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(54) Title: METHODS FOR PASSIVATING METAL POWDER CONDENSATE FROM ADDITIVE MANUFACTURING PROCESSES

(57) Abstract: A method for passivating metal-containing powder condensate and the resulting passivated metal. A metal-containing powder condensate comprising at least one metal is combined with at least one binder and at least one solvent to form a slurry, wherein the solvent only partially dissolves the powder-binder mixture. The resulting slurry is dried to remove excess solvent, forming a passivated metal-binder solid cake.

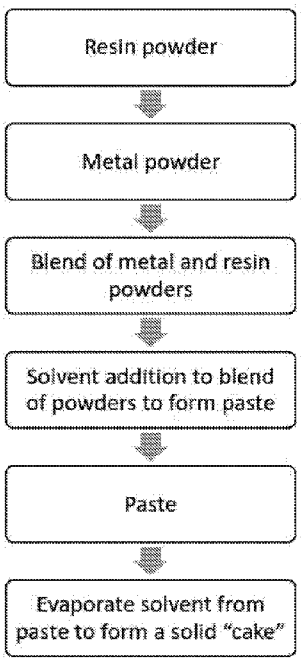

FIG. 1

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METHODS FOR PASSIVATING METAL POWDER CONDENSATE FROM ADDITIVE MANUFACTURING PROCESSES

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to U.S. Provisional Patent Application No. 63/341,685, filed on May 13, 2022, which is incorporated by reference herein.

TECHNICAL FIELD

[0002] The present invention relates to methods for passivating metal-containing powder condensate from powder bed fusion additive manufacturing processes.

BACKGROUND

[0003] During additive manufacturing (AM) processes also known as 3D printing or powder bed fusion (PBF), potentially hazardous waste streams are produced. The waste streams contain solidified particles within a vapor plume resulting from evaporation of a metal or metal alloy being deposited into a build chamber within the additive manufacturing printer. These solidified particles are sometimes called powder condensate and often have hazardous properties or high risks of combustion and require appropriate disposal. Depending on the different filtering systems on the printing machines, the powder condensate may also contain chalk, silica sand, or other non-metallic powders that are part of the current state of the art process to inert the powder condensate.

[0004] Powder condensates also contain one or more metals and/or metal alloys that could be economically recovered for future additive manufacturing processes or metal production. However, due to the hazardous nature of the powder condensate, transportation and reclamation of metal and/or metal alloys from the powder condensate can require multiple time-consuming and costly processes to satisfy hazardous material safety concerns as well as produce a functional feed powder for other additive manufacturing processes or metal production.

[0005] To safely handle the metals and/or metal alloys following the additive manufacturing process, powder condensate has been passivated in the past by mixing the powder condensate with material such as silica sand, glass beads, other non-metallic powders and/or inert liquids such as mineral oil. This process passivates the condensate and makes the transportation possible without risk of ignition, but the metal powder must be transported and disposed of as hazardous. Traditional passivation processes also do not always allow for economic recovery of the metal/metal alloy.

[0006] There is still a need, therefore, for a passivation treatment process that mitigates hazardous material storage and transport logistics and also allows for the efficient and effective recovery of metals/metal alloys from the condensate for future use.

SUMMARY

[0007] Methods for passivating metal-containing powder condensate and passivated metal-containing powder condensates are provided herein. In one method for passivation, a metal-containing powder condensate and at least one binder can be combined to form a powder-binder mixture, wherein the metal-containing powder condensate comprises at least one metal. The powder-binder mixture can then be combined with at least one solvent to form a slurry, wherein the solvent only partially dissolves the powder-binder mixture. The resulting slurry is then dried to form a passivated metal-binder solid. This drying step removes excess solvent from the slurry, forming a passivated metal cake. The passivated metal cake being a plurality of metal particles each substantially coated in binder.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] The present disclosure is best understood from the following detailed description when read with the accompanying Figures. It is emphasized that, in accordance with the standard practice in the industry, various features are not drawn to scale. In fact, the dimensions of the various features may be arbitrarily increased or reduced for clarity of discussion.

[0009] Figure 1 depicts a flow chart of an illustrative process for making a non-reactive metal powder cake, according to one or more embodiments described herein.

[0010] Figure 2 depicts a photograph of two non-reactive metal powder cakes.

