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(54) Title: METHOD FOR PRODUCING METAL MATRIX COMPOSITES

(57) Abstract: A method for manufacturing and producing metal alloy composite products from particulate starting materials, particularly adapted for use with aluminum and aluminum alloy wrought products and their manufacture involving the use of powdered starting materials. The method includes the basic steps of introducing elemental, master alloy or prealloyed metal powders and reinforcement powders, whiskers and fibers into a commercial blender and suitably blended, compacting the blended powder into pellets, introducing and compacting the pellets into a die, forming the compacted powder into a billet by vacuum-hot press or by cold isostatic press/sinter, and removing the billet from the die for subsequent processing.

METHOD FOR PRODUCING METAL MATRIX COMPOSITES

TECHNICAL FIELD

The present invention generally relates to the field of manufacturing and producing metal alloy composite products from particulate starting materials. More particularly, the present invention relates to methods of manufacturing and producing metal alloy composite products containing reinforcement materials. The present methods are particularly adapted for use with aluminum and aluminum alloy wrought products and their manufacture involving the use of powdered starting materials.

BACKGROUND ART

In recent years, powder metallurgy has been gaining increasing attention in efforts to obtain new microstructures and improved mechanical properties in alloys of aluminum. In adapting powder metallurgy to the manufacture of wrought products, the idea has been one of consolidating a powder to form a basic workpiece, which often corresponds to the usual ingot or billet. Ideally, the workpiece can then be hot worked according to conventional methods to produce the desired wrought mill product, such as an extrusion, or forging or sheet.

To produce a workpiece of suitable quality, it has been recognized that the powder should be consolidated in a closely controlled manner. Care is needed, for example, to minimize internal workpiece contamination by oxidation, hydration or other reactions with extraneous elements or compounds. Further, in the known art of processing aluminum powder alloys is the stringent care that has been exercised in order to minimize internal workpiece porosities, particularly porosities which trap gases at any significant pressure.

A problem, which is unique to aluminum powder metallurgy, is the inevitable formation of oxides on the powder. These oxides form as thin films, on the order of several nanometers in thickness, on the surfaces of the individual particles. Unlike the oxides which form on other metals used in powder metallurgy, such as copper, iron and their alloys, the oxide film on aluminum or magnesium cannot be reduced to metal in situ. The oxide film on aluminum and its alloys consists almost exclusively of aluminum and magnesium oxides and their hydrates. These aluminum or magnesium oxide films inhibit the particle-to-particle bonding necessary in forming both good compacts and final products of acceptable ductility and toughness as well as strength. Thus, the formation of aluminum powder products is far more difficult and technically completely distinct from the powder metallurgy of metals with in-situ reducible oxides, such as copper, iron and their alloys.

According to a common prior art practice that has been used in the powder metallurgy industry for about 30 years, consolidation of aluminum powders is carried out in the following manner. First, a porous compact is formed by cold isostatic pressing the powder to about 70% of the theoretical density of the alloy being used. The loading of the blended powder into the isostatic rubber mold is a dangerous operation that must be carried out in a controlled manner that eliminates the escape of powder that might form an explosive cloud of metal powder. Then the compact is encapsulated in a closely-fitting aluminum-alloy container or can. For degassing purposes, air is then evacuated from the can and the compact is heated to about 520degree C for about 6 to 7 hours in a high vacuum. While this temperature and vacuum continue to be maintained inside the can, the canned compact is sealed and then compressed to full density at pressures above about 140 MPa. (620 MPa is generally used.) The compact is then cooled and the container is machined away to expose uncontaminated but fully consolidated billet. Removal of the container is necessary but costly step, since the container is typically formed from an alloy that is compositional different than the powder blend used to form the billet. In addition, since the container typically buckles during compaction, the final machined billet size is often greatly reduced compared to the initial diameter of the consolidated powder to insure that all of the container is removed, resulting in reduced

product recovery. The billet is then heated and extruded in a conventional manner to produce a wrought product or is otherwise hot worked as by forging.

Several variations of this practice were disclosed in United States Patent No. 4,104,061 issued to Roberts. During heat-up of the compact for degassing, the container may be evacuated by vacuum, back filled with a depurative gas such as dry nitrogen, and again vacuum evacuated to facilitate the overall degassing process. In addition, the powder can be packed directly into the container, whereby the initial step of cold forming a compact is simply omitted. The hot consolidating step cannot be omitted.

