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EP 3257608 A
WO 2018/071118 A1
WO 2016/165746 A1
WO 2002/044962 A1
RU 002544047 C1
US 20100140550 A1
EP 0959342 A
WO 2017/036868 A1
WO 2010/036523 A2
WO 2002/044692 A1
US 20170203387 A1

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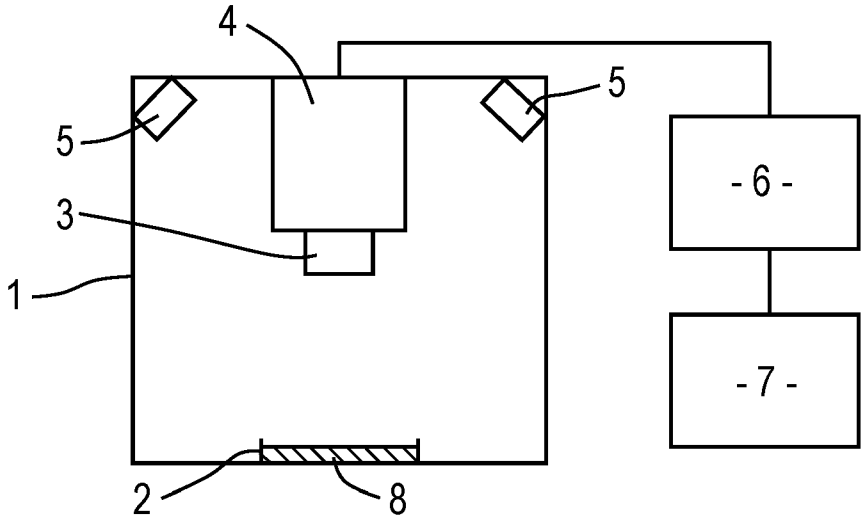

Fig. 1

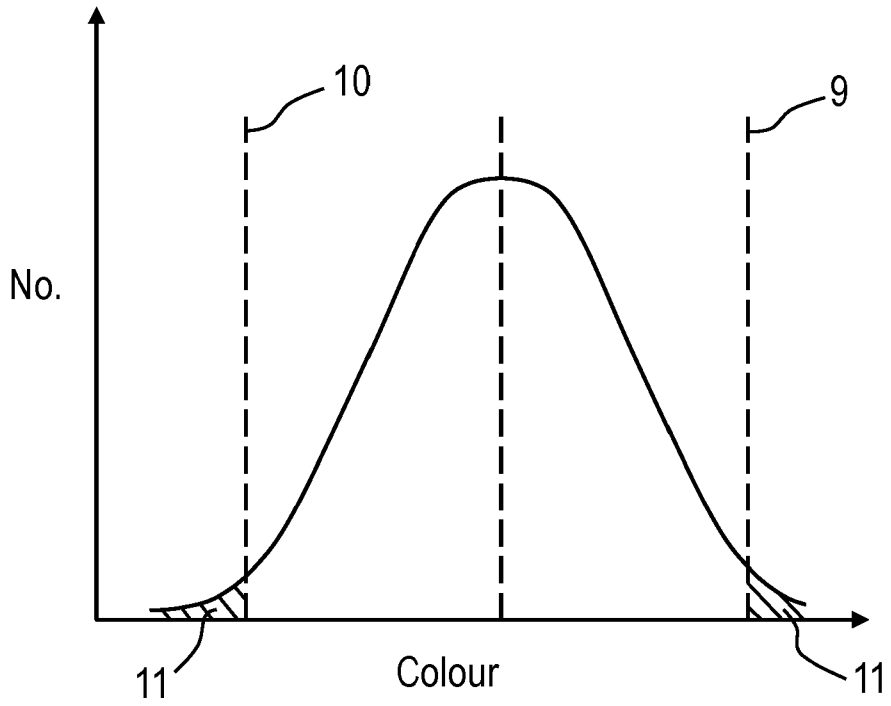

Fig. 2

METHOD AND APPARATUS FOR DETERMINING POWDER CONDITION

Technical Field of the Invention

The present invention relates to a method and apparatus of determining the condition of a metal powder, used in an additive manufacturing (AM) process.

