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(71) Applicant (for all designated States except US):  
BRITISH NUCLEAR FUELS PLC [GB/GB]; Risley, Warrington, Cheshire WA3 6AS (GB).

(72) Inventors; and

(75) Inventors/Applicants (for US only):  
BROMLEY, Anthony, Phillip [GB/GB]; 8 Crofters Lea, Yeadon, Leeds LS19 7WE (GB).  
CUBBON, Robert, Charles, Patrick [GB/GB]; 158 Northwood Lane, Clayton, Newcastle, Staffordshire FY5 4BZ (GB).

(74) Agent:  
JOHNSTONE, Helen, Margaret; Urquhart-Dykes & Lord, Tower House, Merrion Way, Leeds LS2 8PA (GB).

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MANUFACTURE OF ARTICLES WITH CONTROLLED DENSITY DISTRIBUTION

(57) Abstract

An article is formed from particulate material using a method comprising the steps of forming layers from the particulate material and compressing the body, wherein the body (13) is formed from a plurality of layers (15, 16, 17) or regions of particulate material, one or more of the layers or regions having different properties to the other one or more layers or regions so that the density distribution produced when the body is compressed is controllable.
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MANUFACTURE OF ARTICLES WITH CONTROLLED DENSITY DISTRIBUTION

This invention relates to the manufacture of articles and in particular, but not exclusively to the manufacture of metallic articles such as uniaxially die pressed components.

In general articles are commonly formed from particulate material by applying sufficient pressure to the powders to provide a compacted material of sufficient integrity to enable it to be fired so as to provide a component of the required properties. When a particulate material is compacted, frictional affects result in a load distribution in the body. As a consequence, the compacted body has a non-uniform pressed density. This non-uniform pressed density of the compact has a significant detrimental effect on the properties of the final component. This is the case for both ceramic articles and metallic articles, although it leads to different, but significant, problems in final components because of the different properties of the two material classes.

Generally, ceramic green powder compacts have a density in the range of 50 - 60% of the theoretical density and when fired large shrinkage usually occurs. As a result of the density distribution in the green compact, differential shrinkage occurs leading to shape distortion on firing, often necessitating expensive grinding processes, to regain the desired shape.

Powdered metals unlike ceramics have the ability to deform plastically significantly. Consequently green powder metal compacts may have densities of up to 90% of the theoretical density. Nevertheless frictional effects still lead to uneven density distribution. However, due to the high density of the metal powder compact, the shrinkage resulting from firing is not significant. Nevertheless, the uneven density distribution remains in the fired compact and thus leads to heterogeneities and inherent weaknesses.
An object of the present invention is to provide a method of controlling the density distribution in an article manufactured from a powder, such as metal or plastics powders.

According to the present invention there is provided a method of manufacturing an article from particulate material, comprising the steps of:-

forming a body from particulate material and compressing the body,

characterised in that the body is formed from a plurality of layers or regions of particulate material, one or more of the layers or regions having different properties to the other one or more layers or regions whereby the density distribution produced when the body is compressed is controllable.

It is particularly preferred that the article is not a ceramic article.

Preferably it is a physical property and / or bulk density which is used to control the density distribution.

The body is preferably formed from discrete layers. Preferably three discrete layers are used, most preferably to alter the density distribution. When three discrete layers are used, two layers may be regarded as outer or end layers and the third may be regarded as an inner or middle layer. The two outer / end layers may be of the same physical character and/or density whilst the inner / middle layer may be of a physical differing nature and / or density.

The character of the layers may differ in physical nature in their morphology and / or the size of the particles and / or any other suitable physical characteristic. For instance mixed size particles can give a higher density than a single size of particle; spherical particles can pack more densely then
irregular particles; and work hardened particles may achieve a lower compacted density for a given load than non-work hardened particles.

The different layers or regions of materials may be formed from virgin powder (that is non-precompacted) or compacted, crushed, resieved powder (that is precompacted and repowdered). Preferably virgin powder is sandwiched between layers of precompacted powder. In this way we may alter the density distribution obtained on compression in comparison to the prior art. Careful choice of parameters provides control of the density distribution.

