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Ahmad

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[54] **PROCESS FOR FORMING COMPOSITE MATERIAL BY ELECTRODEPOSITION UNDER THE INFLUENCE OF A CENTRIFUGAL FORCE FIELD**

[75] Inventor: **Iqbal Ahmad,** Elnora, N.Y.

[73] Assignee: **The United States of America as represented by the Secretary of the Army**

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[51] **Int. Cl.**.....**C23b 5/48,** C23b 7/02, C23b 5/68

[58] **Field of Search**.....204/38 R, 38 B, 181, 26, 16, 204/199, 9; 117/101

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Primary Examiner—John H. Mack
Assistant Examiner—R. L. Andrews
Attorney—Harry M. Saragovitz, Edward J. Kelly, Herbert Berl and Albert E. Arnold, Jr.

[57] **ABSTRACT**

An improvement in the techniques of forming composite material by electrodeposition whereby the electrolyte slurry containing the reinforcing whiskers or fibers is subjected to a centrifugal force field to press the whiskers or fibers against the cathode and form a permeable mat thereagainst for encapsulation by the deposited matrix metal. Thus, the percent by volume of the whiskers or fibers in the deposited composite material can be increased substantially above that which is possible by conventional electrodeposition technique.

9 Claims, 5 Drawing Figures

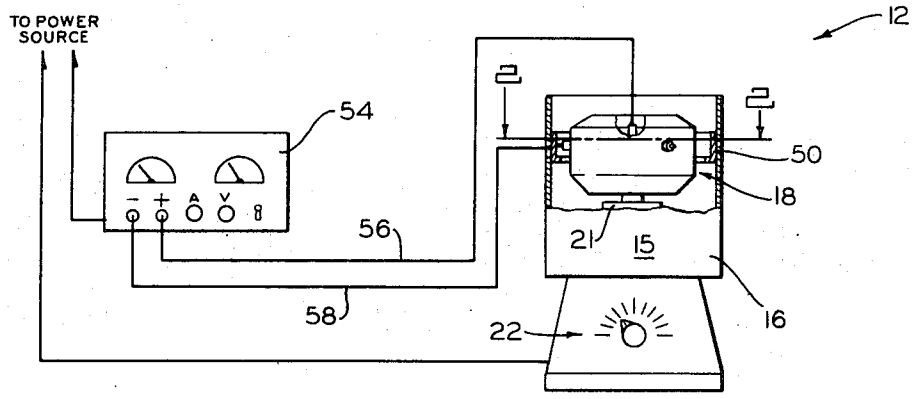


Fig. 1

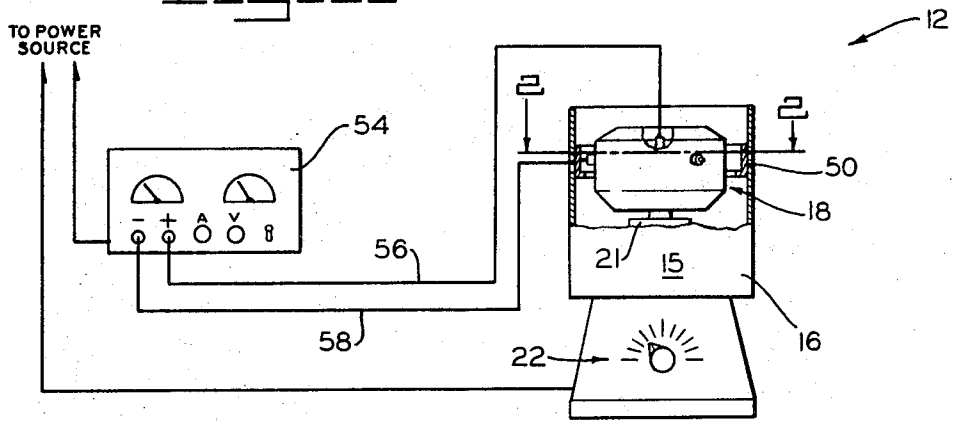
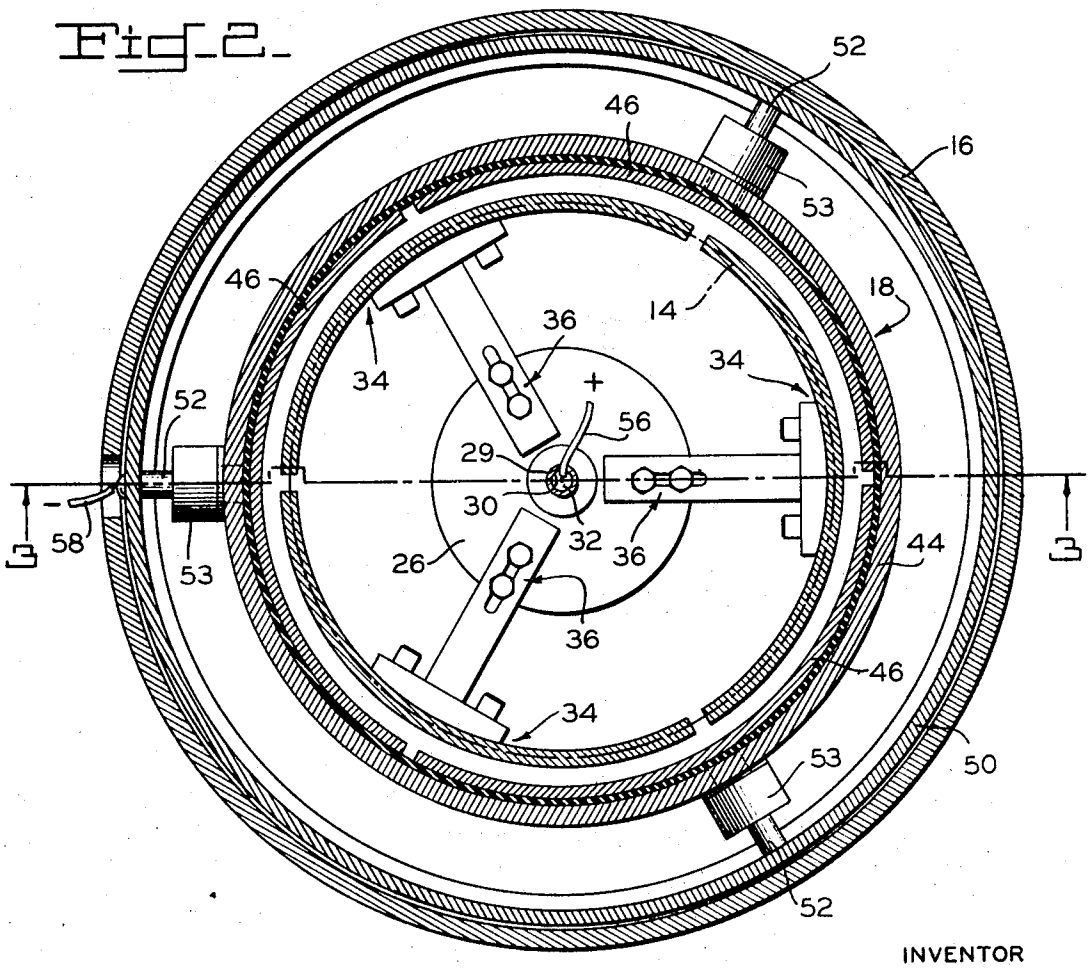
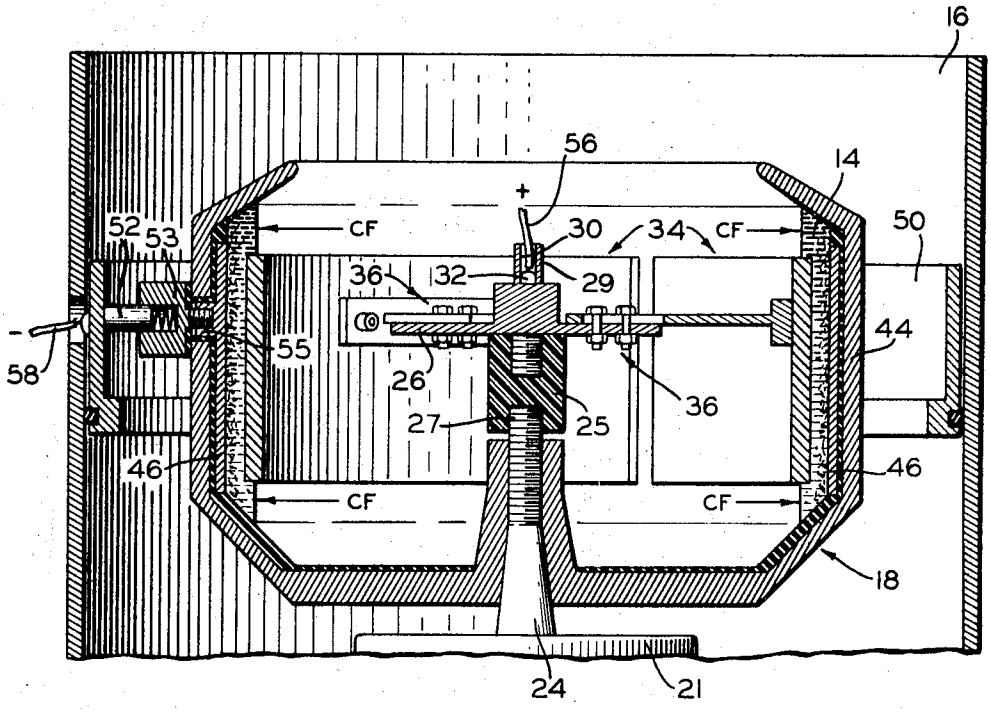