Thus, in the foregoing practice and variations thereof, the use and removal of a container and the use of a vacuum are seen as essential and costly steps.

It has also been known to place aluminum alloy powder in a vacuum hot press, degas the powder as placed in the press, and then hot press the powder to a near solid mass. This process is expensive because of the complex equipment used and the low production rate. Degassing is oftentimes difficult since usually only one end of the powder column is exposed to the vacuum. In vacuum hot pressing the intent is to hot press to 100% density; the result usually is a product of density 96% or more of theoretical.

The manufacture of particle or whisker reinforced aluminum alloys requires the blending of the components. Several methods for the blending of the components have been introduced. For example, United States Patent Nos. 4,557,893 and 4,623,388, both issued to Jatkat *et al.*, described a high energy ball mill technique to ensure blending of a malleable matrix and particles of reinforcing material. United States Patent No. 4,749,545 issued to Begg *et al.* described high energy ball milling of at least 40 volume percent hard reinforcing phase with either aluminum or magnesium alloys. The mixes are consolidated by hot isostatic pressing in this processing technique. United States Patent No. 4,946,500 issued to Zendalis *et al.* and United States No. 4,722,751 issued to Akechi described a method for high energy ball milling rapidly solidified aluminum alloy and reinforcing particles with a reinforcement range of 0.1 to 50 volume percent.

However, these prior art methods have size limitations due to the ball milling operation. These prior art manufacturing methods also have cost implications due to the high energy ball milling and have not been successfully scaled-up for commercial production. Other commercial blending methods have also been employed in the production of aluminum matrix composites.

United States Patent No. 5,561,829 issued to Sawtell *et al.* described the relaxation of the vacuum requirement for the removal of the trapped gasses. It described a process of heating the compacted "green" preform to a processing temperature in a protective atmosphere. The preform is then hot worked. The hot working is critical because the oxide boundaries from the original powder particles are broken and incorporated into the metal matrix during this operation. This practice appears to be viable for small compacts used for direct forging or small-scale extrusion.

United States Patent No. 5,486,233 issued to Carden (hereafter referred to as "*Carden*") described the cold isostatic compaction-vacuum sinter method for production of aluminum matrix composites. This process results in lower production costs due to higher use of the starting material and fewer processing steps for specific size billets that can be processed directly into final product. A process requirement of substantial metal working to transform the powder-metallurgy billet into a wrought product in order to develop high mechanical properties limits the size of the products made by this process. Billets with a diameter of less than 4.5 inches and a length of less than 12 inches have been successfully manufactured by this process. A reinforcement limitation of approximately 25 volume percent has been found with commercial cold isostatic press equipment. Powder blends with greater than approximately 25 volume percent reinforcement require pressures greater than the capacity of commercial isostatic presses, 80,000 psi, for compaction to the desired "green" density of between 88 and 94 percent theoretical. Billets manufactured by the cold isostatic press/sinter process with reinforcement contents at or above 25 volume percent have interconnected porosity and internally oxidize during re-heating for extrusion or other metalworking operation.

United States No. 5,965,829 issued to Haynes *et.al.* (Hereafter "*Haynes*") described a variation of the composite taught by *Carden*. In *Haynes*, the reinforcement is limited to boron carbide with a tight particle size distribution. The body of the patent describes a cold isostatic press and vacuum sinter process that is similar to that of *Carden*. The body of the patent further describes extrusion processes that are common to industry, that can also be applied to metal matrix composites. However, the claims concentrate on the geometric spacing of the boron carbide and the boron-10 isotope content of the boron carbide and the chemical composition limits for secondary elements in the boron carbide rather than the manufacturing methods. Commercial cold isostatic presses are available to manufacture billets that are approximately 8 inch in diameter by 36 inch long.

In summary, all above-described prior art processes of metal matrix composites appear to involve the following sequential steps:

- (1) elemental, master alloy or prealloyed metal powders and reinforcement powders, whiskers or fibers are introduced into a blender and suitably blended;
- (2) the blend is loaded into a die and compacted;
- (3) the compacted powder is formed into a billet by vacuum-hot press or by cold isostatic press/sinter; and
- (4) the billet is removed from the die for subsequent processing.

