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UNITED STATES OF AMERICA

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IN REPLY REFER TO:

Attorney Docket No. 83344
Date: 31 December 2003

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Serial Number	10/637,082
Filing Date	8/4/03
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20040112 195

Attorney Docket No. 83344

STAINLESS STEEL-COPPER COMPOSITE MATERIAL

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT (1) PETER J. HARDRO, citizen of the United States of America, employee of the United States Government, a and (2) BRENT STUCKER, citizen of the United States of America, residents of (1) Seekonk, County of Bristol, Commonwealth of Massachusetts and (2) Logan, County of Cache, State of Utah, have invented certain new and useful improvements entitled as set forth above of which the following is a specification.

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I hereby certify that this correspondence is being deposited with the U.S. Postal Service as U.S. EXPRESS MAIL, Mailing Label No. EL578538873US In envelope addressed to: Commissioner for Patents, Alexandria, VA 20231 on 4 August 2003
(DATE OF DEPOSIT)

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4 August 2003
DATE OF SIGNATURE

Attorney Docket No. 83344

STAINLESS STEEL-COPPER COMPOSITE MATERIAL

STATEMENT OF GOVERNMENT INTEREST

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefore.

CROSS-REFERENCE TO RELATED PATENT APPLICATIONS

This patent application is co-pending with one related patent application entitled MOLYBDENUM-COPPER COMPOSITE MATERIAL (Attorney Docket No. 83346), by the same inventor as this application.

BACKGROUND OF THE INVENTION

(1) Field of the Invention

The present invention relates to a stainless steel-copper composite material which may be used to manufacture parts and tools requiring working temperatures up to 1,000 degrees Centigrade and to a method of making the composite material to a desired form using either cold pressing or selective laser sintering.

desired form using either cold pressing or selective laser sintering.

(2) Description of the Prior Art

It is known in the prior art that corrosion resistance of stainless steel powder moldings may be improved by combining the powder before molding with about 8 to 16% by weight of an additive consisting essentially of about 2 to 30 wt% by weight of tin and 98 to 705 by weight of copper and/or nickel. Stainless steel moldings using this composition may be prepared by compacting the powder at high pressure and heating to sintering temperature. Such a composition and a method are illustrated in U.S. Patent No. 4,662,939 to Reinshagen.

It is also known in the prior art to produce a laser-sinterable powder product using a selective laser sintering machine. Such a product is shown in U.S. Patent Nos. 5,342,919; 5,527,877; and 5,648,450, all to Dickens, Jr. et al.

Other powders for use with a laser sintering process are shown in U.S. Patent Nos. 5,733,497 to McAlea et al., 6,245,281 to Scholten et al., and 5,431,967 to Manthiram et al.

U.S. Patent No. 5,870,663 to Stucker et al. illustrates a wear-resistant Zirconium-DiBoride (ZrB_2)-Copper Alloy composite electrode. Wherein the first furnace cycle produces a sintered shaped form which is about 30 vol.% to about 70 vol.% occupied by sintered ZrB_2. Wherein the first furnace cycle comprises heating the desired form room temperature to about 1,300 decrees C to about 1,900 degrees C. Wherein the sintered ZrB_2 is then contacted with a copper alloy comprised of up to about 3 wt.% boron and up to about 10 wt.% nickel. Wherein a second furnace cycle is used to heat the sintered ZrB_2 and copper alloy above the melting point of the copper alloy to infiltrate the ZrB_2 with copper alloy to form a ZrB_2/copper alloy composite electrode.

Despite the existence of these materials, there exists a need for a material that offers the ability to create tools and prototype parts requiring working temperatures up to 1,000 degrees Centigrade.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a composite material that offers the ability to create tools and prototype parts requiring working temperatures up to 1,000 degrees Centigrade.

It is still a further object of the present invention to provide a method for manufacturing the above composite material.

The foregoing objects are attained by the composite and the method of the present invention.

In accordance with the present invention, a composite material is provided which has a stainless steel particulate and an oxygen free copper matrix. The stainless steel is preferably present in an amount of 35% - 65% by volume with the balance being of oxygen free copper.

