion of the cable overrides the need to minimize resistive losses.

23 Although dependent upon a number of factors, the amount of sag in an overhead high voltage power transmission cable varies as the square of the span length and inversely with the tensile strength of free cable. As may be seen in Figure 3 CM-AMC materials offer substantial improvements in the strength-to-weight ratio over materials commonly used for cable in the power transmission industry. It should be noted that the strength, electrical conductivity and density of CF-AMC materials and cables dependent upon the fibre volume in the composite. For Figures 3, 4a, 4b, and 5 a 50% fibre volume was assumed, with a corresponding density of 3.2 gm/cm³ (approximately 0.1 15 lb/in³), tensile strength of 1.38 GPa (200 ksi), and conductivity of 30% IACS.

28 33 As a result of the increased strength of cables containing CF-AMC wires, cable sag can be substantially reduced. Calculations comparing the sags of CF-AMC cables as a function of span length with a commonly used steel scanting (ACSR) (31 wt% steel having a core of 7 steel wires surrounded by a jacket of 26 aluminium wires), and an equivalent all aluminium alloy conductor (AAAC) are shown in Figures 4a and 4b. All cables had equivalent electrical conductivity and diameter. Figure 4a demonstrates that CF-AMC

cables provide for a 40% reduction in tower height as compared to ACSR for spans of 550 metres (about 1800 feet). Likewise, CF-AMC cables allow for an increase in span length about 25% assuming allowable sags of 15 metres (about 50 feet). Further advantages from the use of CF-AMC cables in long spans are presented in Figures 4b. In Figure 4b, 5 the ACSR cable was 72 wt% steel having a core of 19 steel wind surrounded by a jacket of 16 aluminium wires.

The sag of a high voltage power transmission (HVPT) cable at its maximum operating temperature is also dependent upon the coefficient of thermal expansion (CTE) of the cable at its maximum operating temperature. The ultimate CTE of the cable is 10 determined by the CTE and the elastic modulus of both the reinforcing core and the surrounding strands. Within limits, materials with a low CTE and a high elastic modulus are desired. The CTE for the CP-AMC cable is shown in Figure 5 as a fixation of temperature. Reference values for aluminium and steel are provided as well.

It is noted that the present invention is not intended to be limited to wires and HVPT 15 cables employing composite metal matrix technology; rather, it is intended to include the specific inventive composite materials described herein as well as numerous additional applications. Thus, the composite metal matrix materials described herein may be used in any of a wide variety of applications, including, but not limited to, flywheel rotors, high performance aerospace components, voltage transmission, or many other applications in 20 which high strength, low density materials are desired.

It should be further noted that although the preferred embodiment makes use of the polycrystalline α -Al₂O₃ fibres described in US Patent No 4954462 currently being marketed under the trade name NEXTEL 610 by Minnesota Mining and Manufacturing Company of St Paul, MN, the invention is not intended to be limited to those specific fibres. Rather, 25 any suitable polycrystalline α -Al₂O₃ fibres is intended to be included herein as well. It is preferred, however, that any such fibre have a tensile strength at least on the order of that of the NEXTEL 610 fibres (approximately 2.8 GPa).

In the practice of the invention, the matrix must be chemically inert relative to the fibre over a temperature range between 20°C to 760°C. The temperature range represents the range of predicted processing and service temperatures for the composite. 30 This requirement minimizes chemical reactions between the matrix and fibre which may be

deleterious to the overall composite properties. In the case of a matrix material comprising an alloy of elemental aluminium and up to 2 % by weight copper, the as-cast alloy has a yield strength of 41.4 to 55.2 MPa (6 to 8 ksi). In order to increase the strength of this metal alloy, various treatment methods may be used. In one preferred embodiment, once combined with the metallic fibres, the alloy is heated to 520°C for 5 sixteen hours followed by quenching in water maintained at a temperature of between 60°C to 100°C. The composite is then placed in an oven and maintained at 190°C and maintained at that temperature until the desired strength of the matrix is achieved (typically 0 to 10 days). The matrix has been found to reach a maximum yield strength of 10 68.9 to 89.6 MPa (10 to 13 ksi) when it was maintained at a temperature of 190°C for five days. In contrast, pure aluminium that is not specifically heat treated has a yield strength of 6.9 to 13.8 MPa (1 to 2 ksi) in the as-cast state.

