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(54) Title: CERAMIC PREFORM AND METHOD



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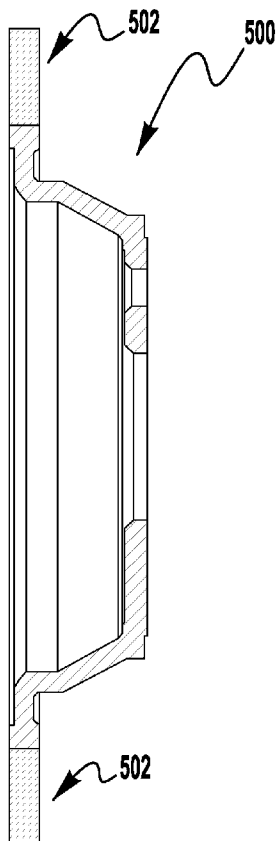


FIG. 5B

(57) Abstract: The present application discloses a ceramic preform, a method of making a ceramic preform, a MMC comprising a ceramic preform, and a method of making a MMC. The method of making a ceramic preform generally comprises preparing reinforcing fibers, preparing a ceramic compound, and forming the compound into a desired shape to create the ceramic preform. In certain embodiments, the ceramic compound is formed as either a disc or a ring for use in a brake disc metal matrix composite. The metal matrix composite generally comprises the ceramic preform infiltrated with a molten metal to form the brake disc metal matrix composite. The method of making the metal matrix composite generally comprises heating the ceramic preform, placing the ceramic preform in a mold cavity of a die cast mold, and introducing molten metal into the mold cavity to infiltrate the ceramic preform to form the brake disc metal matrix composite.

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CERAMIC PREFORM AND METHOD

Cross Reference to Related Application

[0001] This application is a U.S. Non-Provisional Application which claims priority to U.S. Provisional Patent Application No. 62/066,057, filed on October 20, 2014 and titled "Ceramic Preform and Method," which is hereby incorporated by reference in its entirety.

Background

[0002] A metal matrix composite (MMC) is generally made by incorporating a reinforcing material into a metal matrix. For example, a MMC may comprise a ceramic preform that is infiltrated with a metal. A MMC generally has properties and physical characteristics different from metal that may be desirable depending on the application. Other methods for manufacturing MMC's for brake rotors are hampered by the high manufacturing and material cost of the product and inferior performance of the reinforced brake rotor at high temperature. There is also a need for a ceramic preform with increased strength to withstand handling for metal infiltration.

[0003] Vehicles may include drum brakes and/or disc brakes. A drum brake generally comprises a rotating drum-shaped part called a brake drum. Shoes or pads of the drum brake press against the interior surface of the brake drum to cause friction, thereby generating heat, and reduce the rotation of the brake drum. A disc brake generally comprises a rotating brake disc or rotor. Calipers having brake pads that squeeze the exterior and interior of the brake disc to cause friction and reduce the rotation of the brake disc. During the vehicle braking event there is often a high energy

transfer to the frictional surface of the brake drum or brake disc which can lead to a rise in temperature, sometimes as high as 700 degrees C for heavy vehicles such as large trucks or military vehicles.

Summary

[0004] The present application discloses a ceramic preform, a method of making a ceramic preform, a MMC comprising a ceramic preform, and a method of making a MMC.

[0005] In certain embodiments, the method of making a ceramic preform comprises the steps of detangling a mass of reinforcing fibers to separate the reinforcing fibers into individual strands, preparing a ceramic compound, and forming the ceramic compound into a desired shape to create the ceramic preform. The ceramic compound comprises separated strands of reinforcing fibers, ceramic particles, a fugitive porosity generating component, starch, low temperature organic binder, colloidal silica, and water.

[0006] In certain embodiments, the ceramic preform comprises a ceramic compound formed as either a disc or a ring for use in a brake disc metal matrix composite. The ceramic compound comprises between about 41 Wt% and about 47 Wt% ceramic particles; between about 9 Wt% and about 15 Wt% alkaline earth aluminosilicate reinforcing fibers having a diameter greater than 3.5 microns; between about 4.5 Wt% and about 5.5 Wt% fugitive porosity generating component; between about 3 Wt% and about 4 Wt% starch; between about 1.5 Wt% and about 2 Wt% low temperature organic binder; between about 4.5 Wt% and about 11 Wt% colloidal silica; and between about 17 Wt% and about 38 Wt% water.

[0007] In certain embodiments, the metal matrix composite comprises the ceramic preform infiltrated with a molten high temperature performance aluminum alloy to form the brake disc metal matrix composite. The method of making the metal matrix composite comprises the steps of heating the ceramic preform to a preheat temperature, placing the ceramic preform in a mold cavity of a die cast mold, and introducing molten metal into the mold cavity such that the metal infiltrates the ceramic preform to form the brake disc metal matrix composite. The preheat temperature of the ceramic preform is greater than the temperature of the molten metal.

[0008] These and additional embodiments will become apparent in the course of the following detailed description.

Brief Description of the Drawings

[0009] In the accompanying drawings which are incorporated in and constitute a part of the specification, embodiments of the present application are illustrated, which, together with a general description of the invention given above, and the detailed description given below, serve to example the principles of the inventions.

[0010] Figure 1 illustrates a method of making a ceramic preform according to an embodiment of the present application.

[0011] Figure 2 illustrates a method of making a ceramic compound according to an embodiment of the present application.

[0012] Figures 3A and 3B are top and side cross sectional views, respectively, of a ceramic preform blank from which a ceramic preform ring can be cut according to an embodiment of the present application.

[0013] Figures 4A and 4B are top and side cross sectional views, respectively, of

a ceramic preform according to an embodiment of the present application.

[0014] Figure 5A is a top view of a metal matrix composite brake disc according to an embodiment of the present application.

[0015] Figure 5B is a side cross sectional view of the metal matrix composite brake disc of Figure 5A.

[0016] Figure 6A is a top view of a metal matrix composite brake disc according to an embodiment of the present application.

[0017] Figure 6B is a side cross sectional view of the metal matrix composite brake disc of Figure 6A.

Description of Embodiments

[0018] The present application discloses a ceramic preform, a method of making a ceramic preform, a MMC comprising a ceramic preform, and a method of making a MMC. The MMC of the present application is generally less dense, lighter, stronger at higher temperatures and provides a higher wear resistance than certain metal, non-composite materials used for brake drums and brake discs. For example, an aluminum MMC of the present application generally has greater wear resistance and stiffness (i.e., resistance to deformation) than cast iron. As such, the MMC of the present application is useful in applications where high wear resistance, light weight, and strength is beneficial.

[0019] The MMC of the present application may be formed as a brake drum, brake disc or rotor, or any component thereof for a vehicle. For example, the ceramic preform of the present application may be infiltrated with a metal, such as, for example, aluminum alloys and certain special high temperature aluminum alloys, magnesium,

titanium, or copper, and form at least a portion of a brake drum or brake disc. In particular, the MMC of the present application generally forms at least a portion of the braking surface of the brake drum or brake disc. The braking surface for the brake drum is generally located on the interior surface of the brake drum that contacts the shoes or pads of the drum brake. The braking surface for the disc brake is generally located on the surfaces of the brake disc that contact the brake pads. The brake drum and brake disc of the present application may be used for virtually any vehicle, including but not limited to, aircraft, trucks, trains, vans, cars, military vehicles, construction vehicles, motorcycles, hybrid vehicles, SUVs, ATVs, and XUVs. However, the MMC of the present application may be formed as a variety of other items, such as, for example, bearings, pistons, cylinder liners, piston rings, connecting rods, aerospace components, armor, or the like.

[0020] The MMC brake drums and brake discs of the present application generally have greater wear resistance and less weight than a conventional cast iron brake drums or brake discs. One exemplary method of making a MMC brake drum or brake disc is to insert a ceramic preform of the present application into a direct squeeze-casting machine. Molten metal (e.g., aluminum) is then squeezed under high pressure when the mold halves are closed. The molten metal will infiltrate the porous and/or absorbent ceramic preform and fill the die to produce the MMC brake drum, disc or rotor. One difficulty found in conventional MMC brake drum or disc manufacturing processes is developing a preform with enough strength such that it doesn't break during the handling process, the machining process or when the high pressure molten metal is injected filling the preform and the die. The methods and apparatuses for

forming a ceramic preform described in the present application produce a part that is stronger and more dimensionally stable than ceramic preforms made by conventional processes.