DETAILED DESCRIPTION

[0011] It is to be understood that the following disclosure describes several exemplary embodiments for implementing different features, structures, and/or functions of the invention. Exemplary embodiments of components, arrangements, and configurations are described below to simplify the present disclosure; however, these exemplary embodiments are provided merely as examples and are not intended to limit the scope of the invention. Additionally, the present disclosure can repeat reference numerals and/or letters in the various exemplary embodiments and across the Figures provided herein. This repetition is for the purpose of simplicity and clarity and does not in itself dictate a relationship between the various exemplary embodiments and/or configurations discussed in the Figures. Moreover, the exemplary embodiments presented below can be

combined in any combination of ways, i.e., any element from one exemplary embodiment can be used in any other exemplary embodiment, without departing from the scope of the disclosure.

[0012] Described herein are methods for passivating metal-containing powder condensate from additive manufacturing processes and the resulting passivated metal-containing powder cakes. In certain embodiments, metal-containing powder condensate is mixed with one or more solid binder agents to provide a physical blend of the agent and the metal-containing condensate. This physical blend can then be at least partially solubilized in one or more solvents to provide a slurry or solution. In an alternative embodiment, the metal-containing powder condensate can be mixed in one or more solvents to provide a mixture of the solvent and condensate. One or more solid binder agents can then be added to this mixture to at least partially solubilize the binder agent to form a slurry or solution. In yet another alternative embodiment, the one or more solvents can be mixed with the one or more binder agents to create an at least partially solubilized mixture of these components, and the metal-containing powder condensate can be added to the mixture. The solvent can then be removed from the formed solution or slurry, by evaporation, for example, leaving behind a cake of the metal-containing powder particles substantially coated by the binder.

[0013] All numerical values within the detailed description and the claims herein are modified by "about" or "approximately" the indicated value, and take into account experimental error and variations that would be expected by a person having ordinary skill in the art.

[0014] Certain terms are used throughout the following description and claims to refer to particular components. As one skilled in the art will appreciate, various entities may refer to the same component by different names, and as such, the naming convention for the elements described herein is not intended to limit the scope of the invention, unless otherwise specifically defined herein. Further, the naming convention used herein is not intended to distinguish between components that differ in name but not function.

[0015] The term "or" is intended to encompass both exclusive and inclusive cases, i.e., "A or B" is intended to be synonymous with "at least one of A and B," unless otherwise expressly specified herein.

[0016] The terms "binder" and "binding agent" are used interchangeably, and both refer to any material that can adhere to another material and/or coat another material. Any suitable binder can be added to the powder condensation. Suitable binders can include,

for example, polyvinyl butyral (PVB), polylactic acid (PLA), polyurethanes, ethylene vinyl acetate (EVA), polycarbonates, polypropylene (PP), propylene elastomers, ethylene propylene rubber (EPR), ethylene propylene copolymers (EPC), polyisobutylene (PIB), styrene butadiene rubber (SBR), polyolefins, polyethylene-co-poly-1-octene (PE-co-PO), PE-co-poly(methylene cyclopentane) (PE-co-PMCP), acrylics, poly methyl-methacrylate, polyvinylacetacetal resin, polyvinyl acetal resin, stereoblock polypropylenes, polymethylpentene copolymer, polyethylene oxide (PEO), PEO block copolymers, molasses, and the like.

[0017] The term "solvent" refers to any liquid at room temperature that can at least partially dissolve or solvate the binder and powder condensate blend. Any suitable solvent can be used. Suitable solvents, include, but are not limited to, for example, water, silicone, molasses, organic and inorganic acids, any one or more aliphatic hydrocarbons, such as isobutane, butane, pentane, isopentane, hexanes, isohexane, heptane, octane, dodecane, and mixtures thereof; cyclic and alicyclic hydrocarbons, such as cyclohexane, cycloheptane, methylcyclohexane, methylcycloheptane, and mixtures thereof. In an embodiment, the solvent is not aromatic, or aromatics are present in the solvent at less than 1 wt%, or less than 0.5 wt%, or less than 0.05 wt%, based upon the weight of the solvents. More specific solvents can include ethanol, isopropyl alcohol (IPA), isopentane, hexane.