Examples

15 Objects and advantages of this invention are further illustrated by the knowing examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this invention. All parts and percentages are by weight unless otherwise indicated.

Test Methods

20 Fibre strength was measured using a tensile tester (commercially available as Instron 4201 tester from Instron of Canton, MA), and the test is described in ASTM D 3379-75, (Standard Test Methods for Tensile Strength and Young's Modulus for High Modulus Single-Filament Materials). The specimen gauge length was 25.4 mm (one inch), the strain rate was 0.02 mm/mm/min.

25 To establish the tensile strength of a fibre tow, ten single fibre filaments were randomly chosen from a tow of fibres. Each filament was tested to determine its breaking load. At least 10 filaments were tested with the average strength of the filaments in the tow being determined. Each individual, randomly selected fibre had strength ranging from 2.06-4.82 GPa (300-700 kd). The average individual filament tensile strength ranged from 2.76 30 to 3.58 GPa (400-520 ksi).

Fibre diameter was measured optically using an attachment to an optical microscope (Dolan-Jenner Measure Rite Video Micrometer System, Model M25-0002, commercially available from Dolan-Jenner Industries, Inc. of Lawrence MA) at x1000 magnification. The apparatus used reflected light observation with a calibrated stage micrometer.

5 The breaking stress of each individual filament was calculated as the load per unit area.

The fibre elongation was determined from the load displacement curve and ranged from 0.55 % to 1.3 %.

10 The average strength of the polycrystalline α -Al₂O₃ fibres used in the working examples was greater than 2.76 GPa (400 ksi) (with 15 % standard deviation typical). The higher the average strength of the reinforcing fibre, the higher the composite strength. Composites made according to this embodiment of the present invention had a strength of at least 1.38 GPa (200 ksi) (with 5 % standard deviation), when provided with a fibre volume fraction of 60 % (based on the total volume of the composite).

15 **Tensile Testing**

The tensile strength of the composite was measured using a tensile tester (commercially available as an Instron 8562 Tester from Instron Corp. of Canton, MA).

20 This test was carried out substantially as described for the tensile testing metal foils, that is, as described in ASTM E345-93, (Standard Test Methods for Tension Testing of Metallic Foil).

In order to perform tensile testing, the composite was made into a plate 15.24 cm x 7.62 cm x 0.13 cm (6" x 0.05"). Using a diamond saw, this plate was cut into seven coupons (15.24 cm x 0.95 cm x 0.13 cm (6" x 0.375" x 0.05") which were used for testing.

25 Average longitudinal strength (that is, fibre parallel to test direction) was measured at 1.38 GPa (200 ksi) for composites having a matrix of either pure aluminium or pure aluminium with 2 % by weight copper. For composite having a fibre volume content of

30 60 %, average transverse strength (that is, fibre perpendicular to the test direction) was 138 MPa (20 ksi) for composites containing pure aluminium and 262 MPa (38 ksi) for composites made with the aluminium 2 % copper alloy.

Specific examples of various composites metal matrix fabrications are described below.

Example 1 - Preparation of a fibre-reinforced metal composite

A composite was prepared using a tow of NEXTEL 610 alumina ceramic fibres.

5 The tow contained 420 fibres. The fibres were substantially round in cross-section and had diameters ranging from approximately 11 to 13 micrometres on average. The average tensile strength of the fibres (measured as described above) ranged from 2.76 to 3.58 GPa (400 to 520 ksi). Individual fibres had strengths ranging from 2.06 to 4.82 GPa (300 to 700 ksi).