Background to the Invention

In a known AM process an AM machine produces articles from a powdered material, such as a metal or alloy. The machine deposits a layer of powder on a build platform and the powder is subsequently selectively fused or otherwise solidified, typically with a laser or electron beam, to form an article or articles. The process is repeated so that articles are formed layer by layer.

On completion of a build, unfused powder may be re-used in another build.

During a build operation unfused powder is subject to degradation. A metal powder may gradually oxidise, for example, which alters its properties and thus those of an article produced from the powder. The tendency of a powder to oxidise typically increases with temperature, and exposure to temperature may also affect other powder properties. Consequently, the nearer unfused powder is to an article being built, or a heat zone, the more likely it is to suffer degradation.

Also, when powder is fused the process may cause some heated particles of powder to be scattered from the powder bed around the manufactured article, degrading the quality of the unfused powder around the article.

To ensure adequate build quality of an article it is known to analyse used powder and stop recycling the powder when it has been degraded to a certain extent and/or to

blend virgin powder with recycled powder so that the blended powder has an adequate bulk property for continued use. In an alternative approach a fixed upper limit is imposed on the number of times a batch of powder is recycled.

There are a number of problems with these approaches.

Powder condition is typically analysed by making a bulk oxygen content measurement. The measurement process involves oxidising a powder sample, which cannot then be re-used. More significantly, it has now been realised that a bulk oxygen content (or other bulk) measurement can give a false impression as to suitability of a powder for re-use, especially where recycled powder is blended with virgin powder to produce a blend with an overall bulk oxygen content below a desired threshold. This is because it is not sensitive to the presence of highly oxidised or otherwise degraded particles which may have a significant deleterious effect on a build even though the bulk oxygen content is below a desired threshold.

Applying a general limit to the number of times a powder is recycled is a relatively crude approach and does not take account of the likely amount of powder degradation caused by a specific build. The nature and extent of degradation can vary considerably between builds.

It is an object of embodiments of the present invention to address some or all of these problems. In particular it is an object of embodiments of the invention to provide improved methods and apparatus for determining metal powder condition in a non-destructive way which can more accurately determine if the metal powder may be re-used in a particular build.

Summary of the Invention

According to a first aspect of the present invention there is provided a method of determining the condition of a metal powder for use in an additive manufacturing process, the method comprising measuring the colour of the powder.

The method may comprise the steps of:

measuring the colour of the powder; and

determining the proportion of the powder whose measured colour falls outside a pre-determined range.

According to a second aspect of the invention there is provided an apparatus for determining the condition of a metal powder for use in an additive manufacturing process, the apparatus comprising:

a measuring device for measuring the colour of the powder; and

a processor arranged to determine the proportion of the powder whose measured colour falls outside a pre-determined range.

It has been observed that oxidation and other degradation of powders, particularly metal powders, alters surface properties of particles of the powder, such as colour. This, in turn, has a similar effect on the measured colour of a surface of a body of powder. Determining a proportion of a measured colour of a powder (particularly the proportion of a measured surface of the powder) falling outside a predetermined range thus approximates to the proportion of particles of the powder whose individual colours fall outside that range and thus the proportion of particles which exhibit a certain degree of oxidation or other degradation. This allows a decision to be made not to re-use the powder when the proportion of such particles exceeds a threshold. Thus

the method and apparatus allow for the identification of powders where the proportion of highly oxidised or otherwise degraded particles has reached an unsafe level for re-use of the powder.

The measurement may be an optical measurement. The image may be an optical image. The measurement may be a spectroscopic measurement and may be a measure of reflectance or transmission of the powder as a function of wavelength.

The method may involve taking an image of the powder, which may be taken as a single image or by combining multiple images or by scanning a surface to be imaged. The method may also involve processing the image to measure the colour of the observed powder. It may also further involve processing the image to determine the proportion of the image which shows the measured colour outside the predetermined range and thus to determine the approximate proportion of powder or observed particles of the powder having the measured colour outside the predetermined range. To facilitate this the image may be divided into a plurality of elements, the measured colour or average measured colour of each element determined and the proportion of elements having a measured colour or average measured colour outside the predetermined range determined.