[0021] In certain embodiments, the ceramic preform of the present application comprises a ceramic compound that has been kneaded, stamped, rolled and formed into a solid disc or a ring-shaped preform. In certain embodiments, the metal matrix composite of the present application comprises a ceramic preform formed in the shape of a ring. The metal matrix composite may be formed as a brake disc or brake drum and the ceramic preform may form at least a portion of a braking surface of the brake disc.

[0022] In certain embodiments, the method of making a ceramic preform results in a porous ceramic preform with a porosity between about 60% and 65% and improved strength for handling of the preform during metal infiltration. The high temperature aluminum alloy is introduced into the preform by direct squeeze-casting. The preform infiltrated portion of the brake rotor makes up the surface of the brake rotor that is compressed by the brake pads on the calipers to stop the vehicle.

[0023] Figure 1 illustrates an exemplary method 100 of making a ceramic preform according to an embodiment of the present application. As shown, the method 100 comprises the steps of fiber detangling; preparation of the ceramic compound; pressing, rolling, and forming the ceramic compound; drying; heat treatment for binder burnout; and heat treatment for inorganic binder setting or sintering of the ceramic compound. These steps are described in greater detail below.

[0024] The ceramic compound used to make the ceramic preform of the present

application may comprise ceramic particles, reinforcing fiber, a fugitive porosity-generating component, starch, organic low temperature binder, colloidal silica suspension, and water. Table 1 below shows typical weight percentages for the various components of exemplary ceramic compounds of the present application.

Table 1: Exemplary Ceramic Compounds

Component	Weight Percentage, Wt.%	Weight Percentage, Wt.% 7 vol% fiber	Weight Percentage, Wt.% 10 vol% Fiber
Ceramic Particles	41-47	45.7 (42-47)	41.5 (41-42.5)
Reinforcing Fibers	9-15	9.1 (9-11)	11.8 (10-15)
Fugitive porosity-generating component	4.5-5.5	5.2 (4.5-5.5)	4.9 (4.5-5.5)
Starch	3-4	3.5 (3-4)	3.3 (3-4)
Low temperature organic binder	1.5-2	1.7 (1.5-2)	1.7 (1.5-2)
Colloidal silica	4.5-11	4.9 (4.5-11)	9.0 (8-11)
Water	17-38	29.8 (17-36)	27.8 (19-38)

[0025] The ceramic particles of the compound typically provide the ceramic preform with wear resistance and hardness. The ceramic particles may comprise a variety of materials, such as, for example, silicon carbide, alumina, boron carbide, or combinations or mixtures thereof. In certain embodiments, the ceramic particles comprise 360 grit silicon carbide particles.

[0026] The reinforcing fiber or whisker of the ceramic compound may include short or long fibers and may comprise a variety of materials, such as, for example,

carbon, silicon carbide, metallic, or ceramic fibers, whiskers, or chopped filaments. The carbon fiber may be in the form of chopped carbon tape or carbon nanotubes. In certain embodiments, the reinforcing fiber of the ceramic compound is an alkaline earth aluminosilicate fiber, such as, for example, those fibers labeled Fiber D and E in Table 2 below.

[0027] The reinforcing fiber of the ceramic compound may be prepared prior to introducing the fiber into the batch of ceramic compound components (see, e.g., Fiber Detangling step in Figure 1). For example, when an alkaline earth aluminosilicate refractory fiber is used as the reinforcing fiber, the fiber may be prepared prior to introducing the fiber into the batch. The alkaline earth aluminosilicate refractory fiber is spun from melted high purity mineral ores (e.g., melted in an electric arc furnace). The fiber is often received from the manufacturer interlocked together in clumps (e.g., a clumped matted ball of refractory fiber) and including shot glass beads left over from the melt spinning process. At least some pretreatment of the fiber may be needed before introducing the fiber in to the batch such that the fibers are separated into individual strands and the shot glass beads are removed. The alkaline earth aluminosilicate fibers used in the compound of the present application may also be of a compositional family that is high in calcia (CaO) and magnesia (MgO) and may not contain shot when received from the manufacturer. The diameters of alkaline earth glass fibers are generally larger than 3.5 microns which is approximately the diameter of typical aluminosilicate glass fibers (see, e.g., Fiber A of Table 2 below). Alkaline earth aluminosilicate glass fibers are also commercially available at cost effective prices making the total cost of the methods described herein commercially attractive.

[0028] The composition and fiber diameter of several exemplary reinforcing fibers is shown in Table 2 below. The formulations in columns labeled Fiber D and Fiber E of Table 2 are alkaline earth aluminosilicate fibers as discussed above.

Table 2: Exemplary Ceramic Fiber Chemical Compositions, wt%

Name	Fiber A	Fiber B	Fiber C	Fiber D	Fiber E
Compositional family	alumina-silica fiber	alumina-silica-zirconia fiber	alumina-silica-chromia fiber	alkaline earth aluminosilicate fiber	alkaline earth aluminosilicate fiber
SiO ₂	56	50	54.3	38-43	40-50
Al ₂ O ₃	44	34.9	42.6	18-23	10-16
CaO + MgO	0.5	0.09	0.1	23-28	20-35
ZrO ₂		15.2			
Cr ₂ O ₃			2.8		
Fe ₂ O ₃ + TiO ₂	0.15	0.15	0.15		
FeO				4.5-8.0	
K ₂ O + Na ₂ O	0.1	0.1	0.1	4.5	
Others				6.0	6-10
Fiber diameter, μm	3.5	3.5	3.5	5.5	5-6

[0029] In order for the ceramic preform to be infiltrated with molten metal, it is advantageous to have highly interconnected porosity which creates a continuous pathway for the flow of molten metal. The fugitive porosity-generating component of the ceramic compound permits the ceramic preform to have a highly interconnected porosity. For example, in certain embodiments, the porosity-generating component is a carbonaceous material that will burnout of the molded ceramic article during heat treatment (see, e.g., Binder Burnout and Sintering steps in Figure 1). Exemplary carbonaceous materials include, but are not limited to, graphite, carbon black, graphene or organic materials with a high carbon content such as, for example, walnut shell flour, pecan shell flour, rice hulls, and saw dust.

[0030] In certain embodiments, the low temperature organic binder of the ceramic preform compound is methyl cellulose. When methyl cellulose is used, it is generally heat activated. Further, other binders that may be used including, but not limited to, guar gum and Xanthum gum. After molding of the ceramic compound and exposure to a temperature between, for example, about 49 and 60 degrees C (or between about 120 and 140 degrees F), the ceramic preform develops green strength which maintains its shape during handling.

[0031] The colloidal silica solution of the ceramic compound is generally flocced with a starch, such as, for example, a cationic corn starch, to provide a high temperature bonding system in the ceramic preform. For example, in certain embodiments, the colloidal silica solution includes colloidal silica particles having a negative surface charge. A cationic starch is positively charged such that there is electrostatic attraction for flocculation to occur. This flocced bonding system is used to bond the refractory fibers and particles of the ceramic compound and contributes to the green strength of the ceramic preform. The effects of the flocculation are noticeable with a highly loaded ceramic body and a minimum amount of water present (e.g., between about 15 and 30%). As discussed in greater detail below, adding a first water amount followed by adding a second water amount with the colloidal silica solution may contribute to the effectiveness of the flocced bonding system.

[0032] Figure 1 illustrates a method 100 of preparing a preform compound according to an embodiment of the present application. As shown, the method 100 begins with a pretreatment step of the fiber that results in detangling of the fiber. For example, a liquid dispersion of bulk fibrous ceramic materials may be separated and de-

tangled into individual fibers for introduction into the ceramic compound.