10 The fibres were prepared for infiltration with metal by grinding the fibres into a "preform". In particular, the fibres were wet with distilled water and wound around a rectangular drum having a circumference of 86.4 cm (34 inches) in multiple layers to the desired preform thickness of 0.25 cm (0.10 in).

15 The wound fibres were cut from the drum and stacked in the mould cavity to produce the final desired preform thickness. A graphite mould in the shape of a rectangular plate was used. 1300 grams of aluminium metal

(commercially available as Grade 99.99% from Belmont Metals of Brooklyn, NY) were placed into the casting vessel.

The mold containing the fibers was placed into a pressure infiltration casting apparatus. In this apparatus, the mold was placed into an airtight vessel or crucible and positioned at the bottom of an evacuable chamber. Pieces of aluminum metal were loaded into the chamber on a support plate above the mold. Small holes (5 2.54 mm in diameter) were present in the support plate to permit passage of molten aluminum to the mold below. The chamber was closed and the chamber pressure was reduced to 3 milliTorr to evacuate the air from the mold and the chamber. The aluminum metal was heated to 720°C and the mold (and fibrous preform 10 in it) was heated to at least 670°C. The aluminum melted at this temperature but remained on the plate above the mold. In order to fill the mold, the power to the heaters was turned off, and the chamber was pressurized by filling with argon to a pressure of 8.96 MPa (1300 psi). The molten aluminum immediately flowed through the holes in 15 the support plate and into the mold. The temperature was allowed to drop to 600°C before venting the chamber to the atmosphere. After the chamber had cooled to room temperature, the part was removed from the mold. The resulting samples had dimensions of 15.2 cm x 7.6 cm x 0.13 cm (6" x 3" x 0.05").

20 The sample rectangular composite pieces contained 60 volume % fiber. The volume fraction was measured by using the Archimedes principle of fluid displacement and by examining a photomicrograph of a polished cross-section at 200x magnification.

25 The part was cut into coupons for tensile testing; it was not machined further. The tensile strength, measured from coupons as described above, was 1400 MPa (204 ksi) (longitudinal strength) and 140 MPa (20.4 ksi) (transverse strength).

Example 2-Preparation of Metal Matrix Composite Wires

30 The fibers and metal used in this example were the same as those described in Example 1. The alumina fiber was not made into a preform. Instead, the fibers (in the form of multiple tows) were fed into a molten bath of aluminum and then onto a take-up spool. The aluminum was melted in an alumina crucible having

dimensions of 24.1 cm x 31.3 cm x 31.8 cm (9.5" x 12.5" x 12.5") (commercially available from Vesuvius McDaniel of Beaver Falls, PA). The temperature of the molten aluminum was 720°C. An alloy of 95% niobium and 5% molybdenum was fashioned into a cylinder having dimensions of 12.7 cm (5") long x 2.5 cm (1") diameter. The cylinder was used as an ultrasonic horn actuator by tuning to the desired vibration (i.e., tuned by altering the length), to a vibration frequency of 20.0-20.4 kHz. The amplitude of the actuator was greater than 0.002 cm (0.0008"). The actuator was connected to a titanium waveguide which, in turn, was connected to the ultrasonic transducer. The fibers were infiltrated with matrix material to form wires of relatively uniform cross-section and diameter. Wires made by this process had 10 diameters of 0.13 cm (0.05").

The volume percent of fiber was estimated from a photomicrograph of a cross section (at 200x magnification) to be 40 volume %.

The tensile strength of the wire was 1.03 - 1.31 GPa (150-190 ksi).

15 The elongation at room temperature was 0.7-0.8%.

Elongation was measured during the tensile test by an extensometer.