[0018] As used herein the phrase "removing a solvent," refers to the process whereby a solvent is removed or separated from the components or materials set forth herein. The solvent can be removed using any conventional separation technique, including crystallization, evaporation, filtration, membrane separation, distillation, vacuum or other reduced pressure separation techniques. The conditions to effectuate an efficient separation of the solvent could be readily determined by those of ordinary skill in the art of chemical separations.

[0019] As used herein, the term "metal-containing" refers to a material that is or contains at least one metal or a mixture of a metal and at least one other chemical element. A "metal-containing powder condensate" refers to any by-product of an additive manufacturing processes that uses one or more metals or metal alloys. The metals can be selected from any of the metals of Group 3 to Group 12 of the Periodic Table of Elements and/or other metals from Groups 13-15. For example, the metals can be, but not limited to, iron, nickel, titanium, tungsten, cobalt, copper, chrome, gold, silver, platinum, rhodium, mercury or any combinations thereof. Other metals can be aluminium, tin, or

lead. Illustrative alloys can be or can include any two or more metals described herein. Other illustrative alloys can be or can include steel, stainless steel, silicon steel, solder, brass, pewter, duralumin, red gold, white gold, sterling silver, bronze, and amalgams. In a preferred embodiment, the metal-containing powder condensate is a metal alloy with a nickel base.

[0020] A more detailed description of the methods for passivating metal-containing powder condensate from additive manufacturing processes will now be described with reference to the figures provided. Figure 1 depicts an illustrative flow chart of a process for making a non-reactive metal powder cake. A metal-containing powder condensate can be collected from an additive manufacturing process or any other source of metal-containing powder condensate. For example, the metal-containing powder condensate can be collected from an additive manufacturing process whereby the condensate is generated when a laser in a 3D printer, for example, vaporizes a small percentage of the metal/metal allow powder being used to make a part in the build area of the printer. This vapor is flushed from the build area via a gas flow and condensed on a filter that cleans the flowing gas. The condensed powder falls from the filter and is then collected in solid form in a collection bin. This metal-containing powder condensate can then be combined with one or more binders or binding agents to provide a physical mixture or blend thereof. This blend can then be mixed with one or more solvents to at least partially dissolve the binder/metal blend to form a solution or slurry. The resulting solution or slurry can be homogeneous or not. In some embodiments, the blend can be completely dissolved into a homogenous or non-homogenous solution or only partially dissolved into a slurry or paste that can be homogenous or non-homogenous. The solvent is preferably mixed into the blend of the metal-containing powder condensate and binder to provide a homogenous mixture.

[0021] Alternatively, the solvent can first be added to the metal-containing powder condensate and then the binder can be added to the mixture of the solvent and metal-containing powder condensate. In another alternative embodiment, the one or more solvents can be mixed with the one or more binder agents first to create an at least partially solubilized mixture of these components, and then the metal-containing powder condensate can be added to the solution or slurry of the one or more solvents and binder(s).

[0022] After the formation of the slurry or solution, a desirable amount of the solvent can be removed. For example, the solvent can be subjected to thermal energy and evaporated,

resulting in a passivated metal cake. The passivated metal cake being a plurality of metal or metal alloy particles each substantially coated in the binder. In one or more embodiments, the removed solvent can be condensed and recycled to treat another blend batch. Preferred solvents can be evaporated at atmospheric pressure at temperatures less than 300°C, 250°C, 200°C, or 150°C.

[0023] The coated metal-containing powder particles within the cake are passivated using the binder coating and thus, are non-reactive such that the metal cake does not explode, combust, corrode, or otherwise react with its surrounding environment. The non-hazardous and non-explosive, passivated metal cakes can be stored, transported, and recycled, without regulatory restrictions. Upon receipt of the passivated powder, the binder can be separated from the metal through the appropriate use of heat, chemical treatments, and the like. The metal will then be suitable as raw material to produce new metal products or powders.

[0024] Each non-reactive metal-containing powder cake can have a mass ratio of metal powder to binder. The mass ratio of metal-containing powder condensate to binder preferably ranges from a low of 1 kg/22 g, 1 kg/20 g, or 1 kg/18 g, to a high of 1 kg/12 g, 1 kg/10 g, or 1 kg/8 g. The binder/metal-containing powder condensate blend can contain at least 50 wt%, 60 wt%, 70 wt%, 80 wt%, 90 wt%, 95 wt% or 99 wt% of the metal condensate, based on the total weight of the blend; the balance being the one or more binders.