The image may comprise a plurality of pixels. In this case each region may conveniently consist of a single pixel. It is preferred that the ratio of pixels in the image to the number of particles in the imaged region or surface of the powder is at least of the order of 1:1. That way the colour of a particular pixel is likely to be influenced only by the colour of a single particle or small number of particles, say no more than four or

so. In this way the calculated proportion of measured colours will more accurately correspond to the proportion of particles having those properties.

The method may involve the step of indicating that the tested powder is not suitable for re-use when the proportion of measured colour that falls outside the pre-determined range exceeds a predetermined value. The pre-determined range and the proportion may be established depending on the particular powder being analysed and its intended use. Typically, though, as the intention is to identify the presence of significantly oxidised or degraded particles the range is preferably set to encompass values for the measured colour reflective of powder that has suffered what may be regarded as normal degradation as a result of being used in a build process, as might typically be caused by exposure to oxygen and low temperatures. Thus, those particles having a measured colour outside of this range are outliers. Their measured colour reflects the fact that they have been exposed to abnormal degradation, typically as result of being exposed to a high temperature (but without becoming fused to form part of a constructed article). It is thought that when the population of such outliers exceeds a certain proportion of the overall population of particles re-use of the powder carries an increased risk.

The method may also involve determining the average measured colour of the proportion of measured powder, and thus the approximate proportion of measured particles, whose measured colour falls within the predetermined range. This measure is indicative of the overall average degradation of the powder excluding the outlying significantly degraded particles. This measure therefore gives an indication of the level of degradation resulting from normal degradation.

The method may also involve indicating that the tested powder is not suitable for re-use when average measured colour of the proportion of powder, and thus approximate proportion of measured particles, whose measured colour falls within the predetermined range is greater or less than a predetermined threshold. Thus, powder can be indicated as no longer suitable for re-use as a result of the, possibly cumulative, effect of normal degradation.

The method may also involve determining the average measured colour of all of the measured powder, and so all measured particles. Such a measure is indicative of the overall average degradation of all the particles, and so may give a similar indication to a bulk oxygen measurement, save that a bulk oxygen measurement will also measure "internal" oxygen of a particle, that being oxygen present inside a particle, as well as oxygen of any oxide outer layer the build-up of which affects a surface property of the particle.

The method may also involve indicating that the tested powder is not suitable for re-use when average measured colour of all the powder, and so all measured particles, is greater or less than a predetermined threshold.

Where an average measured colour of a powder, and thus particles of interest, is required this may be a mean, and may be obtained by determining the average measured colour represented by those parts of an image showing the particles of interest. The parts of the image may be elements of the image or pixels forming the image.

In an alternative approach the method may involve processing an image to resolve individual particles and measuring the colour or average colour of each

individual particle. This information can then be used to compute the various measures discussed above. Of course, it will be appreciated that any suitable image processing technique may be employed to derive the desired information or measures or equivalents.

The measurement may be made over a surface of the powder. The measurement may be made over a substantially planar surface of the powder. The method may involve placing the powder into a suitable container so that the powder has a substantially planar upper surface, and then performing the measurement over that surface. The powder preferably has sufficient depth so that the bottom of the container or other surface on which it is supported is not visible through the powder. The powder may be a sample of powder taken from a batch of powder, such as a batch of powder recovered from an AM machine following a build operation.

The measuring device may be a spectroscopic device such as a spectrophotometer, an imaging device or some other suitable device or sensor. Where the measuring device is an imaging device it may comprise a microscope and/or camera and/or sensor. Indeed, any appropriate image capture device capable of measuring an observable colour of particles of powder may be used.

The measurement and/or image may be taken in an enclosure. The enclosure maybe substantially light tight. The interior of the enclosure may be illuminated.

The processor may be a programmed computer, and may be arranged to cause the apparatus to perform some or all of the method steps discussed above.

As oxidation or other degradation of particles of a powder may alter their colour, measuring the colour of powder is a convenient way to assess degradation. Colour of the powder may be measured using any suitable technique.

The method may comprise taking an image of the powder. The image may be an optical image. The method may further comprise processing the image to measure the colour of the powder, and preferably of observed particles of the powder.

The average colour of the powder, or observed particles of the powder, may be measured, such as by processing an image of the powder. It may be determined whether or not tested powder is suitable for re-use depending whether or not the measured colour is inside or outside a predetermined range. The method may also comprise features of the first aspect of the invention.