Example 3-Composite Metal Matrix Materials Using an Al/Cu Alloy Matrix

This example was carried out exactly as described in Example 1, except that instead of using pure aluminum, an alloy containing aluminum and 2% by weight copper was used. The alloy contained less than 0.02% by weight iron, and less than 0.05% by weight total impurities. The yield strength of this alloy ranged from 41.4 - 103.4 MPa (6-15 ksi). The alloy was heat treated according to the following schedule:

20 520°C for 16 hours followed by a water quench (water temperature ranging from 60 -100°C); and

immediately placed into an oven at 190°C and held for 5 days.

25 The processing proceeded as described for Example 1 to produce rectangular pieces to make coupons suitable for tensile testing except that the metal was heated to 710°C and the mold (with the fibers in it) was heated to greater than 660°C.

The composite contained 60 volume % of fiber. The longitudinal strength ranged from 1.38 - 1.86 GPa (200 - 270 ksi) (with the average of 10 measurements of 1.52 GPa (220 ksi)) and the transverse strength ranged from 239 - 328 MPa (35 - 48 ksi) (with an average of 10 measurements of 262 MPa (38 ksi)).

5

Equivalents

Various modifications and alterations to this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention. It should be understood that this invention is not intended to be unduly limited by the illustrative embodiments and examples set forth herein and that such examples and embodiments are presented by way of example only with the scope of the invention intended to be limited only by the claims set forth herein as follows.

Throughout this specification, unless the context requires otherwise, the word "comprise", or variations such as "comprises" or "comprising" or the term "includes" or variations thereof, will be understood to imply the inclusion of a stated element or integer or group of elements or integers but not the exclusion of any other element or integer or group of elements or integers. In this regard, in construing the claim scope, an embodiment where one or more features is added to any of the claims is to be regarded as within the scope of the invention given that the essential features of the invention as claimed are included in such an embodiment.

The claims defining the invention are as follows:

1. A composite material comprising a plurality of continuous polycrystalline α -Al₂O₃ fibers contained within a matrix of aluminium, said matrix being substantially free of material phases or domains capable of enhancing the brittleness of said matrix, wherein said composite material has an average tensile strength greater than 1.17 GPa.
2. The composite material according to claim 1 wherein said plurality of continuous polycrystalline α -Al₂O₃ fibers includes at least one tow of continuous polycrystalline α -Al₂O₃ fibers.
3. The composite material according to claim 2 wherein said polycrystalline α -Al₂O₃ fibers have an average tensile strength of at least 2.8 GPa.
4. The composite material according to claim 3 wherein said aluminium matrix contains less than 0.03 % by weight iron, based on the total weight of said matrix.
5. The composite material according to claim 3 comprising between 40-60% by volume of said polycrystalline α -Al₂O₃ fibers, based on the total volume of said composite material.
6. The composite material according to claim 5 wherein said matrix of substantially pure elemental aluminium has a yield strength of less than 20 MPa.
7. A wire according to claim 5 wherein said matrix of an alloy of aluminium and up to 2% copper has a yield strength of less than 90 MPa.
8. A wire comprising composite material according to claim 2.

9. The wire according to claim 8 wherein said wire has an average tensile strength of at least 1.38 GPa.
10. An overhead high voltage power transmission cable which comprises a plurality of said aluminium matrix composite wires according to claim 8.
11. An overhead high voltage power transmission cable according to claim 10 which further includes at least one conductive jacket comprising a plurality of conductive aluminium or aluminium alloy wires.
12. The composite material according to claim 1 wherein said continuous polycrystalline α -Al₂O₃ fibers are free of an exterior protective coating.
13. A method of making a continuous composite wire, said method comprising the steps of :
 - (a) melting a matrix material selected from the group consisting of aluminium and an alloy of aluminium with up to 2% by weight copper, based the total weight of said alloy matrix, wherein said matrix material contain less than 0.05 percent by weight impurities, based on the total weight of said matrix materials, to provide a contained volume of melted metallic matrix material;
 - (b) imparting ultrasonic energy to cause vibration of the contained volume of melted metallic matrix material of step (a);
 - (c) immersing a plurality of continuous polycrystalline α -Al₂O₃ fibers into said contained volume of melted metallic matrix material while maintaining said vibration to permit the melted metallic matrix material to infiltrate into and coat said plurality of fibers such that an infiltrated, coated plurality of fibers is provided; and

- (d) withdrawing said infiltrated, coated plurality of fibers from said contained volume of melted metallic matrix material under conditions which permit the melted metallic matrix material to solidify to provide a wire comprising composite material according to claim 2.