[0025] When the one or more solvents are added to the binder/metal-containing powder condensate blend, the resulting mixture can contain at least 5 wt%, 10 wt%, 15 wt%, 20 wt%, 35 wt%, 55 wt%, 65 wt%, 80 wt%, 85 wt%, 90 wt%, or 95 wt% of the blend, the balance being solvent. The amount of the metal-containing powder condensate in the resulting mixture can range from a low of about 15 wt%, 25 wt% or 30 wt% to a high of about 50 wt%, 70 wt%, or 90 wt%.

[0026] While compositions and methods are described herein in terms of "comprising" various components or steps, the compositions and methods can also "consist essentially of" or "consist of" the various components and steps. As used herein "consisting essentially of" means that the described/claimed composition does not include any other

components that will materially alter its properties by any more than 5% of that property, and in any case, does not include any other component to a level greater than 3 wt%.

[0027] As used herein, "substantially no," and "substantially free of" are intended to mean that the subject item is not intentionally used or added in any amount but can be present in very small amounts existing as impurities resulting from environmental or process conditions.

[0028] To provide a better understanding of the embodiments of the present invention, the following non-limiting examples of preferred or representative embodiments are given. In no way should the following examples be read to limit, or to define, the scope of the invention.

EXAMPLES

[0029] Three examples were prepared as summarized in Table 1 below. Examples 1 and 2 were Haynes 282 metal alloy powder condensates and were filtered using a filtering system that does not contain chalk. Example 3 was an Inconel 718 metal alloy powder condensate and was filtered using a filtering system that contained chalk.

[0030] Examples 1 and 2 were mixed with a PVB binder using a plastic spoon while adding IPA solvent to form a paste of desired consistency such that the fluidity of the mixture allowed for the metal powder to be fully coated in liquidus resin. The slurries were left to dry at ambient temperature (about 23°C) on an aluminum tray with parchment paper to prevent sticking to the tray.

[0031] Table 1: Blend compositions of Examples 1-3

	Powder (grams)	Binder (grams)	Solvent (mL)
Example 1	250	5	40
Example 2	1,000	15	150
Example 3	237	5	40

[0032] The three slurries formed a dense paste that air dried after a few hours. Examples 1-2 were not a continuous solid and with little pressure became crumbs. Example 3 was a complete and continuous solid.

[0033] Figure 2 depicts a photograph of two non-reactive metal powder cakes. The cake on the left 210 had a mass ratio of 1 kg of Inconel 718 metal to 20 g PVB binder. The cake on the right 220 had a mass ratio of 1 kg of Haynes 282 metal alloy to 10 g PVB binder. As depicted, both powder cakes looked like burned hamburger patties with similar consistency. Both were continuous solids and had little to no crumbs.

[0034] During the trials no hazardous situations were reported, and the air quality remained within acceptable limits.

[0035] Preliminary results from these trials determined that Example 3, that was 237g of metal-containing powder condensate mixed with 5 g of PVB and 40 mL of IPA, formed a solid, passivated cake that can be safely transported. While Examples 1 and 2 were also successful in creating a solid passivated cake, they resulted in a non-optimal, brittle solid.

[0036] Various terms have been defined above. To the extent a term used in a claim is not defined above, it should be given the broadest definition persons in the pertinent art have given that term as reflected in at least one printed publication or issued patent. All numerical values in this disclosure may be exact or approximate values unless otherwise specifically stated. Accordingly, various embodiments of the disclosure may deviate from the numbers, values, and ranges disclosed herein without departing from the intended scope.

[0037] Furthermore, all patents, test procedures, and other documents cited in this application can be fully incorporated by reference to the extent such disclosure is not inconsistent with this application and for all jurisdictions in which such incorporation is permitted.

[0038] While the foregoing is directed to more preferred embodiments of the present invention, other and further embodiments of the invention may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

CLAIMS

What is claimed is:

1. A method for passivating metal-containing powder condensate, comprising:

combining a metal-containing powder condensate and at least one binder to form a powder-binder mixture, wherein the metal-containing powder condensate comprises at least one metal;

combining the powder-binder mixture with at least one solvent to form a slurry, wherein the solvent only partially dissolves the powder-binder mixture; and

drying the slurry to form a passivated metal-binder solid, wherein drying removes excess solvent from the slurry.