Fig. 2



INVENTOR
Iqbal Ahmad
BY
Harry M. Saragovitz, Edward J. Kelly, Herbert Berl, & Albert P. Zarnoch
ATTORNEYS

Fig. 3



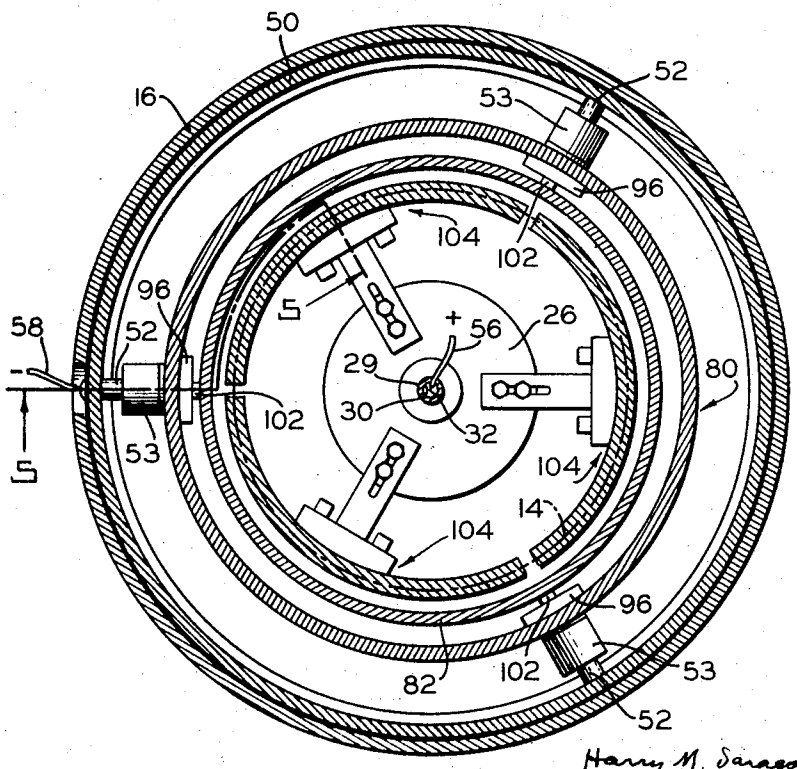
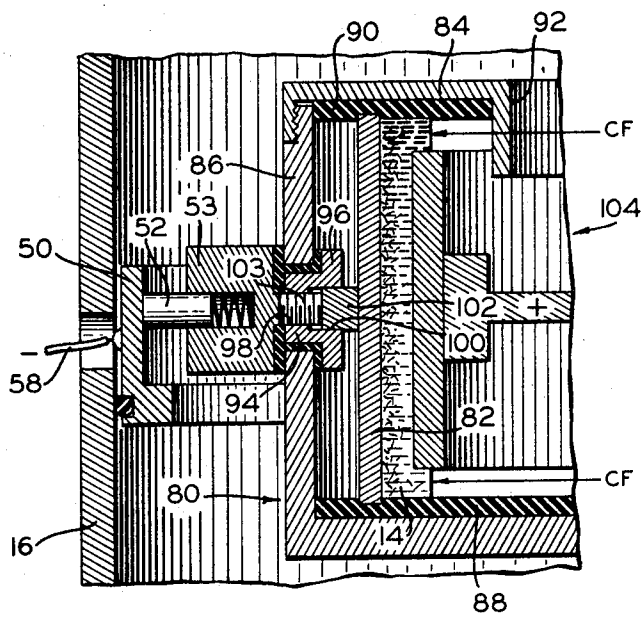