14. A composite material comprising a plurality of continuous polycrystalline α -Al₂O₃ fibers within a matrix selected from the group consisting of an aluminium matrix and a matrix selected from the group consisting of an aluminium matrix and a matrix of an alloy of aluminium and up to 2% by weight copper, based on the total weight of said alloy matrix, wherein said matrices contain less than 0.05 percent by weight impurities, based on the total weight of said matrices, and wherein said composite material has an average tensile strength of at least 1.17 GPa.

15. The composite material according to claim 14 wherein said plurality of continuous polycrystalline α -Al₂O₃ fibers includes at least one tow of continuous polycrystalline α -Al₂O₃ fibers.

16. The composite material according to claim 15 wherein said polycrystalline α -Al₂O₃ fibers has an average tensile strength of at least 2.8 GPa.

17. The composite material according to claim 16 wherein said aluminium matrix contains less than 0.03 % by weight iron, based on the total weight of said matrix.

18. The composite material according to claim 16 comprising between 40-60% by volume of said polycrystalline α -Al₂O₃ fibers, based on the total volume of said composite material.

19. The composite material according to claim 18 wherein said matrix of substantially pure elemental aluminium has a yield strength of less than 20 MPa.
20. A wire according to claim 18 wherein said matrix of an alloy of aluminium and up to 2% copper has a yield strength of less than 90 MPa.
21. A wire comprising composite material according to claim 15.
22. The wire according to claim 21 wherein said wire has an average tensile strength of at least 1.38 GPa.
23. An overhead high voltage power transmission cable which comprises a plurality of said aluminium matrix composite wires according to claim 21.
24. An overhead high voltage power transmission cable according to claim 23 which further includes at least one conductive jacket comprising a plurality of conductive aluminium or aluminium alloy wires.
25. The composite material according to claim 14 wherein said continuous polycrystalline α -Al₂O₃ fibers are free of an exterior protective coating.
26. A method of making a continuous composite wire, said method comprising the steps of:
 - (a) melting a metallic matrix material selected from the group consisting of aluminium and an alloy of aluminium with up to 2% by weight copper, based on the total weight of said alloy matrix, wherein said

matrix material contain less than 0.05 percent by weight impurities, based on the total weight of said matrix materials, to provide a contained volume of melted metallic matrix material;

- (b) imparting ultrasonic energy to cause vibration of the contained volume of melted metallic matrix material of step (a);
- (c) immersing a plurality of continuous polycrystalline α -Al₂O₃ fibers into said contained volume of melted metallic matrix material while maintaining said vibration to permit the melted metallic matrix material to infiltrate into and coat said plurality of fibers such that an infiltrated, coated plurality of fibers is provided; and
- (d) withdrawing said infiltrated, coated plurality of fibers from said contained volume of melted metallic matrix material under conditions which permit the melted metallic matrix material to solidify to provide a wire comprising composite material according to claim 15.

27. A composite material substantially as hereinbefore described with reference to the Examples and accompanying drawings.

28. A method of making a continuous composite wire substantially as hereinbefore described with reference to the Examples and accompanying drawings.