Also, in accordance with the present invention, a method for manufacturing a stainless steel-copper composite material broadly comprises forming a mixture of stainless steel, phenolic, and wax, forming the mixture into a green form using either a selective sintering process or a cold pressing process, placing the mixture in green form into a furnace, placing the oxygen free copper into the furnace adjacent to the green form, and subjecting the green form and the oxygen free copper to a furnace cycle. During this furnace cycle the wax and phenolic thermoset resin is vaporized and the stainless steel is sintered. Additionally, the sintered stainless steel substrate, which is contacted with copper, is heated above the melting point of the copper which causes the copper to infiltrate the stainless steel substrate, forming the stainless steel-copper composite part.

Other details of the stainless steel-copper composite material, as well as other objects and advantages attendant thereto, are set forth in the following detailed description.

DESCRIPTION OF THE PREFERRED EMBODIMENT(S)

The stainless steel-copper composite material is a particulate composite created from stainless steel, phenolic, wax, and oxygen free copper. The material is manufactured using an indirect selective laser sintering (SLS) process where a stainless steel/phenolic/wax powder mixture is initially sintered into a green form. Upon completion of sintering, the green form is placed through a furnace cycle for de-binding and infiltration of the oxygen free copper.

Stainless steel is a family of iron based alloys that must contain at least 10.5% chromium by weight. The presence of the chromium creates an invisible surface film that resists oxidation and makes the material passive or corrosion resistant, i.e. stainless. This family can be simply and logically grouped into five branches. Each of these branches has specific properties and a basic grade or type. In addition, further alloy modifications can be made to tailor the chemical composition to meet the needs of different corrosion conditions, temperature ranges, strength requirements, or to improve weldability, machinability, work hardening and formability.

420 stainless steel is a high carbon version of martensitic 12% by weight chromium family of stainless steels. It has a higher heat treated strength, hardness and better wear resistance than 410 grade stainless. 420 stainless develops its maximum corrosion resistance when hardened and polished.

The desired material properties of the stainless steel 420 particulate is as follows:

Property	S.S. 420
Density (g/cm^3)	8.03
Tensile Yield Strength (MPa)	290
Ultimate Tensile Strength (MPa)	579
Modulus of Elasticity (GPa)	193
Hardness (Vickers, Gpa)	260
Coefficient of Thermal Expansion (m/m/°C)	16.02E-6
Thermal Conductivity (W/mK)	16
Melting point (°C)	1,385
Maximum Service Temperature (°C)	1,100 – 1,900

The desired stainless steel chemical composition is as follows:

Element	%
Carbon	0.15 min.
Chromium	12 – 14
Iron	Balance
Manganese	1.0 max.
Phosphorus	0.04 max.
Silicon	1.0 max.
Sulfur	0.03 max.

Phenolic is a thermoset synthetic resin generally employed as a molding material for the making of mechanical and electrical parts. There are hundreds of different phenolic molding compounds and in general they have a balance of moderately good mechanical and electrical properties and are generally suitable in temperatures up to 160 degrees Centigrade. The resins are marketed usually in granular form, partly polymerized for molding under heat and pressure which completes the polymerization process, making the product infusible and relatively insoluble.

The desired material properties of the phenolic thermoset resin is as follows:

Property	Phenolic
Density (g/cm^3)	1.1 – 1.3
Tensile Yield Strength (MPa)	52.0
Ultimate Tensile Strength (MPa)	60.0
Ultimate Compressive Strength (MPa)	140.0
Modulus of Elasticity (GPa)	6.0
Hardness	130.0, Rockwell M
Coefficient of Thermal Expansion (m/m/°C)	77.0E-6
Thermal Conductivity (W/mK)	0.2
Electrical Resistivity (Ohm-cm)	5.0E11
Melting point (°C)	---
Maximum Service Temperature (°C)	160

Oxygen free high conductivity copper such as alloy C10100 is produced by the direct conversion of selected refined cathodes and castings under carefully controlled conditions to prevent any contamination of the pure oxygen-free metal during processing. The method of producing oxygen free high conductivity copper insures extra high grade of metal with a copper content of 99.9% by weight. With so small a content of extraneous elements, the inherent properties of elemental copper are brought forth to a high degree. Characteristics are high ductility, high electrical and thermal conductivity, high impact strength, good creep resistance, ease of welding, and low volatility under high vacuum. Some typical uses for Copper Alloy 10100 in the electrical and electronic industries are bus bars, bus conductors, wave guides, hollow conductors, lead-in wires and anodes for vacuum tubes, glass to metal seals and others.