[0033] In certain embodiments, alkaline earth aluminosilicate fibers are received from the manufacturer in a tangled and agglomerated mass. One exemplary method of separating the tangled alkaline earth aluminosilicate fibers into the individual fibers involves first placing a given quantity into a vessel and adding an aqueous solution comprised of distilled water and an alkylolamino amide additive at a 1.0 to 2.5 vol% concentration. The fiber material and solution within the vessel are held in a temperature controlled environment above 70 degrees F. The contents of the vessel are then gently stirred over a minimum period, for example 18 hours. The additive solution is then decanted or poured off from the vessel leaving the treated fiber material on the bottom and sides of the vessel. The fiber is then washed with water to remove any residual additive solution. The vessel may be backfilled with water such that the settled out ceramic fibers become re-dispersed in the water and not caked on the bottom of the vessel. The washing procedure is performed generally at least five times giving adequate time, typically one hour, for the ceramic fibers to settle out so that the wash water can be decanted without the loss of suspended fibers. Water is added to wet the fiber in an amount needed for a first water addition to the batch (e.g., between about 14 wt% and about 27 wt% of the batch composition) so that it can be added to the other batch constituents as an aqueous washed dispersed fiber suspension.

[0034] Figure 2 illustrates the steps in the method for preparing the ceramic compound. As illustrated, dry powders, such as, for example, the silicon carbide particles, burnout material, starch and organic binder, are added and mixed. For example, in certain embodiments, the silicon carbide particles are dry mixed with the

walnut shell flour, starch, and methyl cellulose in a tumble mixer or other suitable mixer for a period of time (e.g., approximately 30 minutes). A US Stoneware Roller Mill, Model 755, with an enclosed cylindrical container may be used for this dry tumbling step.

[0035] As illustrated in Figure 2, the addition of water to the mixture is generally divided into two parts, the first water addition and a second water addition. As discussed above, the first water addition (e.g., between about 14 wt% and about 27 wt% of the batch composition) which is used to form the detangled fiber suspension that is placed in a mixer (e.g., Hobart Mixer) along with the tumbled dry powders discussed above. The mixture is mixed for a period of time (e.g., approximately 20 minutes) with a paddle mixer blade. This amount of wet mixing time generally insures that the starch is completely wetted out in the batch. During the second water addition, a certain amount of water (e.g., between about 3.5 wt% and about 9 wt% of the batch composition) is mixed with the colloidal silica and is added into the mixer and mixed for a period of time (e.g., approximately 1-2 minutes). As such, the batch clumps together. The blade on the mixer is changed from a paddle blade to a dough hook. The ceramic compound is mixed for approximately 1 minute with the dough hook kneading the batch. It is also to be appreciated that a commercially available equipment such as a kneader/extruder available from Charles Ross & Sons, Inc. can be used to work the batch and to extrude the ceramic compound in a continuous ribbon of desired thickness that is then transported on a conveyor belt for a continuous forming operation.

[0036] The ceramic compound may be molded to form a brake drum or brake disc preform using a process which generally includes pressing, rolling, and forming the

compound as illustrated in Figure 1. In certain embodiments, a commercial available kneader/extruder can be used to work the batch and to extrude a cylindrical part that is about 6 inches in diameter. The 6-inch diameter cylindrical part is extruded onto a conveyor belt which moves the extruded part to the pressing step. Pressing increases the diameter and reduces thickness of the ceramic compound, for example from a diameter of approximately 6 inches to a diameter of approximately 10 inches and a height of about one inch.

[0037] As illustrated in Figure 1, the next step in the method for forming a brake drum or brake disc preform is to roll the ceramic compound. The rolling step removes air pockets from the ceramic compound and contributes to the uniformity of the material. The rolling process further increases the diameter and reduces the height of the ceramic compound, for example to a diameter of approximately 14 inches and a height of about 0.5 inches.

[0038] As illustrated in Figure 1, the ceramic compound is formed (e.g., by stamping or cutting) into a shape for use in a MMC brake drum or brake disc. In certain embodiments, a stamp or die is used to form the ceramic compound into a cylindrical, disc, or ring shape. For example, after the ceramic compound is pressed and rolled, the ceramic compound may be formed as a disc for use as brake disc or rotor preform. Figures 3A and 3B illustrate a disc shaped ceramic compound 300 according to an embodiment of the present application. The disc shaped ceramic preform 300 was made by forming a disc shape of the required dimensions from a ceramic compound. In certain embodiments, the outer diameter (OD) of the disc shaped preform 300 is between about 9 inches and about 16 inches and the height (H) is between about 1/8

inch and about 2 inches after the compound has been pressed, rolled and stamped.

[0039] The ceramic compound may also be formed as a ring for use as a brake disc or rotor preform. For example, a stamp or die may be used to stamp or cut a ring-shaped preform from the ceramic compound. Figures 4A and 4B illustrate a ring shaped ceramic compound 400 according to an embodiment of the present application. The ring shaped ceramic preform 400 was made by forming a ring shape of the required dimensions from a ceramic compound. In certain embodiments, the outer diameter (OD) of the ring shaped preform 300 is between about 9 inches and about 16 inches; the inner diameter (ID) is between about 5 inches and about 12.5 inches; and the height (H) is between about 1/8 inch and about 2 inches.

[0040] The dimensions of the stamped preforms are generally greater than the desired dimensions by a certain amount (e.g., 2%-4% greater) to make possible the final dimensions needed for the brake drum or brake disc or rotor preform, which is to be infiltrated with molten metal to form the MMC. The over-sized dimensions of the stamped preforms allow for shrinkage of the ceramic compound during further processing steps and machining of the preform to the desired dimensions for metal infiltration of the preform.

[0041] As illustrated in Figure 1, after the ceramic preform is formed from the ceramic compound, the ceramic preform goes through the further processing steps of drying, binder burnout, and heat treatment. The ceramic preform is generally dried in the drying oven at a certain temperature (e.g., about 60 degrees C or about 140 degrees F) for a period of time. The length of the oven drying time will often vary based on the water content of the ceramic preform and the size of the part. A ceramic preform

is generally considered dry when the weight loss is between about 20 wt% and about 35 wt% due to the removal of water from the part.

[0042] During the binder burnout step illustrated in Figure 1, a low temperature heat treatment is conducted to remove the organics or volatile components from the ceramic preform. In certain embodiments, these organics include the walnut shell flour, the starch, and the methyl cellulose. The low temperature heat treatment cycle is generally an approximately 1 hour ramp to a certain temperature (e.g., about 260 degrees C or about 500 degrees F) with an approximately two hour hold at about the same temperature.

[0043] Sintering is a high temperature heat treatment conducted to seal the ceramic bond created by the colloidal silica. The high temperature heat treatment generally has a hold at a certain temperature (e.g., about 985 degrees C or about 1800 degrees F) for approximately two hours. In certain embodiments, after the high temperature heat treatment, the colloidal silica particles remain in the ceramic preform and the ceramic preform comprises silicon carbide particles, refractory fiber and the silicate bond between the silicon carbide particles and the refractory fibers. In these embodiments, the burnout materials, starch, and methyl cellulose are all removed from the porous ceramic preform body after the low and high temperature heat treatments. After the sintering step, the ceramic preform can be cut to shape and/or machined to achieve final dimensional requirements for accepting a ceramic preform into a brake drum or disc brake rotor high pressure die-casting mold.

[0044] In one exemplary embodiment, a disc shaped ceramic preform was made by stamping a shape of the required dimensions from the ceramic compound. The

ceramic compound formulation was made by combining 43.2 wt.% Silicon Carbide 360 grit particles (Washington Mills Carborex 360), 8.6 wt.% alkaline earth aluminosilicate refractory fiber, 7 vol% fiber (Fibrox Technology LP Fibrox 300D), 4.9% walnut shell flour – 100 mesh (Echo-Shell, Inc.), 3.3 wt% flaked cationic corn starch (Wesbond Westar +3), 1.6 wt% hydroxypropyl methyl cellulose (Dow Chemical Methocel™ A4M), 24.1 wt% of first water addition, 4.1 wt% of second water addition and 10.1 wt% colloidal silica solutions (Wesbond Bindzil® 1440). The concentration of fiber in this ceramic compound is 7 vol%. The fiber detangling of the alkaline earth aluminosilicate fiber was accomplished using an aqueous mixture of 2.105 wt% of an alkylolamino amide (Altana Disperbyk-109) in distilled water.