Dated this 31st day of March, 1999

MINNESOTA MINING AND MANUFACTURING COMPANY

By its Patent Attorneys

DAVIES COLLISON CAVE

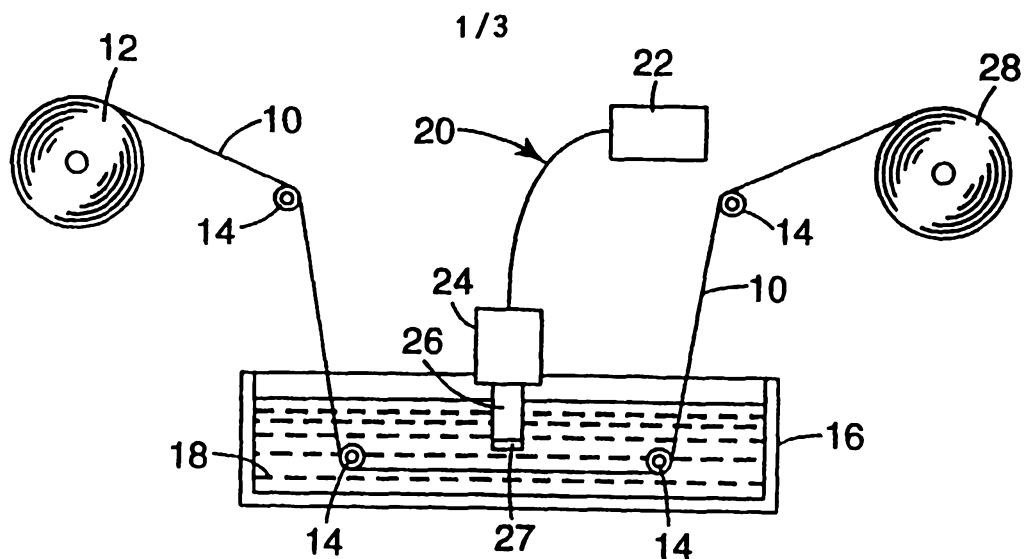