The desired material properties of the oxygen free copper is as follows:

Property	**O_2 Free Cu**
Density (g/cm^3)	8.96
Tensile Yield Strength (MPa)	33.3
Ultimate Tensile Strength (MPa)	210
Modulus of Elasticity (GPa)	110
Hardness (Vickers, Gpa)	49
Coefficient of Thermal Expansion (m/m/°C)	17.64E-6
Thermal Conductivity (W/mK)	346
Melting point (°C)	1,083

To form the composite material of the present invention, a mixture of stainless steel, phenolic, and wax is formed. The mixture may be shaped into the form of a part or a component to be produced. The mixing of stainless steel particles with wax and phenolic thermoset resin particles should be done in a way such that the particles are evenly dispersed. The preferred stainless steel, wax, and phenolic thermoset resin particle size for the selective laser sintering shaping method should be between 10 microns and 145 microns, with an average particle size of between 20 microns and 45 microns. This is because the powder in the selective laser sintering machine is moved by a counter-rotating roller, and this method of powder transfer does not work well for finer powders.

In mixing the particles, the recommended stainless steel, wax, and phenolic thermoset resin mixture is 3% phenolic thermoset resin particles, 2.5% wax particles, balance stainless steel particles, by weight. The proper ratio of wax, phenolic thermoset resin particles, and stainless steel particles has an effect on shrinkage during selective laser sintering of the particle mixture to "tack" together the stainless steel particles and during sintering of the desired form which vaporizes the wax and phenolic thermoset resin and sinters the stainless steel particles.

Where mass production of simple shaped parts is desired, "cold pressing" the mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles is the preferred method of shaping a desired form.

A version of "rapid prototyping" is preferred where the part to be manufactured is of complex or varying topography or where limited numbers of parts are to be manufactured. "Rapid prototyping" is a known technology to facilitate rapid product development. The version of rapid prototyping as disclosed herein is suitable for processing or shaping a mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles into a desired form. This is particularly advantageous for complex or varying topographies.

In rapid prototyping, a 3-D model produced on a computer-aided design (CAD) system is mathematically divided into a large number of thin layers, a few thousandths of an inch thick. The different processes for rapid prototyping generally work on the same basis principle, i.e., the desired part is built up in small layers, about 0.003" thick to about 0.005" thick, one layer at a time, starting from the bottom and working up until the entire part is finished. Thus, the layers are built, and simultaneously consolidated to the preceding layer, using the description of that layer from the computer.

The preferred rapid prototyping technique is "selective laser sintering" ("SLS"). SLS uses a CO_2 laser to sinter a mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles by scanning in the horizontal plane only as dictated by a current layer description in a CAD model. The three dimensional solid is built up by the addition of material layers.

The SLS machine consists of hardware and software components. The hardware components include the process chamber and powder engine, the controls cabinet, and the atmospheric control unit. The process chamber incorporates the laser, pre-heater, and the powder handling equipment. The controls cabinet interprets the CAD drawing and controls and monitors the SLS process. The atmospheric control unit regulates the temperature and amount of N_2 flowing through the air in the chamber. It also filters the air that flows through the process chamber. The software components utilize the UNIX operating system and other DTM Corporation proprietary applications.

The CAD drawing is geometrically modified to horizontally divide the desired form into thin horizontal layers. These layers can be adjusted in thickness, but are typically about 0.003" to about 0.005" in thickness. The thin layers represent sintering planes to be traced by the CO_2 laser. In operation, a layer of a mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles is spread out. When the desired cross section of the layer is traced out by the CO_2 laser, the temperature of the mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles is increased, and the wax and phenolic thermoset resin particles fuse the stainless steel particles together. The part is then lowered in the SLS machine by 0.003" to 0.005" (depending upon the layer thickness), and new layers are added in a similar fashion to form the solid mass. The SLS machine builds the part one layer at a time by creating the bottom layer first, and then adding layers until the part is finished.

The mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles was laser sintered using the DTM SINTERSTATION 2500 plus machine which sinters only the wax and phenolic thermoset resin particles and not the stainless steel particles. As described below, post processing is necessary to vaporize, sublime, or "burn off" the wax and phenolic thermoset resin and sinter the stainless steel particles. After this, the sintered stainless steel substrate, which is porous, is infiltrated with an oxygen free copper. This post-SLS processing generally results in a small shrinkage due to the vaporization of the wax and phenolic thermoset resin and sintering of the stainless steel particles. By holding the processing variable constant, this shrinkage may be compensated for in the CAD design of the part, i.e., the CAD design provides for a slightly larger stainless steel, wax, and phenolic shaped form, such that upon shrinkage, the stainless steel-copper composite part will be the desired size.