[0045] In another exemplary embodiment, a disc shaped ceramic preform was made by stamping a shape of the required dimensions from the ceramic compound. The ceramic was made by combining 41.5 wt.% Silicon Carbide 360 grit particles (Washington Mills Carborex 360), 11.8 wt.% alkaline earth aluminosilicate refractory fiber, 10 vol% fiber (Lapinus Rockseal® RS480-Roxul®1000), 4.9% walnut shell flour – 100 mesh (Echo-Shell, Inc.) 3.3 wt% flaked cationic corn starch (Wesbond Westar +3), 1.7 wt% hydroxypropyl methyl cellulose (Dow Chemical Methocel™ A4M), 23.8 wt% of first water addition, 4.0 wt% of second water addition and 9.0 wt% colloidal silica solutions (Wesbond Bindzil® 1440). The ceramic compound was kneaded, pressed, rolled and stamped for form a preform that was approximately 14 inches in diameter and approximately ½ inch thick. The ceramic part was dried, fired for binder burnout, and sintered. The resulting finished part had a weight of approximately 1256 gm and it measured 14.3 inches in diameter and 0.564 inch thick. The composition of the final

part was 72.9 wt% silicon carbide, 20.7 wt% alkaline earth aluminosilicate fiber and 6.4 wt% silicate binder.

[0046] The ceramic preform of the present application may be infiltrated with a metal, such as, for example, aluminum, magnesium, titanium, or copper, to form a MMC of the present application. For example, the ceramic preform may be introduced into a die-casting mold for infiltration of metals that are capable of being die cast, such as, for example, aluminum, magnesium, or copper, to form a MMC of the present application. Certain high temperature performance aluminum alloys may be used for infiltration. High temperature performance aluminum alloys have improved elevated temperature tensile and creep properties when compared to 356 or A356 die-casting alloys. These high temperature performance aluminum alloys are often used for common rail diesel cylinder heads. The compositional family is the aluminum/silicon/copper alloys (AlSi7Cu) alloys with varying amounts of magnesium, manganese, vanadium, zirconium, titanium and iron (Mg, Mn, V, Zr, Ti and Fe). In certain embodiments, the high temperature performance aluminum alloy comprises AlSi7Cu3.5Mg0.15Mn0.15V0.12Zr0.12Ti0.12 with Fe <0.15%.

[0047] A die cast mold generally includes two die parts: a first die part, the lower part or cope, that is generally stationary and coupled to a non-moving platen of the die casting machine and a second die part, the upper part or drag, that is movable relative to the first die part and is generally coupled to a movable platen of the die casting machine. Within a die part is a mold cavity that receives the injected molten metal. The mold cavity is representative of the final product shape with calculated shrink factored in and draft added to aid in part release. In certain embodiments, the amount of shrinkage

is between about .07 % and about 2.19%. The cavity also generally includes a nesting area that accepts and locates the ceramic preform within the mold cavity.

[0048] For introduction of the molten metal in a direct squeeze-casting process, the molten metal is introduced from above the preform while the two die parts are open. The pre-heated ceramic preform is placed within the mold cavity of the drag portion of the mold. The molten metal is introduced on top of the preform. The two die parts, cope and drag, are closed applying pressure to the part being infiltrated. Molten metal infiltrates the preform during the squeezing process. The preform is generally at a temperature that is higher than the molten metal during the squeeze casting process. If the molten metal hits a preform of a lower temperature than the metal, the metal freezes in contact with the surface of the preform and does not infiltrate.

[0049] The die parts are typically machined from tool steel with various treatments to improve durability. Heating and cooling circuits may also be added throughout the die parts to aid in attaining and retaining optimum temperatures for the casting process. These circuits may use various fluids to transfer temperatures into or out of predetermined areas of the die and are typically placed close to the mold cavity but do not enter the cavity.

[0050] The ceramic preform is placed within the mold cavity of the die cast mold. The ceramic preform may be preheated to a certain temperature prior to introduction into the mold cavity. For example, the ceramic preform may be preheated to a temperature that is above the temperature of the molten metal that is being injected into the mold cavity (e.g., aluminum). In certain embodiments, the ceramic preform is heated to at least 50 degrees F above the temperature of the molten metal that is being

injected into the mold cavity. In other embodiments, the ceramic preform is heated to at least 100 degrees F above the temperature of the molten metal that is being injected into the mold cavity.

[0051] Molten metal is injected into the mold cavity at a low velocity and infiltrates the porous body of the ceramic preform. The velocity of the molten metal is such that the ceramic preform does not deform during injection of the molten metal. The molten metal infiltrates substantially through the entire wall thickness. Further, in certain embodiments, the molten metal infiltrates substantially through a ceramic preform having a wall thickness of 2 inches or less.

[0052] In one example, a 4X4 inch preform was infiltrated with molten metal to form an Al-MMC. The 4X4 inch preform made by the methods described herein and containing 7 vol% Fiber E from Table 2 (Fibrox Fiber) and silicon carbide particles was infiltrated with a high temperature performance aluminum die-casting alloy. The preform temperature was about 1500 °F and the metal temperature was about 1420 °F. The composition of the alloy was 6.83% silicon, 0.125% iron, 3.46% copper, 0.0082% manganese, 0.344% magnesium, 0.129% titanium, 0.114% zirconium, 0.114% vanadium and a balance of aluminum. The pressure exerted during the squeeze casting on the sample was 12,570 pounds per square inch with a dwell time of 9 seconds. The preform sample was completely infiltrated with metal. The sharp corners of the preform edges of the infiltrated sample were maintained indicating that there was no deformation of the preform during the squeeze casting. Microstructure development of the high temperature alloy was developed with a solution heat treatment at 941°F (505°C) for five hours followed by a water quench. Subsequently, the preform sat at

room temperature in air for 24 hours before an aging heat treatment at 392°F (200°C) for five hours.

[0053] Figures 5A and 5B illustrate a MMC brake disc 500 according to an embodiment of the present application. As shown, the MMC brake disc 500 is formed using a ring shaped ceramic preform 502 having approximately the same shape as the MMC brake disc. Each ceramic preform was infiltrated with a metal (e.g., aluminum) to form the MMC brake disc 500, such as during a direct squeeze die casting process as discussed herein. The ceramic preform 502 and MMC brake disc 500 may be a variety of sizes and shapes for a variety of different vehicles. For example, in certain embodiments, the MMC brake disc 500 is between about 9 inches and about 16 inches in diameter and between about 1/8 and about 2 inches thick.

[0054] Figures 6A and 6B illustrate a MMC brake disc 600 according to an embodiment of the present application. As shown, the MMC brake disc comprises a vented and finned brake disc design having a raised center portion or hat 604 and a channel 606 that facilitates cooling of the disc brakes. Further, as illustrated in Figure 6B, the MMC brake disc 600 has two reinforced ceramic surfaces formed using two ring shaped ceramic preforms 602 having approximately the same shape as the MMC brake disc. These ceramic surfaces are engaged by the brake calipers for stopping the vehicle. Each ceramic preform 602 was infiltrated with a metal (e.g., aluminum) to form the MMC brake disc 600, such as during a die casting process as discussed herein. The ceramic preforms 602 and MMC brake disc 600 may be a variety of sizes and shapes for a variety of different vehicles. For example, in certain embodiments, the MMC brake disc 600 is between about 9 inches and about 16 inches in diameter and

between about 1 inch and about 2.0 inches thick.

[0055] The strength of the ceramic preform facilitates the handling that is required to prepare the ceramic preform for placement in the die-casting mold. The ceramic preform disclosed herein have improved strength over similar type preforms made using a different forming method.

[0056] For example, five ceramic preforms of different composition were produced and the bulk density, porosity and compressive strength of each preform was measured. Table 3 below shows the bulk density and porosity as measured by ASTM C830 and the compressive strength as measured by ASTM C133 for the five ceramic preforms (Preforms 1-5). Four of the five ceramic preforms (Preforms 2-5) were made using the same method described herein and illustrated in Figures 1 and 2.