These processes typically also include the step of loading blended powder into a mold. Due to the nature of the operation, this powder loading operation is very difficult to seal completely and some metal powders always escape to air. This step is harmful to the operators and to environment and is inefficient. The step is also a dangerous operation for aluminum powder. The escape of aluminum powder might form an explosive cloud of aluminum powder.

Therefore, it is desirable to provide a method for consolidating metal powder, particularly aluminum powder, which results in mechanical and physical properties

comparable to those produced by the above-described prior art processes. It is also desirable to provide a method for handling fine metal powder such as aluminum powder blends in a safe, environmental friendly and efficient manner. It is further desirable to provide a method for consolidating metal powder such as aluminum powder in a cost effective and scaleable manner.

DISCLOSURE OF INVENTION

The present invention is a novel and unique method of manufacturing and producing metal matrix composites.

It is a primary object of the present invention to provide a method for consolidating metal powder, particularly aluminum powder, which results in mechanical and physical properties comparable to those produced by the above-described prior art processes.

It is also an object of the present invention to provide a method for handling fine metal powder such as aluminum powder blends in a safe, environmental friendly and efficient manner.

It is another object of the present invention to provide a method for consolidating metal powder such as aluminum powder in a cost effective and scaleable manner.

Basically, the present invention concerns powder metallurgy and is aimed at making a billet or similar workpiece, which is suitable for being hot-worked to produce a wrought metal product.

In a preferred embodiments, the basic process of the present invention method includes the following sequential steps:

- (1) elemental, master alloy or prealloyed metal powders and reinforcement powders, whiskers and fibers are introduced into a commercial blender and suitably blended;
- (2) the blended powder is compacted into pellets;
- (3) the pellets are introduced into a die and compacted;
- (4) compacted powder is formed into a billet by vacuum-hot press or by cold isostatic press/sinter; and
- (5) the billet is removed from the die for subsequent processing.

As discussed earlier, one of the most time consuming and dangerous procedures in the powder process method for manufacture of metal matrix composites involves the introduction of the blended powder into dies for compaction and further processing to produce billets.

Blended fine metal powders have very low densities, typically less than 30% of theoretical. Moving the blended powder often results in the formation of clouds of the powder in the area around the area of movement. Aluminum powder in a dust cloud condition becomes explosive with sufficient amount of aluminum particles in the air and the presence of an ignition source, such as a static electrical charge.

This safety concern dictates that all fine aluminum powder be handled in a way to minimize the explosive hazard of such powder.

The present invention deals with the blended powder immediately after the blending operation. The powder is rendered safe by being compacted into pellets that are too large to escape to air, yet small enough to be poured easily into appropriate containers. Unlike loading blended powders to a die, moving powders from powder containers to blending machine and then to pelletizing equipment can be well sealed. Therefore, the present invention allows the manufacturing process of metal matrix composite become dustless. The present invention also increases the efficient of loading blended powder to a die and make it possible to automate whole powder handling process without generating metal dust.

Further novel features and other objects of the present invention will become apparent from the following detailed description and discussion.

MODES FOR CARRYING OUT THE INVENTION

Although specific embodiments of the present invention will now be described with reference to the drawings, it should be understood that such embodiments are by way of example only and merely illustrative of but a small number of the many possible specific embodiments which can represent applications of the principles of the present invention. Various changes and modifications obvious to one skilled in the art to which the present invention pertains are deemed to be within the spirit, scope and contemplation of the present invention.

A typical way to process blended powder into pellets is to use a commercial palletizing machine. Blended powder is mechanically fed to the gap between two counter rotating rolls with pellet shaped depression in the roll surfaces. The powder is compacted as the rolls rotate to the point of closest approach. The compacted pellets and uncompact powder is released on to a series of screens as the rolls rotate past the point of closest approach. Pellets are separated by the screens and the powder that passes through the screens is recycled through the compacting rolls. A commercial device that performs these operations Chilsonator® manufactured by the Fitzpatrick Company in Elmhurst, Illinois.

After the pellets being loaded into a die and compacted, a standard solidifying process of powder metallurgic manufacturing process of metal matrix composite is applied to produce a final MMC billet.