When the precompacted powder is compacted under the same conditions as the virgin powder a lower density is achieved than is achieved with virgin powder. This may be caused by the fact that the powder undergoes plastic deformation and work hardening when previously compacted from virgin powder and thus is less compressible when being recompressed.

The same effect on the control of the density distribution may be achieved by the use of the other physically different properties discussed above. This uses the same principle as with the above-mentioned virgin and precompacted powder to control eventual density.

By employing layers or regions of particulate material having different physical characteristics and densities prior to compression in accordance with the present invention, the load distribution occurring on compaction due to frictional effects is compensated for and a compact having a more uniform density distribution can be obtained. This in turn provides, upon sintering, a more uniform final component.

In the method according to the present invention the body of particulate material may be formed by introducing powder or particles of iron into a mould or die cavity having a
substantially right circular cylindrical shape.

The said body may prior to compaction comprise discrete layers having different densities. Such layers may have interfaces which are substantially planar, e.g. in a plane orthogonal to the axis of the body. Alternatively, the layer interfaces may be non-planar e.g. convex or concave to provide a suitable density profile.

Alternatively, the density change in the said body prior to compaction may take place gradually, e.g. linearly with distance, over a portion of the length of the body.

The said body prior to compaction desirably includes regions at the ends of the body which when compressed under a given load will achieve a lower density than that in the interior region i.e. near the middle of the body.

The different regions of powders of differing properties in the said body may be achieved by the introduction of particulate material into a die or mould cavity from a plurality of sources containing different particulate materials providing different bulk densities, the release of material from the plurality of sources being controlled so as to give the required density profile in the body.

The different particulate materials may comprise for example materials of the same composition but which have been treated differently or which have different particle morphology.

The different particle types may comprise, for example, (a) particles produced from a single powder but pre-compacted using different pressures, or (b) particles of the same powder which have on the one hand been milled and on the other hand have not been milled or (c) particles which on the one hand have plate-like morphology and on the other hand, have a sphere-like morphology or (d) combinations of these different types.
Where granulation is employed to produce different density granules various known granulation methods may be used for the production of granules one or more of the different density types. Roll compaction may be employed.

The particles may be coated with a small quantity, of a solid lubricant such as zinc stearate (a standard die lubricant used in the field) which has been employed in a pre-treatment process or is used in the compaction step, to treat the powders from which the body is formed (directly or indirectly as pre-compacted particles).

The pre-compaction and sintering steps in the method according to the present invention may be carried out in a manner as employed in the prior art.

Although the present invention as exemplified below uses the example of right cylindrical metallic components to illustrate the technique any particular applications may also be produced in accordance with the method of the invention.

The shape of the pellets produced by the present invention may be of any component shape.

The technique can be applied with any compaction process, not only the uniaxial die-pressing technique used as an illustration here.

The present invention will now be described by way of example only with reference to and as illustrated in the accompanying drawings, in which:-

Figure 1 is a diagrammatic representation of the density distribution of a green uniaxially double ended compressed right cylindrical pellet of metallic particulate material produced according to a prior art technique; and
Figure 2 shows a pellet according to one embodiment of the method of the invention.

Referring to Figure 1, the density and distribution of a green uniaxially double ended compressed right cylindrical pellet of a metallic particulate material is shown. The areas labelled HD are high density areas, whilst those labelled LD are low density areas. Due to the variation and density known pellets suffer from heterogeneities and inherent weaknesses.

As shown in Figure 2 a right circular cylindrical pellet body shape 13 is formed by filling the die in three parts 15, 16 and 17 prior to the pellet being compressed. For example part 15 can be one of previously compacted and repowdered material, part 16 of virgin material and part 17 again of previously compacted and repowdered material.

When an uniaxially, double ended die is compressed in the normal way the density variation introduced by the various frictional effects is counter balanced due to the regions of powders of different properties to give a product which is even in density.

The pellet can then be sintered in a conventional way.

The following illustrative examples demonstrate the benefit of the invention over the prior art in an example in which a right cylindrical component is uniaxially double ended die pressed. In the case of a right cylindrical component three layers can be used to alter the density distribution.