According to a fourth aspect of the invention there is provided apparatus for determining the condition of a metal powder for use in an additive manufacturing process, the apparatus comprising a measuring device for measuring the colour of the powder.

The measuring device may be a spectroscopic device such as a spectrophotometer, an imaging device for taking an image, or some other suitable device or sensor. Where the measuring device is an imaging device it may be for taking an optical image and may comprise a microscope and/or camera and/or sensor. Indeed, any appropriate image capture device capable of measuring an observable surface property of particles of powder may be used.

Where colour of the powder is not measured directly the apparatus may further comprise a processor arranged to determine the colour of the powder from measured

information. For example, the processor may determine the colour of powder form an image of the powder. The processor may be arranged to perform the method of the third aspect of the invention. The apparatus may include features of the second aspect of the invention.

Embodiments of aspects of the invention provide a non-destructive method and apparatus for determining metal powder condition and deciding whether or not a metal powder sample is suitable for re-use in a particular build operation. Where the determination is made by looking at the proportion of outlier particles this provides a new and useful measure of metal powder condition which enables improved decision making, and therefore metal powder use, over current measurements of bulk powder properties.

The method and apparatus are also useful for identifying the presence of contaminant particles where those particles a surface property which differs to the same property of particles of interest.

Detailed Description of the Invention

In order that the invention may be more clearly understood one or more embodiments thereof will now be described, by way of example only, with reference to the accompanying drawings, of which:

Figure 1 is a schematic view of apparatus for analysing powder condition; and

Figure 2 is a graph showing number of particles against colour.

Referring to the drawings, apparatus for analysing metal powder comprises an openable substantially light tight enclosure 1. The enclosure houses a container 2 for

metal powder 8 which may take the form of a dish or slide, or any other suitable form. The container is open to the top and has a substantially square opening with a side of about 6mm, giving it a surface area of about 36mm^2. It has a depth of at least 2mm. The container may be removed from the enclosure. The enclosure also houses a microscope 3 which is mounted to a digital camera 4, and lamps 5. The camera 4 comprises a substantially square sensor, such as a CCD sensor, with approximately four mega pixels and is connected to a computer 6 which comprises a keyboard and mouse or other user interface and is connect to a display 7 and/or other output device.

In use, a sample of metal powder 8 taken from a batch of metal powder to be analysed is introduced into the container, either with the container in or out of the enclosure 1. The metal powder is introduced in sufficient quantity to form a depth of powder which entirely obscures the bottom of the container 2 when viewed from above. So the depth of metal powder typically comprises at least two, and preferably more than two, layers of particles. The powder is also levelled in the container so that it has a substantially flat upper, planar surface. If powder has been introduced into the container whilst outside the enclosure, the container is then positioned in the enclosure beneath the microscope and the enclosure closed.

The lamps 5 are then activated. The lamps may be controlled by the computer 6. The lamps are arranged to illuminate the powder 8 in the container 2. Illuminating the metal powder with lamps in a substantially light tight enclosure enables powder to be analysed in repeatable light conditions.

The camera 4 is then caused to take an image of the upper surface of the metal powder in the container and to transmit it to the computer 6. The camera and microscope

are arranged to take an image of substantially all of the surface of the metal powder in the container. The field of view of the camera and microscope thus images an area of about 36mm^2. Metal powders used in AM processes typically have an average diameter of the order of tens of microns. As such, the number of particles visible to the surface of the powder imaged by the camera is of the same order of, or approximately corresponds to, the number of pixels of the sensor. So the image produced by the camera is substantially able to resolve individual particles of the metal powder.

The image taken by the camera is then processed by the computer 6. Principally the computer is arranged to determine the colour detected by each pixel. The information is stored by the computer 6 in any suitable form, for example using an RGB colour scheme and recoding the proportions of red, green and blue light observed by each pixel of the sensor. A visual representation of example data taken from an image of a metal powder sample is shown in figure 2 with the number of pixels, and thus an adequate approximation of the number of particles, plotted against colour. The plotted points form a smooth generally bell-shaped curve.