Manufacturing samples are given for helping to understand the invention. Example 1 is a sample of a typical prior art process. Examples 2 to 5 are samples of the present invention.

EXAMPLE 1 (PRIOR ART)

Powder blend of 6092 aluminum is blended with boron carbide particles in a 85 volume percent aluminum 15 volume percent boron carbide composition. The blended

powder is transferred to a metal die and compacted to a theoretical density of 65 percent. The die is 125 mm (8 inches) in diameter and approximately 500 mm (20 inches) tall. The filling of the die must be done slowly to maintain a safe working environment. This process takes approximately 4 hours to complete. The die and powder is placed in a vacuum retort and heated to an elevated temperature under vacuum and pressed to greater than 97 percent theoretical density. The billet is heated and extruded to a plate with an extrusion ratio of 20:1. Tensile tests are conducted on samples oriented in the extrusion direction and 90 degrees from the extrusion direction. Samples are taken from the start of the extrusion, in the middle of the extrusion and at the end of the extrusion. The test data are contained in Table 1.

EXAMPLE 2

Powder blend of 6092 aluminum is blended with boron carbide particles in a 85 volume percent aluminum 15 volume percent boron carbide composition. The blended powder is introduced into a mechanical device that contains roll dies that densifies the powder into pellets that vary in size from approximately 3 mm to 15 mm in diameter. The particles that are smaller than 3 mm are passed through the rolls another time to produce the proper sized pellets. The pellets are placed in storage drums, 55 gallon shipping drums or equivalent. The pellets are introduced into a metal die and are further compacted to a density of approximately 65 to 80 percent. Add additional pellets to fill the die cavity. The die and powder is placed in a vacuum retort and heated to an elevated temperature under vacuum and densified to greater than 97 percent theoretical density. The billet is heated and extruded to a plate with an extrusion ratio of 20:1. Tensile tests are conducted on samples oriented in the extrusion direction and 90 degrees from the extrusion direction. Samples are taken from the start of the extrusion, in the middle of the extrusion and at the end of the extrusion. The test data are similar to those from the extrusion made from the standard processing, Table 1. Two important features were observed, 1, the strain at failure was not altered by the pelletized powder and 2, the fracture surface showed no evidence of the prior pelletized powder.

TABLE 1

Tensile Test Data for 6092/B4C/15p Composite Heat Treated to T-6P Condition

	Yield Strength MPa (ksi)	Ultimate Strength MPa (ksi)	Strain at Failure (%)	Hardness Rockwell (B)
<u>Extrusion Lead</u>				
Longitudinal				
Average	367(53.3)	452(65.5)	8.26	79.21
Std. Dev.	10.6(1.54)	7.02(1.02)	1.11	1.08
Transverse				
Average	376(54.5)	448(65.0)	6.14	78.99
Std. Dev.	5.0(0.73)	7.4(1.07)	1.26	1.11
<u>Extrusion Middle</u>				
Longitudinal				
Average	372(53.9)	453(65.7)	8.28	79.99
Std. Dev.	7.9(1.14)	4.6(0.66)	0.90	0.70
Transverse				
Average	370(53.6)	445(64.6)	7.64	78.49
Std. Dev.	4.5(0.65)	3.4(0.49)	0.79	1.19
<u>Extrusion End</u>				
Longitudinal				
Average	372(53.9)	445(64.6)	7.45	78.86
Std. Dev.	7.4(1.07)	6.6(0.96)	1.36	0.87
Transverse				
Average	374(54.3)	445(64.6)	7.00	78.67
Std. Dev.	9.0(1.31)	7.4(1.07)	1.42	0.94

EXAMPLE 3

The pellets produced in example 2 are introduced into a rubber mold made to produce 89 mm (3.5 inch) diameter billets, the mold is sealed and evacuated and cold compacted to a theoretical density of approximately 90%. The compacted "green" billet is then vacuum-sintered. The sintered billet is heated to approximately 425 degrees C (800 degrees F) and extruded to a rod with a diameter of 14.3 mm (0.560 inch), the area ratio of the extrusion is 44:1. The rod is machined into tensile samples, heat treated and tested. The test data is similar to that from the standard process with a small increase in strain to failure, which is attributed to the higher level of extrusion, 44:1 verses 20:1. The fracture surfaces also show no evidence of prior pelletized powder.