Fig. 1

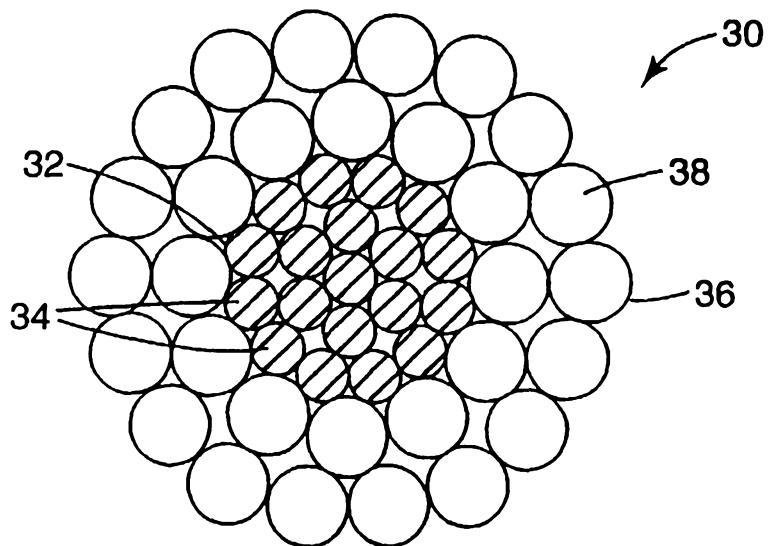


Fig. 2a

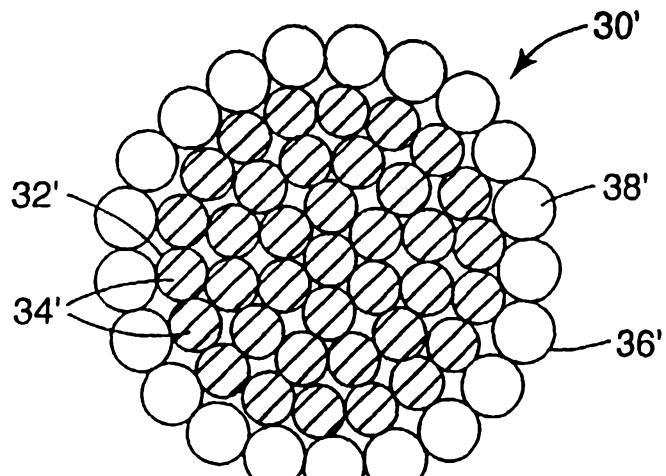


Fig. 2b

2/3

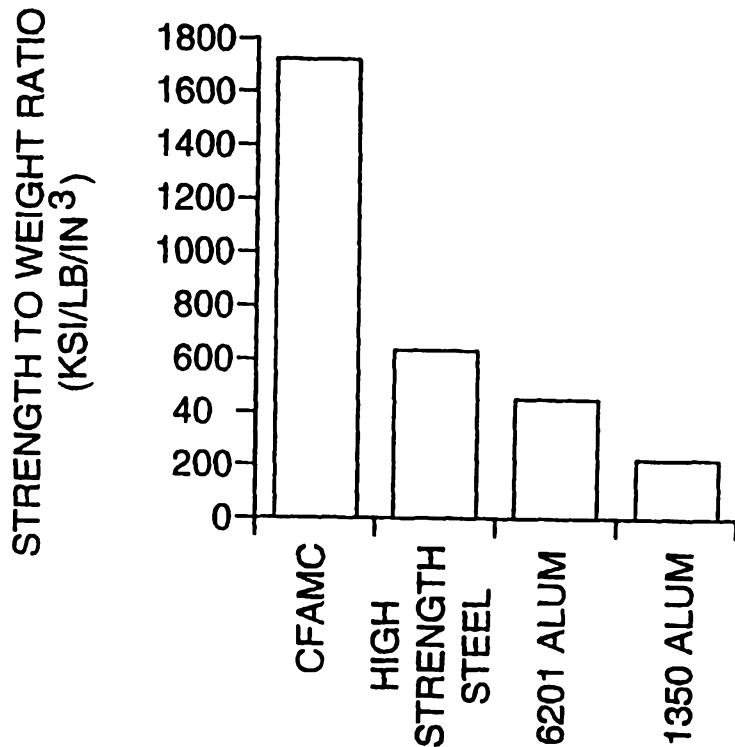