Two sponge iron powders of differing particle size grades MH 300.25 and HC 100.25, manufactured by Hoganas AB of Sweden were used.

The powder preparation details common to each experiment are described below:-
0.5 wt % zinc stearate was mixed with the powder samples taken from either of the samples mentioned above by placing the components in a glass jar on rollers for five minutes.

The zinc stearate acts as a lubricant during die compaction and is a standard lubricant common to the field.

Material was placed in a die and then compacted with the same load from either side, uniaxially, i.e. double ended compaction. The die used was a tungsten carbide lined steel die of diameter 9.833 mm. The pressure of compaction was 400 MPa with a dwell of 10 seconds at pressure. After ejection from the die the resultant green pellets were fractured in three approximately equal parts, using a fine hack saw. The density of each section was then measured using a standard mercury immersion technique to give the density distribution of each pellet.

Example 1 - Prior Art

The MH300.25 powder was prepared as described above and then 8g was placed in die and compressed at a pressure of 400 MPa. As described above the resulting pellet was then divided into three approximately even parts, the first and third sections corresponding to the end thirds and a second section corresponding to the middle section. The density of each section of the cylindrical pellet was measured. The density distribution was as follows:

First section: - 6.11 g cm\(^{-3}\)
Second section: - 6.06 g cm\(^{-3}\)
Third section: - 6.18 g cm\(^{-3}\)

The density distribution obtained in the prior art example is as illustrated in the schematic diagram shown in Figure 1, HD representing high density and LD representing low density. The middle section of the pellet being of lower
density than the end sections. This gives rise to the problems addressed by the present invention as claimed, and discussed herein above.

Example 2 - according to the invention

A pellet was prepared as described above. The pellet was then crushed between steel platens and then passed through a 1.18mm sieve to produce repowdered precompacted iron powder.

Following this a pellet was produced using two layers of the repowdered precompacted iron and a layer of virgin MH300.25 iron powder. The pellet was produced by placing a 2.66g layer of repowdered precompacted iron powder into the die, followed by a 2.66g layer of virgin iron powder and then a further 2.66g layer of the repowdered precompacted iron powder. The layers were then compacted under pressure at 400 MPa.

The density distribution was as follows:

First section: - 5.97 g cm\(^{-3}\)
Second section: - 6.08 g cm\(^{-3}\)
Third section: - 5.95 g cm\(^{-3}\)

The density distribution has thus reversed from that of the prior art. The second section now having the higher density than the first and third sections.

Example 3

The powders were prepared in the same manner as in Example 2 except that in this example, the die was first filled with virgin MH300.25 iron powder, then repowdered precompacted iron powder and then a third layer of virgin MH300.25 iron powder. After compaction at 400 MPa the following density distribution was achieved: -
First section: - 6.00 g cm\(^{-3}\)
Second section: - 5.89 g cm\(^{-3}\)
Third section: - 6.17 g cm\(^{-3}\)

This produced a density in the second section much lower than produced by prior art techniques.

Example 4

The same procedure was used as in Example 2, except the repowdered iron powder was prepared by prior compaction at 600 MPa and not 400 MPa as described above. This resulted in the following density distribution:

First section: - 6.01 g cm\(^{-3}\)
Second section: - 6.02 g cm\(^{-3}\)
Third section: - 6.00 g cm\(^{-3}\)

As in Example 2, the density distribution has been altered from that of the prior art and is now ostensibly uniform throughout the pellet.

Example 5

Again, the same methods were used as in Example 3, except that the repowdered MH300.25 iron powder was made by precompacting at a pressure of 600 MPa and not 400 MPa. The following density distribution was obtained:

First section: - 6.09 g cm\(^{-3}\)
Second section: - 5.88 g cm\(^{-3}\)
Third section: - 6.08 g cm\(^{-3}\)

As in Example 3 the second section has a much lower density than the first and third sections and is much lower than expected in the light of the prior art.
Example 6

The same procedure was used as in Example 1, except HC100.25 was compacted in place of MH300.25. The following density distribution resulted:

First section:  6.33 g cm$^{-3}$
Second section:  6.17 g cm$^{-3}$
Third section:  6.38 g cm$^{-3}$

This again shows a lower density in the second section as compared with the first and third sections as expected from the prior art.