EXAMPLE 4

The pellets produced in example 2 are blended with aluminum pellets prior to being introduced into the die body. The die and powder is placed in a vacuum retort and heated to an elevated temperature under vacuum and densified to greater than 97 percent theoretical density. The resulting billet is hot extruded to produce a material with improved impact resistance.

EXAMPLE 5

Pellets produced in example 2 are placed in a die in a layer. Pellets with other SiC contents are also produced. Pellets containing 20 volume percent SiC are added as a layer above the original 15 volume percent SiC pellet layer. Additional layers of pellets containing different amounts of SiC and aluminum alloy are added to the die. The die and powder is placed in a vacuum retort and heated to an elevated temperature under vacuum and densified to greater than 97 percent theoretical density. The resulting billet is hot extruded to produce a material with graded properties. In this example, pellets containing between 15 and 40 volume percent SiC are added to produce a material with elastic modulus variations between 96 and 138 MPa (14 and 20 Msi) along with a coefficient of thermal expansion variation between 19 and 12 ppm/ C along the length of the extrusion. Material with other gradient properties can also be produced by combining appropriate pellets with different reinforcement contents.

It is also to be appreciated that although the invention has been described in terms of invention has been described in terms of a preferred embodiment in which the metal is an aluminum alloy, other metals may also be used. The invention will also find special utility with regard to magnesium alloys. In addition, alloys of copper, steel, titanium and nickel may also be used in the present invention. Furthermore, what is believed to be the best mode of the invention has been described above. However, it will be apparent to those skilled in the art that numerous variations of the type described could be made to the present invention without departing from the spirit of the invention. The scope of the present invention is defined by the broad general meaning of the terms in which the claims are expressed.

Of course the present invention is not intended to be restricted to any particular form or arrangement, or any specific embodiment, or any specific use, disclosed herein, since the same may be modified in various particulars or relations without departing from the spirit or scope of the invention hereinabove shown and described of which the apparatus or method shown is intended only for illustration and disclosure of an operative embodiment and not to show all of the various forms or modifications in which this invention might be embodied or operated.

The present invention has been described in considerable detail in order to comply with the patent laws by providing full public disclosure of at least one of its forms. However, such detailed description is not intended in any way to limit the broad features or principles of the present invention, or the scope of the patent to be granted.

THE CLAIMS

1. A method of producing metal matrix composites, comprising the steps of:
 - a. blending metal powders in a blender;
 - b. pelletizing said blended metal powders into pellets;
 - c. loading said metal powder pellets in a die; and
 - d. pressing said metal powder pellets into metal billits.
2. The method as claimed in Claim 1, wherein said metal powders includes elemental metal powders.
3. The method as claimed in Claim 1, wherein said metal powders includes master alloy metal powders.
4. The method as claimed in Claim 1, wherein said metal powders includes pre-alloyed metal powders.
5. The method as claimed in Claim 1, wherein said blending step further comprises the step of blending reinforcement powders with said metal powders.
6. The method as claimed in Claim 1, wherein said blending step further comprises the step of blending whiskers with said metal powders.
7. The method as claimed in Claim 1, wherein said blending step further comprises the step of blending fibers with said metal powders.
8. The method as claimed in Claim 1, wherein said loading step further comprse the step of compacting said metal powder pellets in said die.
9. The method as claimed in Claim 1, wherein said pressing step is performed by vacuum-hot press.

10. The method as claimed in Claim 1, wherein said pressing step is performed by cold isostatic press.
11. The method as claimed in Claim 1, further comprising the step of removing said billets from said die for subsequent processing.

INTERNATIONAL SEARCH REPORT

International application No.

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A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :B22F 1/00, 1/02, 3/12

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According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 419/10, 32

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST: pellet\$5 and "metal powder?" and press\$3

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3,785,801 A (BENJAMIN) 15 January 1974, col. 3, line 59 to col. 4, line 47.	1-11
Y	GB 2042376 A (HASPEL et al) 24 September 1980, page 1, line 24-79.	1-11
A	US 4,097,415 A (MOULIN et al) 27 June 1978.	
A	US 4,141,719 A (HAKKO) 27 February 1979.	
A	US 6,248,150 B1 (AMICK) 19 June 2001.	

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		

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