2. The method of claim 1, wherein the at least one metal is nickel.

3. The method of claim 1, wherein the binder is polyvinyl butyral.

4. The method of claim 1, wherein the solvent is isopropyl alcohol.

5. The method of claim 1, wherein the drying is performed by evaporation.

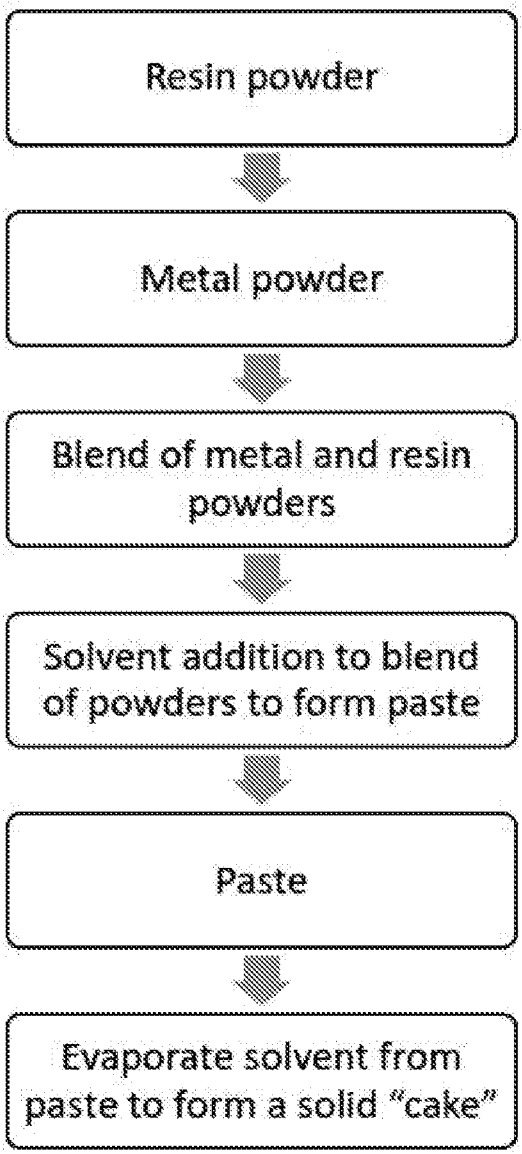
Resin powder
Metal powder
Blend of metal and resin powders
Solvent addition to blend of powders to form paste
Paste
Evaporate solvent from paste to form a solid "cake"

FIG. 1

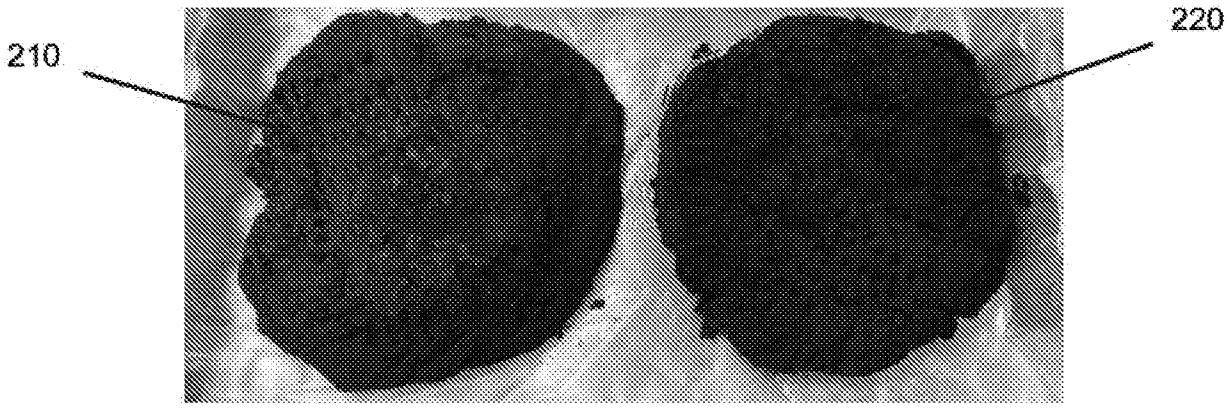

FIG. 2