Having done this, the computer is the arranged to determine one or more of the following measures:

- The proportion of pixels having a colour which lies outside a predetermined range. This is also shown in figure 2, the range being defined between the broken lines 9 and 10 which define upper and lower colour limits respectively, and the number of pixels lying outside the threshold thus being represented by the shaded regions 11. It will be appreciated that predetermined range could be defined by only a lower

or upper limit. The desired proportion is thus represented by the ratio of the area of the shaded regions and the total area under the curve.

- The mean colour of pixels lying within the predetermined threshold.
- The mean colour of all pixels.

It has been found that the colour of metal particles changes as the particles degrade. In particular it changes as particles oxidise and/or are exposed to heat. The more a particle is oxidised or the higher temperature a particle is exposed to the greater its colour changes. So, the amount of colour change is related to the degree of degradation a particle has suffered and thus also its suitability for re-use.

It has further been found that, notwithstanding the average condition of a batch of powder, the presence of highly degraded particles can render the batch unsuitable for re-use. This is because inclusion of even a single highly degraded particle in a build can significantly affect properties of the build. Where a highly degraded particle or particles become(s) incorporated into a article this could render the article unsafe, especially if the particle(s) is/are incorporated into the article at a location where there will be a stress concentration in use.

As the number of pixels of the camera sensor approximates to the number of particles visible in the upper surface of the powder sample the colour of each pixel approximately represents the colour of a single particle. The first measure above thus enables the number or proportion of significantly degraded particles present in the observed sample to be determined. Assuming that the batch of powder from which the sample is taken is well mixed this proportion will generally mirror the proportion of significantly degraded particles throughout the sample and throughout the batch,

although multiple samples may be taken from a given batch and analysed separately in order to improve accuracy. And/or a particular sample could be analysed, mixed, and then reanalysed. An appropriate colour range and threshold minimum proportion outside that range can be determined for a given powder and build and where the proportion of particles outside the threshold exceeds the chosen limit the batch of powder is deemed unsuitable for re-use, at least for the build in question.

Thus, this measure enables metal powder condition to be determined independent of a bulk quantity.

The second measure provides an indication of the average degradation of the remaining powder when the particles with colours lying outside the threshold have been discounted. Such a measure is more akin to the result of a conventional bulk oxygen measurement, but obtained in a more convenient and non-destructive way, save that it excludes the influence of significantly degraded particles (or any internal oxygen). Powder may be deemed unsuitable for re-use where the average colour of the remaining particles, when the particles with colours lying outside the predetermined range have been discounted, lies outside another predetermined range of colours.

The third measure is similar to the second measure, but takes account of the significantly degraded particles. Metal powder may be deemed unsuitable for re-use where the average colour of particles lies outside another predetermined range of colours.

A decision whether or not to re-use metal powder can be based on one or more of the three measures described above. Typically a metal powder would not be re-used if any measure determines that the powder should not be re-used. In one embodiment

the first and second measures are calculated and metal powder deemed unsuitable for re-use if either measure indicates this.

The computer is provided with suitable software to cause the camera to take an image, to process the image to determine colour distribution amongst pixels, to enable a user to input ranges and proportions, to calculate one or more of the three measures, to determine if a particular sample may or may not be re-used having regard to the range(s) and proportion specified by a user and to output this result to a user via the display 7 or otherwise.

In one example a sample of used Ti64 alloy powder was analysed using the described apparatus. The image of the metal powder produced showed the vast majority of the powder to have a silver/grey colour generally similar to the colour of virgin powder, and a very small proportion to have a green, brown, blue, purple or black colour, these colours being indicative of increased oxidation or other degradation. A pre-determined range of colour was therefore chosen to encompass silver/grey particles and exclude the other colours. This range effectively encompassed particles which had suffered no or limited oxidation and been subjected only to low temperatures. A proportion of particles having a colour outside this range, effectively a proportion of particles that have suffered significant oxidation or degradation through exposure to high temperatures, could then be chosen as the limit beyond which the powder should not be re-used. When the colour of the observed particles was plotted in the manner shown in figure 2 it could be seen that the particles with colours other than silver/grey brown effectively represented outliers to the general distribution of particles.

The above embodiments are described by way of example only. Many variations are possible without departing from the scope of the invention as defined in the appended claims.