[0057] For Preform 2, the ceramic preform formulation was made by combining 43.2 wt.% Silicon Carbide 360 grit particles; 8.6 wt.% Fiber D, an alkaline earth aluminosilicate refractory fiber with a fiber diameter of approximately 5.5 micron; 4.9% walnut shell flour – 100 mesh; 3.3 wt% flaked cationic corn starch 1.6 wt% hydroxypropyl methyl cellulose; 24.1 wt% of first water addition; 4.1 wt% of second water addition; and 10.1 wt% colloidal silica solutions. The concentration of fiber in this ceramic compound is 7 vol%. The fiber detangling of the alkaline earth aluminosilicate fiber was accomplished using an aqueous mixture of 2.105 wt% of an alkylolamino amide in distilled water.

[0058] For Preform 3, the ceramic preform formulation was made by combining 43.2 wt.% Silicon Carbide 360 grit particles; 8.6 wt.% Fiber E, an alkaline earth aluminosilicate refractory fiber with a fiber diameter of 5-6 microns by population; 4.9%

walnut shell flour – 100 mesh; 3.3 wt% flaked cationic corn starch; 1.6 wt% hydroxypropyl methyl cellulose; 24.1 wt% of first water addition; 4.1 wt% of second water addition; and 10.1 wt% colloidal silica solutions. The concentration of fiber in this ceramic compound is 7 vol%. The fiber detangling of the alkaline earth aluminosilicate fiber was accomplished using an aqueous mixture of 2.0 wt% of an alkylolamino amide in distilled water.

[0059] For Preform 4, the ceramic preform formulation was made by combining 41.5 wt.% Silicon Carbide 360 grit particles; 11.8 wt.% Fiber D, an alkaline earth aluminosilicate refractory fiber with a fiber diameter of approximately 5.5 micron; 4.9% walnut shell flour – 100 mesh; 3.3 wt% flaked cationic corn starch; 1.7 wt% hydroxypropyl methyl cellulose; 23.8 wt% of first water addition; 4.0 wt% of second water addition; and 9.0 wt% colloidal silica solution. The concentration of fiber in this ceramic compound is 10 vol%. The fiber detangling of the alkaline earth aluminosilicate fiber was accomplished using an aqueous mixture of 2.0 wt% of an alkylolamino amide in distilled water.

[0060] For Preform 5, the ceramic preform formulation was made by combining 41.5 wt.% Silicon Carbide 360 grit particles; 11.8 wt.% Fiber E, an alkaline earth aluminosilicate refractory fiber with a fiber diameter of 5-6 microns by population; 4.9% walnut shell flour – 100 mesh; 3.3 wt% flaked cationic corn starch; 1.7 wt% hydroxypropyl methyl cellulose; 23.8 wt% of first water addition; 4.0 wt% of second water addition; and 9.0 wt% colloidal silica solution. The concentration of fiber in this ceramic compound is 10 vol%. The fiber detangling of the alkaline earth aluminosilicate fiber was accomplished using an aqueous mixture of 2.0 wt% of an alkylolamino amide

in distilled water.

[0061] Preform 1 was a ceramic preform made by combining 47.63 wt% Silicon Carbide 360 grit particles; 9.53 wt% hammermilled and sieved Fiber A, an aluminosilicate refractory fiber with a fiber diameter of 3.5 micron; 5.4 wt% walnut shell flour -100 mesh; 3.63 wt% flaked cationic corn starch; 1.77 wt% hydroxypropyl methyl cellulose; 11.3 wt.% of first water addition; 9.64 wt% of second water addition; and 11.1 wt% colloidal silica solution. The process for preparing the fiber for introduction into the batch included the steps of hammermilling and sieving the fiber received from the manufacturer. After hammermilling, the fiber included less shot and consisted of clumps that were less than about 6 mm in length. The fiber clumps were then pressed to further break down the clumps. The pressed fiber was then screened (e.g., through a 500 micron wire mesh screen) to remove loose shot (e.g., shot having a diameter greater than 500 microns). The fiber left on top of the screen was then added to the batch of ceramic compound components. The fiber added to the batch after this pre-treatment was in the form of small clumps of fiber that were not fully detangled.

[0062] Table 3 below shows the bulk density and porosity as measured by ASTM C830 and the compressive strength as measured by ASTM C133 for the five ceramic preforms 1-5. The compressive strength is measured parallel and perpendicular to the pressing direction of the ceramic preform. As shown, the formulations made by the methods disclosed herein and using Fiber D and Fiber E (i.e., Preforms 2-5) have increased strength by approximately as much as a factor of four over the formulation using Fiber A.

Table 3: Bulk Density, Porosity and Strength of Various Ceramic Preforms

Preform	1	2	3	4	5
Fiber	7 vol% Fiber A	7 vol% Fiber D	7 vol% Fiber E	10 vol% Fiber D	10 vol% Fiber E
Bulk density, g/cc	1.05	1.02	1.00	0.96	1.01
Porosity, %	65	66	67	68	67
Strength, psi (parallel/perpendicular to pressing direction)	40/40	145/214	190/200	150/250	97/210

[0063] It is believed that Preforms 2-5 are stronger than Preform 1 due to the well dispersed fibers within the ceramic formulation of Preforms 2-5 having larger fiber diameters than Preform 1 (5-6 microns vs. 3.5 microns). The individually separated larger diameter fibers in Preforms 2-5 is able to create an interlocking structure between the fiber and silicon carbide particles that is very strong. This interlocking strong network is held together by a silicate bond. The pre-treatment of Fiber A is laborious, costly and wasteful as half of the received fiber is used as a raw material in the ceramic formulation. Fiber A includes 50 wt% useable fiber and 50 wt% shot. Shot is unacceptable in a ceramic preform used for a brake disc or rotor, or a brake drum, because when a shot particle is on the braking surface the braking event may be compromised. The aqueous detangling process for the fiber using an alkykolamino amide additive incorporated into the processing of Preforms 2-5 is simple, lends itself to a scaleable manufacturing process, and is cost effective.

[0064] As described herein, when one or more components are described as being connected, joined, affixed, coupled, attached, or otherwise interconnected, such interconnection may be direct as between the components or may be in direct such as through the use of one or more intermediary components. Also as described herein, reference to a "member," "component," or "portion" shall not be limited to a single

structural member, component, or element but can include an assembly of components, members or elements.

[0065] While the present invention has been illustrated by the description of embodiments thereof, and while the embodiments have been described in considerable detail, it is not the intention of the applicants to restrict or in any way limit the scope of the invention to such details. Additional advantages and modifications will readily appear to those skilled in the art. For example, where components are releasably or removably connected or attached together, any type of releasable connection may be suitable including for example, locking connections, fastened connections, tongue and groove connections, etc. Still further, component geometries, shapes, and dimensions can be modified without changing the overall role or function of the components. Therefore, the inventive concept, in its broader aspects, is not limited to the specific details, the representative apparatus, and illustrative examples shown and described. Accordingly, departures may be made from such details without departing from the spirit or scope of the applicant's general inventive concept.

[0066] While various inventive aspects, concepts and features of the inventions may be described and illustrated herein as embodied in combination in the exemplary embodiments, these various aspects, concepts and features may be used in many alternative embodiments, either individually or in various combinations and sub-combinations thereof. Unless expressly excluded herein all such combinations and sub-combinations are intended to be within the scope of the present inventions. Still further, while various alternative embodiments as to the various aspects, concepts and features of the inventions--such as alternative materials, structures, configurations,

methods, devices and components, alternatives as to form, fit and function, and so on-- may be described herein, such descriptions are not intended to be a complete or exhaustive list of available alternative embodiments, whether presently known or later developed. Those skilled in the art may readily adopt one or more of the inventive aspects, concepts or features into additional embodiments and uses within the scope of the present inventions even if such embodiments are not expressly disclosed herein. Additionally, even though some features, concepts or aspects of the inventions may be described herein as being a preferred arrangement or method, such description is not intended to suggest that such feature is required or necessary unless expressly so stated. Still further, exemplary or representative values and ranges may be included to assist in understanding the present disclosure, however, such values and ranges are not to be construed in a limiting sense and are intended to be critical values or ranges only if so expressly stated. Moreover, while various aspects, features and concepts may be expressly identified herein as being inventive or forming part of an invention, such identification is not intended to be exclusive, but rather there may be inventive aspects, concepts and features that are fully described herein without being expressly identified as such or as part of a specific invention, the inventions instead being set forth in the appended claims. Descriptions of exemplary methods or processes are not limited to inclusion of all steps as being required in all cases, nor is the order that the steps are presented to be construed as required or necessary unless expressly so stated. The words used in the claims have their full ordinary meanings and are not limited in any way by the description of the embodiments in the specification.