Fig. 4

INVENTOR
Iqbal Ahmad
BY
Harry M. Saragovitz, Edward J. Kelly
Herbert Berl, & Albert E. Arnold
ATTORNEYS

Fig. 5



INVENTOR
Iqbal Ahmad
BY
Harry M. Saragovitz, Edward J. Kelly
Herbert P. Price
ATTORNEYS

PROCESS FOR FORMING COMPOSITE MATERIAL BY ELECTRODEPOSITION UNDER THE INFLUENCE OF A CENTRIFUGAL FORCE FIELD

The invention described herein may be manufactured, used, and licensed by or for the Government for governmental purposes without the payment to me of any royalty thereon.

BACKGROUND OF THE INVENTION

This invention is related to the art of forming composite material through the addition of reinforcing particles to a matrix to improve the properties thereof, and pertains more particularly to the technique of forming these composite materials by electro-deposition.

The phenomenal progress in space science and technology and the developments in weaponry and logistics during the past two decades have created a pressing demand for materials which have a high strength and modulus-to-density ratio, and high-temperature capability. As a result, a large number of super alloys have been developed. These, however, still fall short of some of the rigorous requirements of the advanced engineering concepts.

The success achieved in reinforcing plastics with glass filaments and the availability in recent years of high strength and high temperature metallic and ceramic fibers and especially single crystals, now known in the art as whiskers, have initiated great activity in their utilization for reinforcement of weaker metallic matrices to produce high temperature, high strength and light weight composite materials. Theoretically, for instance, nickel with 50 volume percent alpha Al_2O_3 whiskers will be superior to any of the available high temperature super alloys.

A number of techniques are being explored for the fabrication of these reinforced metal composites. One of these techniques showing considerable promise is that of electrodeposition because in this technique the reinforcing particles such as whiskers, refractory powder or fibers are not subjected to damaging pressures or heat as they are suspended in an electrolyte and are codeposited with the matrix metal at relatively low temperatures and at atmosphere pressure. This technique, however, has limitations as the maximum content of the dispersoids in the matrix is limited to approximately 10 volume percent by the density of the slurry.

SUMMARY OF THE INVENTION

It is a principal object of this invention to provide an improvement in the technique of producing composite material by electro-deposition whereby a greater volume percent of reinforcing particles can be added to the composite material than has been possible before by this technique.

It is another object of this invention to produce composite material by electrodeposition under the influence of a centrifugal force field so as to increase the volume percent of the reinforcing particles in the material.

It is another and still further object of this invention to provide a process for producing composite material by electro-deposition under the influence of a centrifugal force field in which the volume percent of the reinforcing particles is controlled by the amount thereof in the electrolyte, the magnitude of the centrifugal force field and the densities of the slurry and the electrolytic current.

It is still another object of this invention to provide a process for producing composite material by electrodeposition under the influence of a centrifugal force field so that the resulting material is sound, imporous and has a large volume percent of reinforcing particles.

It is a further object of this invention to produce by electro-deposition an aluminum oxide whisker-nickel composite in which the volume percent of aluminum oxide whiskers is greater than 15 percent.

Further objects and advantages of the invention will be apparent from the following specification and the accompanying drawings which are for the purpose of illustration only.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic view of an apparatus which can be used in the process of this invention for forming composite material by electrodeposition under the influence of a centrifugal force field;

FIG. 2 is an enlarged view taken along line 2—2 of FIG. 1;

FIG. 3 is a view taken along line 3—3 of FIG. 2 showing the slurry subjected to the centrifugal force field and displaced thereby upwardly along the wall of the rotating cell and between the anode and cathode;

FIG. 4 is a reduced view similar to FIG. 2 but shows an alternate embodiment in which the apparatus is adapted for plating the composite material on the inside of a cylinder; and

FIG. 5 is an enlarged view taken along line 5—5 of FIG. 4.

DESCRIPTION OF PREFERRED EMBODIMENTS

Shown in FIGS. 1–3 is an electrolytic apparatus **12** for depositing a composite material as arcuate strips from a slurry **14** by electro-deposition under the influence of a centrifugal force field CF. Apparatus **12** comprises a centrifuge **15** having a cylindrical housing **16** and a cell **18** therein. Cell **18** is rotated by a high speed motor **21** to produce centrifugal force field CF. The rate of rotation, and therefore the magnitude of centrifugal force field CF, is controlled by rheostat means **22** which is operationally disposed between motor **21** and a source of power not shown.