CLAIMS

1. A method of determining the condition of a metal powder, the method comprising measuring the colour of the powder.

2. A method as claimed in claim 1 comprising taking an image of the powder.

3. A method as claimed in claim 2 comprising processing the image to measure the colour of the powder.

4. A method as claimed in any of claims 1 to 3 wherein the average colour of the powder is measured.

5. A method as claimed in claim 4 wherein the average colour of measured particles of the powder is measured.

6. A method as claimed in any of claims 1 to 5 comprising the step of indicating that the powder is not suitable for re-use when the measured colour is outside a predetermined range.

7. A method as claimed in any preceding claim wherein the measurement is an optical measurement.

8. A method as claimed in any preceding claim wherein the measurement is a measurement of the reflectance or transmission of the powder as a function of wavelength.

9. A method as claimed in any preceding claim comprising the step of processing the image to determine the proportion of the image which shows the measured colour outside the predetermined range.

10. A method as claimed in claim 9 wherein the image is divided into a plurality of elements and the measured colour or average measured colour of each element is determined and the proportion of elements having a measured colour or average measured colour outside the predetermined range is determined.

11. A method as claimed in claim 10 wherein the image comprises a plurality of pixels and each region consists of a single pixel.

12. A method as claimed in claim 11, wherein the ratio of pixels in the image to the number of particles in the imaged region of the powder is at least about 1:1.

13. A method as claimed in any of claims 6-12 comprising the step of indicating that the tested powder is not suitable for re-use when the proportion of measured powder whose measured colour falls outside the pre-determined range exceeds a predetermined value.

14. A method as claimed in any of claims 6-13 comprising determining the average measured colour of the measured powder whose measured colour falls within the predetermined range.

15. A method as claimed in any of claims 6-14 comprising the step of indicating that the tested powder is not suitable for re-use when average measured colour of the powder whose measured colour falls within the predetermined range is greater or less than a predetermined threshold.

16. A method as claimed in any preceding claim comprising determining the average measured colour of all of the measured powder.

17. A method as claimed in claim 16 comprising the step of indicating that the tested powder is not suitable for re-use when the average measured colour of all the measured powder is greater or less than a predetermined threshold.

18. A method as claimed in any of claims 15 to 17 when dependent, directly or indirectly, on claim 2 wherein determining the average measured colour of the, or a proportion of the, powder is achieved by determining the average measured colour of the or relevant parts of the image.

19. A method as claimed in claim 18 wherein the parts of the image are elements of the image or pixels forming the image.

20. A method as claimed in any of claims 2-19 comprising the step of processing the image to resolve individual particles and measuring the colour of each individual particle.

21. A method as claimed in any preceding claim wherein the powder has a surface and the colour of that surface is measured.

22. A method as claimed in claim 21 wherein the surface is substantially planar.

23. A method as claimed in any preceding claim comprising the step of placing the powder into an enclosure and illuminating the powder in the enclosure when measuring the colour.

24. Apparatus for determining the condition of a metal powder for use in an additive manufacturing process, the apparatus comprising:

a measuring device for measuring a colour of the powder; and

a processor arranged to process an image of the powder to determine the proportion of the powder whose measured colour falls outside a pre-determined range.

25. Apparatus as claimed in claim 24 wherein the processor is arranged to perform the method of any of claims 1 to 23.

26. Apparatus as claimed in either claim 24 or 25 wherein the processor is a programmed computer.

27. Apparatus as claimed in any of claims 24 to 26 wherein the measuring device comprises a microscope.

28. Apparatus as claimed in any of claims 24 to 26 wherein the measuring device comprises a camera.

29. Apparatus as claimed in any of claims 24 to 26 wherein the measuring device comprises a sensor.

30. Apparatus as claimed in any of claims 24 to 26 wherein the measuring device comprises a spectrophotometer.

31. Apparatus as claimed in any of claims 24 to 26 comprising an illuminated enclosure for receiving the powder, enabling the measuring device to measure the colourof the powder under repeatable light conditions.

32. Apparatus for determining the condition of a metal powder for use in an additive manufacturing process, the apparatus comprising a measuring device for measuring the colour of the powder.

33. Apparatus as claimed in claim 32 comprising a processor and the processor is arranged to perform the method of any of claims 1 to 23.