Fig. 3

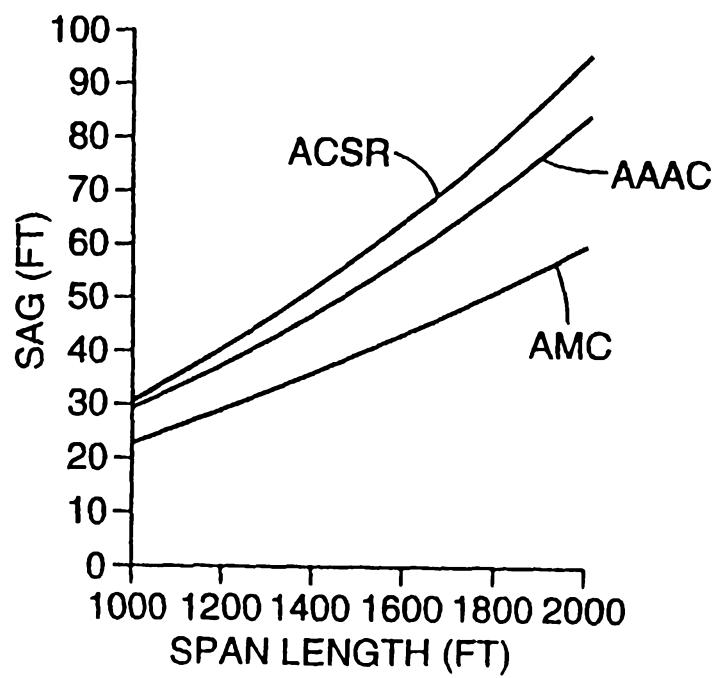
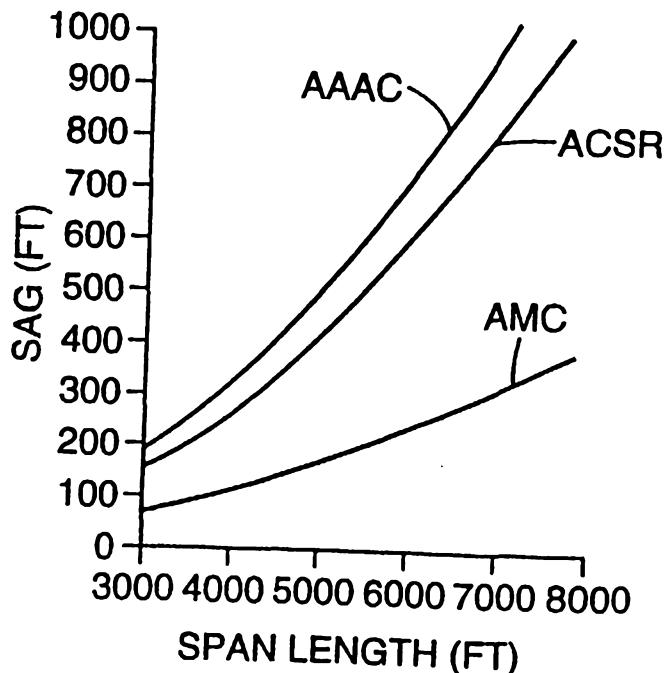
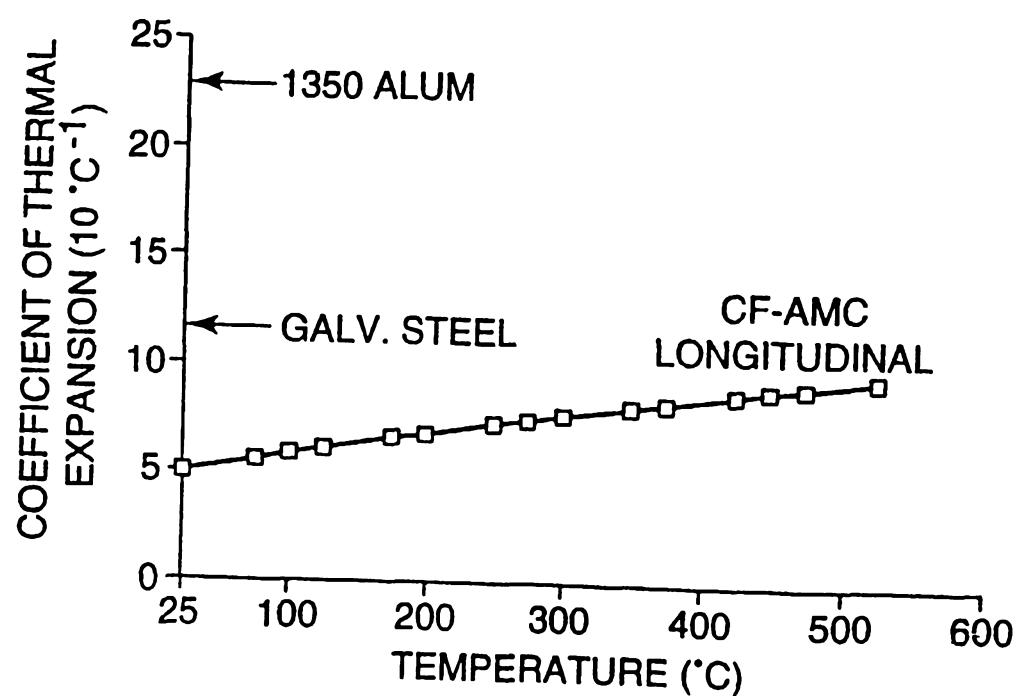


Fig. 4a

3/3

**Fig. 4b****Fig. 5**