Shaft **24** of motor **21**, to which cell **18** is mounted for rotation, extends axially into the cell and a disc **26** is mounted to such extending end **27** by means of an insulating connector **25** for simultaneous rotation therewith. Disc **26** is formed from copper as it is an excellent conductor of electricity, and is provided with an axial hub **29** having a well **30** extending downwardly thereinto to receive mercury pool **32**. Disc **26** carries three nickel anodes **34** which are disposed 120° apart, as shown in FIG. **2**, and parallel to the axis of shaft and to the vertical wall **44** of cell **18**. Anodes **34** are also mounted to disc **26**, as noted at **36**, so as to be radially adjustable, respective to disc **26**.

Mounted to the inside of wall **44** of cell **18** so as to be electrically insulated therefrom are three cathodes **46** which are disposed in radial alignment with anodes **34** and concentric therewith. A collector ring **50** is mounted to the inside of housing **16** and electrical con-

nection is made between each cathode **46** and such ring by means of a carbon brush **52** which is displaceably mounted in a mount **53** so as to be spring-pressed against the ring. Each cathode **46** is provided with a cylindrical boss **55** which extends through a hole in wall **44**, and mount **53** is threadingly engaged with the boss to provide electrical connection between carbon brush **52** and the cathode and also firmly hold the cathode to the wall. Mounts **43** and bosses **55** are, of course, insulated from wall **44**.

DC current is supplied to anodes **34** and cathodes **46** from a source of power (not shown) through a regulator **54** which is adapted for adjusting the voltage and current density output therefrom. Positive lead **56** from regulator **54** terminates in mercury pool **32** so as to apply a positive potential to anodes **34**, through disc **26**, during rotation of cell **18**. Negative lead **58** from regulator **54** is connected to ring **50** so as to apply a negative potential to cathodes **46** through carbon brushes **52**.

Shown in FIGS. **4** and **5** is another embodiment of apparatus **12** wherein 80 **is adapted for plating the inside of a cylinder** 82. **In this embodiment, top** 84 **of cell** 80 **is threadingly mounted on wall** 86 **thereof to permit removal therefrom for insertion of cylinder** 82 **into the cell. Before cylinder** 82 **is inserted, a gasket** 88 **is installed in the bottom of cell** 80 **and after the cylinder is placed on top of such gasket another gasket** 90 **is placed on top of the cylinder. When top** 84 **is installed on wall** 86 **and tightened, a seal is made between cylinder** 82 **and gaskets** 88 **and** 90 **to form a reservoir for slurry** 14 **when poured thereinto through opening** 92 **in top** 84.

Electrical connection is made between negative lead **58** and cylinder **82**, so that it will be made cathodic when current is applied thereto, by means of ring **50** and at least three carbon brushes **52** and mounts **53** therefor as described in the basic embodiment. In addition, provided for each mount **53** is a bushing **94** with an external flange **96**, which is mounted through a hole in wall **86** so as to be insulated therefrom and so that the flange is located on the inside of the wall. Each bushing **94** is provided with a threaded central bore **98** which is counterbored at **100** to slidingly receive a pad **102**. Threaded portion **103** of mount **53** is received by bore **98** so as to act against pad **102**, when tightened, to press the pad against the side of cylinder **82**. Together, bushings **94** cooperate, when tightened, to center cylinder **82** in cell **80**, hold the cylinder against displacement, and provide electrical contact therewith.

Anodes **104** are substantially similar to anodes **34** and, as shown in FIGS. **4** and **5**, sufficient space must be left between the top and bottom of anodes **104** and therebetween to permit the flow of slurry **14** therethrough.

Apparatus **12** is utilized to form strips of aluminum oxide whisker-nickel composite material by the process which comprises the steps of forming slurry **14** by mixing a predetermined amount of aluminum oxide whiskers, as explained later, in an electrolyte. A suitable electrolyte which has been used to advantage is a sulphamate bath which comprises:

Nickel sulphamate	43 oz/gal
Nickel as metal	10 oz/gal
Boric Acid	3/4 oz/gal
Nickel Bromide (conc.)	7 oz/gal

Diluted to a specific gravity of 29–31 (Baume scale).