Example 7

The die was filled in layers, as described above. A 2.66g layer of MH300.25 was placed in the die, followed by a 2.66g layer of HC100.25 and finally 2.66g of MH300.25. The layers were compacted under pressure at 400 MPa. The following density distribution resulted:

First section:  6.06 g cm$^{-3}$
Second section:  6.26 g cm$^{-3}$
Third section:  6.08 g cm$^{-3}$

The density of the pellet second section again increased, to the extent that it was significantly higher than the pellet first and third sections.

Example 8

The same procedure was used as Example 7 except HC100.25 was used at the pellet ends sections and MH300.25 in the pellet second section. The following density distribution was obtained:
First section:  6.25 g cm$^{-3}$
Second section:  5.93 g cm$^{-3}$
Third section:  6.30 g cm$^{-3}$

The second section had a much lower density in comparison to the first and third sections.

The results of the Examples 1 to 8 are summarised in Table 1 given below. It illustrates how the density distribution in green compacts can be readily controlled using the method according to the invention and tailored to fit the requirements of the green and final fired component. Knowledge of the density distribution in a compact of any given shape prepared by any compaction process can be used with this technique to control the green component density distribution as desired.
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Table 1. Summary of experimental results
CLAIMS:

1. A method of manufacturing an article from particulate material, comprising the steps of:
   forming a body from particulate material and compressing the body,
   characterised in that the body is formed from a plurality of layers or regions of particulate material, one or more of the layers or regions having different properties of the one or more layers or regions, whereby the density distribution produced when the body is compressed is controllable.

2. A method according to claim 1, wherein the bulk density of the layers or regions is used to control the density distribution of the body.

3. A method according to claim 1 or claim 2, wherein the body is formed from discrete layers.

4. A method according to claim 3, wherein the body comprises three discrete layers.

5. A method according to claim 5, wherein the three discrete layers comprise two outer layers and an inner layer, and the two outer layers are of the same physical character, whereas the inner layer has a different physical character.

6. A method according to any one of claims 3 to 5, wherein the layers have interfaces which are substantially planar, for example, in a plane orthogonal to the axis of the body.

7. A method according to any one of claims 3 to 5, wherein the layers have interfaces which are non-planar, for example, convex or concave.

8. A method according to any one of claims 3 to 7,
wherein one layer comprises virgin powder on which one layer is sandwiched between layers of precompacted powder.

9. A method according to any one of the preceding claims, wherein the body of particulate material is formed by introducing powder or particles of iron into a mould or die cavity having a substantially right circular cylindrical shape.

10. A method according to claim 1 or claim 2, wherein the density change in the body prior to compaction takes place gradually over at least a portion of the length of the body.

11. A method according to any one of the preceding claims, wherein the body, prior to compaction, comprises regions at the ends of the body which regions when compressed under a given load will achieve a lower density than that in the interior region.

12. A method according to any one of the preceding claims, wherein different properties of the different regions or layers of the body are achieved by the introduction of particulate material into a die or mould cavity from a plurality of sources containing different particulate materials providing different properties.
PRIOR ART

FIG. 1

FIG. 2

SUBSTITUTE SHEET (RULE 26)
**INTERNATIONAL SEARCH REPORT**

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC 6 B22F3/11

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 6 B22F

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

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

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<td>EP 0 446 664 A (ASEA BROWN BOVERI) 18 September 1991 see column 9, line 10 - line 50; claim 7</td>
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<td>US 5 279 786 A (CHO SEONG S) 18 January 1994 see claim 1</td>
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<td>GB 1 148 558 A (MAX KOHLER) 16 April 1969 see claims 1,3,5</td>
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<td>WO 95 27556 A (INST GAS TECHNOLOGY) 19 October 1995 see page 10, line 6 - line 25; claims 12,14</td>
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Further documents are listed in the continuation of box C.

**Date of the actual completion of the international search**

5 September 1997

**Date of mailing of the international search report**

15.09.1997

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Te. 31 651 epo nl,
Fax (+ 31-70) 340-3016

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Schruers, H

Form PCT/ISA/210 (second sheet) (July 1992)

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