[0067]

We claim:

1. A method of making a ceramic preform, comprising the steps of:
 - detangling a mass of reinforcing fibers to separate the reinforcing fibers into individual strands;
 - preparing a ceramic compound comprising the separated strands of reinforcing fibers, ceramic particles, a fugitive porosity generating component, starch, low temperature organic binder, colloidal silica, and water; and
 - forming the ceramic compound into a desired shape to create the ceramic preform.
2. The method of claim 1, wherein the ceramic compound comprises: between about 41 Wt% and about 47 Wt% ceramic particles; between about 9 Wt% and about 15 Wt% reinforcing fibers; between about 4.5 Wt% and about 5.5 Wt% fugitive porosity generating component; between about 3 Wt% and about 4 Wt% starch; between about 1.5 Wt% and about 2 Wt% low temperature organic binder; between about 4.5 Wt% and about 11 Wt% colloidal silica; and between about 17 Wt% and about 38 Wt% water.
3. The method of claim 2, wherein the reinforcing fibers are alkaline earth aluminosilicate reinforcing fibers and the ceramic particles are silicon carbide particles.
4. The method of claim 1, wherein the reinforcing fibers are alkaline earth aluminosilicate reinforcing fibers comprising between about 38 Wt% and about 43 Wt% SiO₂, between about 18 Wt% and about 23 Wt% Al₂O₃, and between about 23 Wt% and about 28 Wt% CaO + MgO.
5. The method of claim 1, wherein the reinforcing fibers are alkaline earth aluminosilicate reinforcing fibers comprising between about 40 Wt% and about 50 Wt% SiO₂, between

about 10 Wt% and about 16 Wt% Al_2O_3 , and between about 20 Wt% and about 35 Wt% $\text{CaO} + \text{MgO}$.

6. The method of claim 1, wherein the detangling step comprises:

placing the mass of reinforcing fibers into a vessel and adding an aqueous solution comprised of distilled water and an alkylolamino amide additive at a 1.0 to 2.0 wt% concentration;

stirring the contents of the vessel;

decanting the aqueous solution;

washing the reinforcing fibers with water to remove residual additive;

decanting the wash water; and

adding between about 14 wt% and about 27 wt% of a first water to the washed reinforcing fibers to form a detangled fiber suspension.

7. The method of claim 6, wherein preparing the ceramic compound comprises:

mixing the dry powders of the ceramic compound, the dry powders comprising the ceramic particles, fugitive porosity generating component, starch and low temperature organic binder;

mixing the mixed dry powders and the detangled fiber suspension together to form a first mixture;

mixing the colloidal silica and between about 3.5 Wt% and about 9 Wt% of a second water together to form a second mixture; and

mixing the first and second mixtures together to form the ceramic compound.

8. The method of claim 1 further comprising pressing and rolling the ceramic compound.

9. The method of claim 1, wherein the ceramic compound is formed as either a disc or a ring for use in a brake disc metal matrix composite.

10. The method of claim 1 further comprising:

drying the ceramic preform in an oven until the weight loss percentage due to the removal of the water from the ceramic preform is between about 20 percent and about 70 percent;

heat treating the ceramic preform at a temperature sufficient to remove the fugitive porosity generating component, the starch, and the low temperature organic binder from the ceramic preform; and

heat treating the ceramic preform at a temperature sufficient to seal the ceramic bond created by the colloidal silica.

11. The method of claim 1, wherein the reinforcing fibers have a diameter greater than 3.5 microns.

12. The method of claim 11, wherein the diameter of the reinforcing fibers is between 5 and 6 microns.

13. A ceramic preform comprising a ceramic compound formed as either a disc or a ring for use in a brake disc metal matrix composite, the ceramic compound comprising:

between about 41 Wt% and about 47 Wt% ceramic particles; between about 9 Wt% and about 15 Wt% alkaline earth aluminosilicate reinforcing fibers having a diameter greater than 3.5 microns; between about 4.5 Wt% and about 5.5 Wt% fugitive porosity generating component; between about 3 Wt% and about 4 Wt% starch; between about 1.5 Wt% and about 2 Wt% low temperature organic binder; between about 4.5 Wt% and about 11 Wt% colloidal silica; and between about 17 Wt% and about 38 Wt% water.

14. The ceramic preform of claim 13, wherein the ceramic particles are silicon carbide particles.
15. The ceramic preform of claim 13, wherein the alkaline earth aluminosilicate reinforcing fibers comprise between about 38 Wt% and about 43 Wt% SiO₂, between about 18 Wt% and about 23 Wt% Al₂O₃, and between about 23 Wt% and about 28 Wt% CaO + MgO.
16. The ceramic preform of claim 13, wherein the alkaline earth aluminosilicate reinforcing fibers comprise between about 40 Wt% and about 50 Wt% SiO₂, between about 10 Wt% and about 16 Wt% Al₂O₃, and between about 20 Wt% and about 35 Wt% CaO + MgO.
17. The ceramic preform of claim 13, wherein the diameter of the alkaline earth aluminosilicate reinforcing fibers is between 5 and 6 microns.
18. The ceramic preform of claim 13, wherein the ceramic preform comprises at least a portion of a braking surface of the brake disc metal matrix composite.
19. A method of making a metal matrix composite, comprising the steps of:
 - heating the ceramic preform of claim 13 to a preheat temperature;
 - placing the ceramic preform in a mold cavity of a die cast mold; and
 - introducing molten metal into the mold cavity such that the metal infiltrates the ceramic preform to form the brake disc metal matrix composite, wherein the preheat temperature is greater than the temperature of the molten metal.
20. The method of claim 19, wherein the molten metal is introduced into the mold cavity using a direct squeeze-casting process.

21. The method of claim 19, wherein the molten metal is a high temperature performance aluminum alloy.
22. The method of claim 19, wherein the ceramic preform comprises at least a portion of a braking surface of the brake disc metal matrix composite.
23. A metal matrix composite, comprising: the ceramic preform of claim 13 infiltrated with a molten high temperature performance aluminum alloy to form the brake disc metal matrix composite.
24. The metal matrix composite of claim 23, wherein the ceramic preform comprises at least a portion of a braking surface of the brake disc metal matrix composite.