Cell **18** is energized for rotation at a predetermined speed by rheostat means **22** and slurry **14** is thoroughly mixed to distribute the aluminum oxide whiskers evenly therein. Slurry **14**, while still thoroughly mixed, is poured into cell **18** and immediately, through centrifugal force field CF created by the rotation thereof, the slurry rises along wall **44** and between the facing surfaces of anodes **34** and cathodes **46**, as shown in FIG. **3**, to provide electrical communication therebetween. Through the influence of centrifugal force field CF the aluminum oxide whiskers are moved through the electrolyte against cathodes **46** to form a permeable mat thereagainst. The degree of compactness of the matted whiskers is determined by the magnitude of centrifugal force field CF and therefore this is one of the factors which determines the volume percent of the whiskers in the deposited composite because the greater the whiskers are compacted together the greater will be their volume percent in the resulting composite. It is, however, important that the magnitude of centrifugal force CF is not so great that the whiskers form an impermeable mat against cathodes **46**. If this should happen, the distributing ions would be prevented from penetrating the mat and encapsulating the whiskers therein.

Other factors which control the volume percent of the whiskers, and must be considered in determining the amount thereof to be added to the electrolyte in slurry **14**, are the densities of the slurry, and the current applied to anodes **34** and cathodes **46**. The latter is particularly important because when the current density is low the distributing ions passing to cathodes **46** from anodes **34** pass freely through the matted whiskers to completely encapsulate the whiskers from the inside of the mat out and thereby form a solid, void-free mass. When the current density is too high the depositing ions could form bridges between the whiskers and so create voids in the mass.

When the deposit is formed to the desired thickness the current is shut off, motor **21** is deenergized and cathodes **46** removed from cell **18**. The deposited composite is then stripped from each of cathodes **46**.

In a laboratory set-up wherein cell **18** was 5 inches in diameter, 8 grams of alpha-Al_2O_3 whiskers were added to 150 cc of the sulphamate electrolyte, thoroughly mixed by an electrosonic probe and poured into the cell which was rotating at 1,600 rpm. A 3-volt potential and 26 amp/sq. ft. current density was then applied to anodes **34** and cathodes **46**. This resulted in a deposited strip of composite material on each cathode which was 4 inches long, one-half inch wide and 0.060 inch thick and having a 16.8 volume percent of aluminum oxide whiskers. The highest volume percent of aluminum oxide whiskers which could be obtained without the utilization of centrifugal force was 10 volume percent.

It is obvious that other metals can be used in this same process as the matrix for a composite material. It is also obvious that particles of other refractory materials can be used as reinforcements. Also, that combinations of these particles from different refractory materials can be used to achieve a composite material with improved properties in different fields.

The same process described above can be also used to form a plating on the inside wall of a cylinder as illustrated in FIGS. **4** and **5**.

I wish it to be understood that I do not desire to be limited to the exact details of construction shown and described, for obvious modifications will occur to a person skilled in the art.

I claim:

1. The process of producing by electrodeposition a composite material including a metallic matrix and reinforcing particles, comprising the steps of:

mounting metal collecting cathode means in a rotatable cell concentrically around the outside of anode means radially spaced therefrom;

mixing a predetermined amount of the reinforcing particles in a liquid electrolyte to form a slurry;

placing said mixed slurry into the space between said cathode means and said anode means;

rotating said cathode means, anode means, and slurry simultaneously at a speed sufficient to form and hold a permeable mat of said reinforcing particles against said cathode means by centrifugal force; and

applying an electric current to said anode means and said cathode means while rotating said cathode means, anode means, and said slurry to produce a deposit of said composite material on said cathode means by electrodeposition.

2. The process as defined in claim 1 and including the step of adjusting the speed of the rotation of the cell so as to create a centrifugal force field of sufficient magnitude to produce a desired volume percent of the reinforcing particles in the composite material.

3. The process as defined in claim 1 and including the step of adjusting the current density applied to the anode and the cathode means below that which would cause bridging and the creation of voids in the composite material.

4. The process as defined in claim 1 and including the step of adding particles of more than one reinforcing material to the slurry to improve different properties of the matrix metal.

5. The process as defined in claim 1 wherein the reinforcing particles are in the form of whiskers, fibers or discontinuous filaments.

6. The process as defined in claim 1 wherein the cathode means is in the configuration of a cylinder and the deposited composite material is formed on the inside thereof as a surface plating.

7. The process as defined in claim 1 wherein said cathode means comprises a plurality of arcuate cathodes.

8. The process as defined in claim 1 and including the steps of removing said cathode means after sufficient electrodeposition of composite material, and stripping the collected composite material from said cathode means.

9. The process as defined in claim 1 wherein said composite material is an aluminum oxide whiskers and nickel composite.

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