The CO_2 laser used in the SLS machine is generally only capable of producing enough heat to fuse low-melting thermoset synthetic resin such as phenolic; as such, it is these and similar low-melting point materials, such as wax, which are used to mix with the stainless steel particles when the SLS process is employed. Additionally, the wax and phenolic thermoset resin used must suitably vaporize or sublime in the vaporization step prior to sintering the stainless steel particles.

The desired parameters for SLS shaping of a mixture of stainless steel particles, wax particles, and phenolic thermoset resin particles to the desired form are as follows:

Layer thickness: 0.003 inches
Right and left feed heater temperature: 55 degrees C
Part heater set point: 100 degrees C
Laser power: 35 Watts
Scan spacing: 0.003 inches
Scan speed: 150 inches per second

After sintering or cold pressing has been completed, the green form mixture is placed on an aluminum oxide plate which is located in a graphite crucible. Oxygen free copper is placed on top of tabs, which are also formed from the stainless steel, phenolic, wax mixture, that are adjacent to the green form. The amount of oxygen free copper to be used is 0.67 x green weight including the green form and the tabs. The oxygen free copper is placed on the tabs and the entire green form, tabs, and oxygen free copper infiltrant material is then covered with aluminum oxide in particulate form. The crucible is then placed in a furnace with a process gas of 100% nitrogen and a process pressure of 750 Torr. The furnace cycle is room temperature (approximately 68 degrees Fahrenheit) to 1,150 degrees centigrade in 9 hours; hold at 1,150 degrees centigrade for 1 hour; and then cool down from 1,150 degrees Centigrade to room temperature in six hours. During this single furnace cycle, vaporization of the wax and phenolic binder, sintering of the stainless steel particulate, and infiltration of the sintered stainless steel particles with oxygen free copper are accomplished. The vaporization step may be referred to by those skilled in the art as "burn-out"; however, this terminology is somewhat misleading in that it is preferred that substantially no oxygen be present during the sintering step. Oxygen present in the sintering step may lead to reduced wetting in the copper infiltration step.

Vaporization and sintering produces a sintered stainless steel shaped form that is about 35 volume % to about 65 volume %

occupied by sintered stainless steel, i.e., about 35% to about 65% dense. The density may advantageously be varied, within these limits, depending upon the desired application. The density or porosity may be altered by varying the size or size distribution of the stainless steel particles used, varying the size or size distribution of either the wax or phenolic thermoset resin particles used, varying the particle mixture ratio used, and/or varying the manufacturing technique, etc. The density or porosity determines the stainless steel-copper ratio and may be optimized to meet specific objectives.

During the furnace operation the oxygen free copper is heated above its melting point (1,083 degrees C), such that by capillary action, the copper infiltrates into the open area of the sintered stainless steel particles to produce the stainless steel-copper composite in the desired form with an about 100% density. The resulting mixture is a stainless steel-copper composite with a volume fraction of stainless steel of between 35% and 65% with the balance oxygen free copper.

The composite material of the present invention is unique in that it offers the ability to create tools and prototype parts requiring working temperatures up to 1000 degrees Centigrade. A wide variety of parts may be made from the composite material of the present invention and the method of the present invention.

It is apparent that there has been provided in accordance with the present invention a stainless steel-copper composite material which fully satisfies the objects, means, and advantages set forth hereinbefore. While the present invention has been described in the context of specific embodiments thereof, other alternatives, modifications, and variations will become apparent to those skilled in the art having read the foregoing description, Accordingly, it is intended to embrace those alternatives, modifications, and variations as fall within the broad scope of the appended claims.

Attorney Docket No. 83344

STAINLESS STEEL-COPPER COMPOSITE MATERIAL

ABSTRACT OF THE DISCLOSURE

The present invention relates to a stainless steel-copper composite material. The composite material is formed by forming a mixture of stainless steel, phenolic, and wax, laser sintering the mixture to form a green form, placing the green form and oxygen free copper into a furnace, and subjecting the green form and oxygen free copper to a furnace heating cycle.