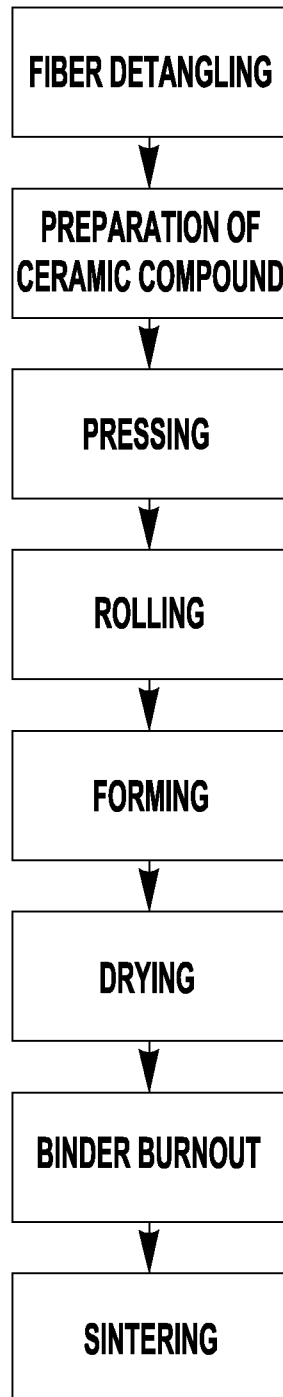


FIG. 1

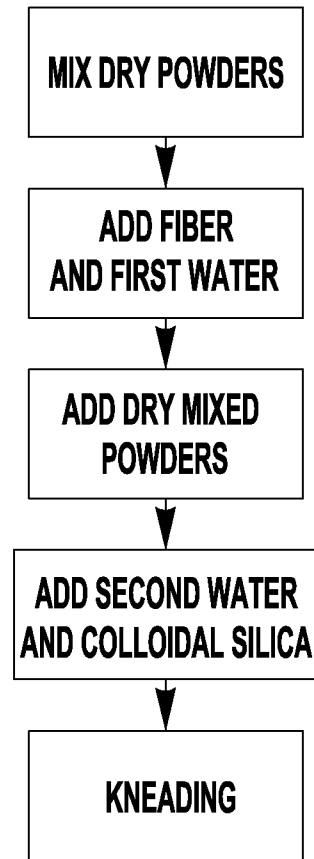


FIG. 2

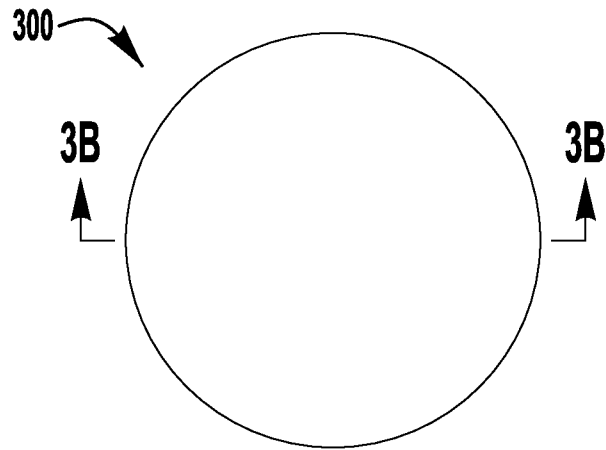


FIG. 3A

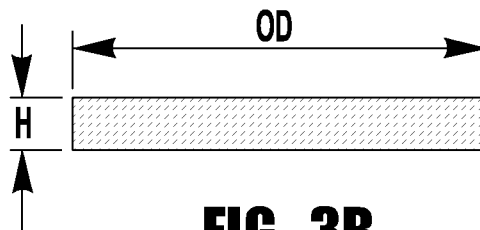


FIG. 3B

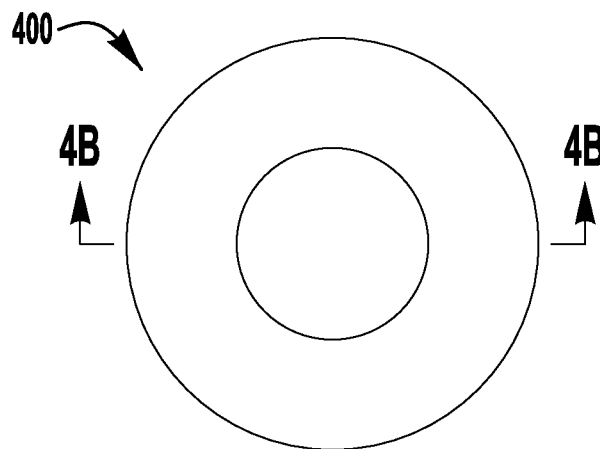


FIG. 4A

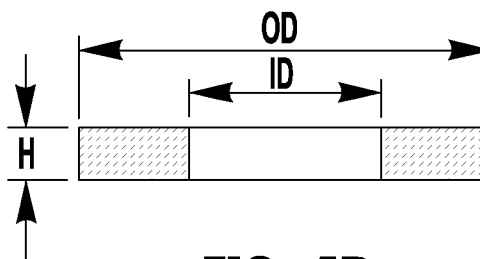


FIG. 4B

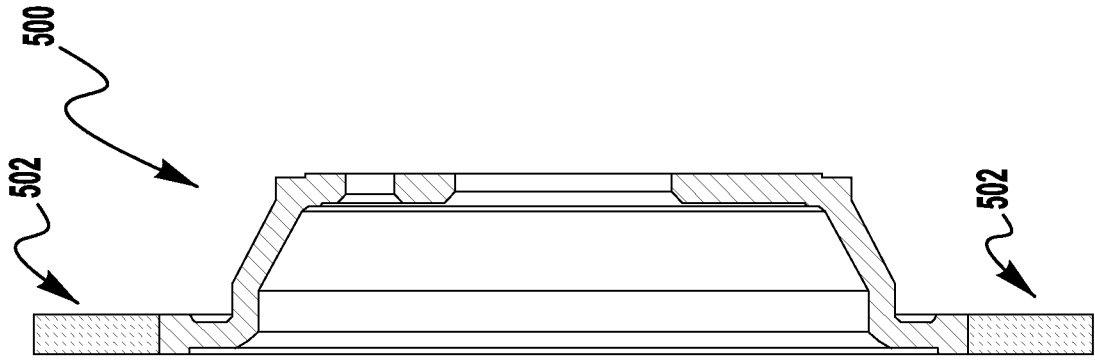


FIG. 5B

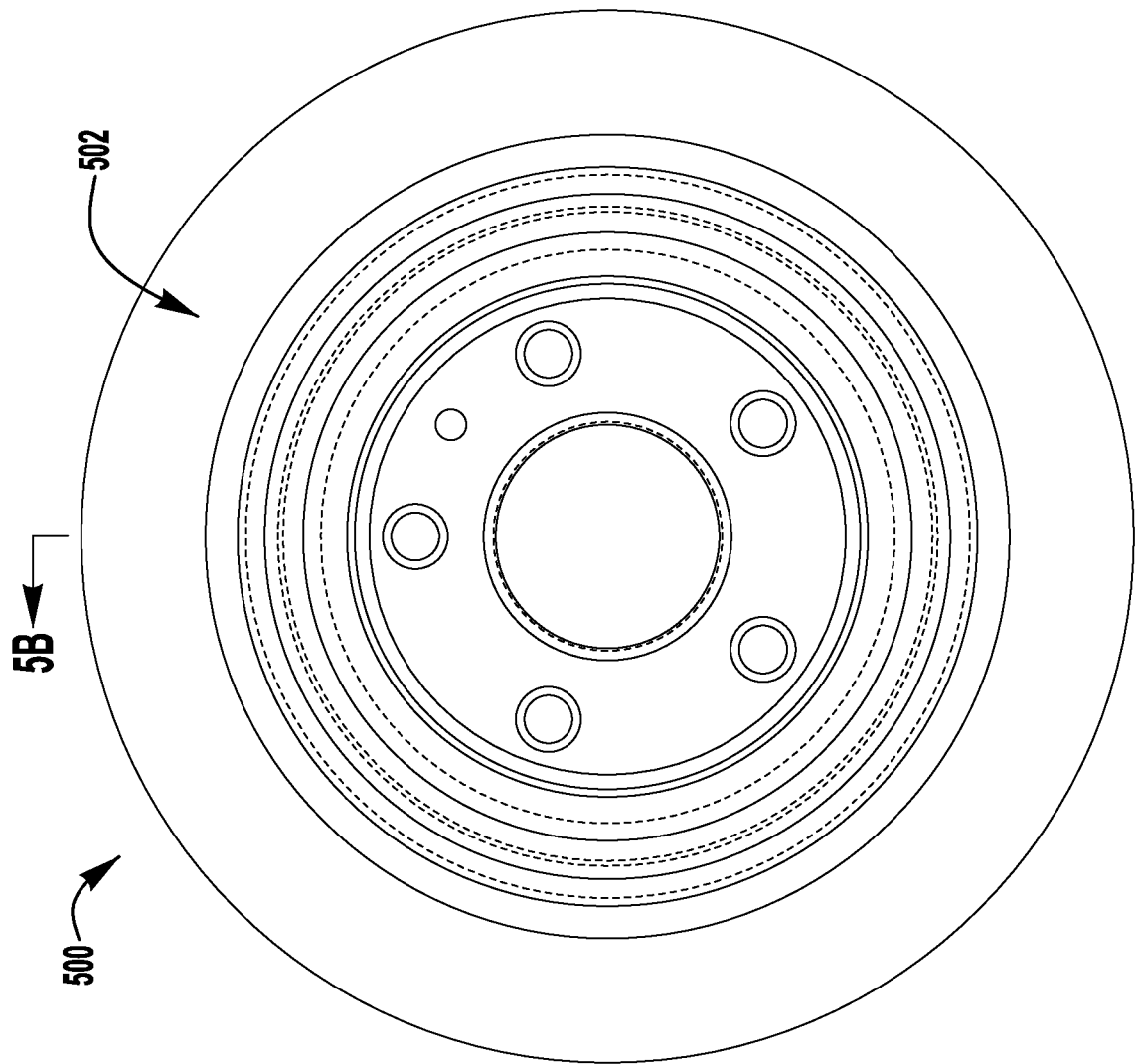


FIG. 5A

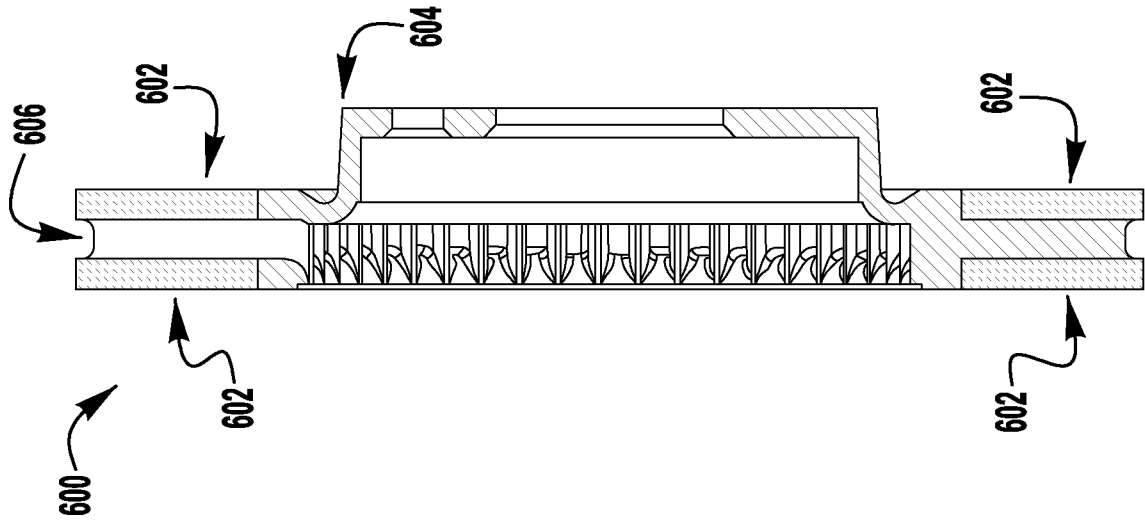


FIG. 6B

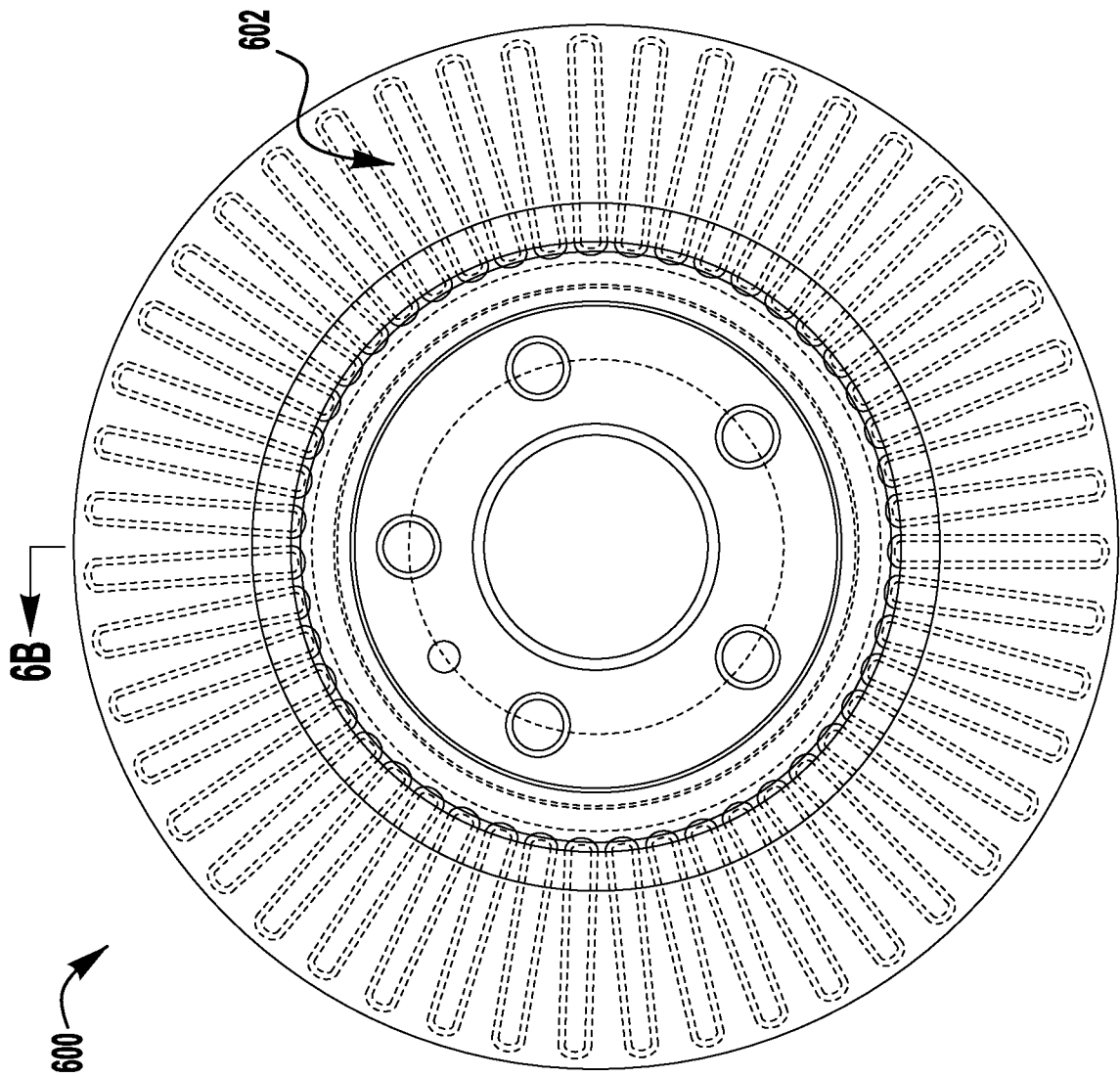


FIG. 6A

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 14/64624

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - B28B 11/00 (2015.01)

CPC - C04B 41/009; C04B 2111/00948; B28B 1/00

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)
IPC(8)-B28B 11/00 (2015.01)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
CPC-C04B 41/009; C04B 2111/00948; B28B 1/00
USPC-428/34.4,34.5

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
PatBase, Google Patents, Google Scholar (without Patents)

Keywords: ceramic preform detangling reinforcing fibers individual strands fibers ceramic particles fugitive porosity generating component starch low temperature organic binder colloidal silica

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US 2013/0295304 A1 (Moore et al.) 07 November 2013 (07.11.2013) Abstract, para [0025], para [0026], para [0027], para [0036], para [0052], para [0066], para [0063], para [0066], para [0037]	13-14,18-24 ----- 1-12,15-17
Y	US 2012/0227624 A1 (Loukus et al.) 13 September 2012 (13.09.2012) Abstract, para [0078], para [0059], para [00230]-para [00236]	1-12,15-17

Further documents are listed in the continuation of Box C.

* 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 application or patent but 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	

Date of the actual completion of the international search
13 July 2015 (13.07.2015)

Date of mailing of the international search report

11 AUG 2015

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