

Aug. 10, 1954

P. E. CAVANAGH

2,686,118

METHOD OF MAKING METAL PRODUCTS DIRECTLY FROM ORES

Filed Dec. 23, 1952

3 Sheets-Sheet 1

Fig. 1

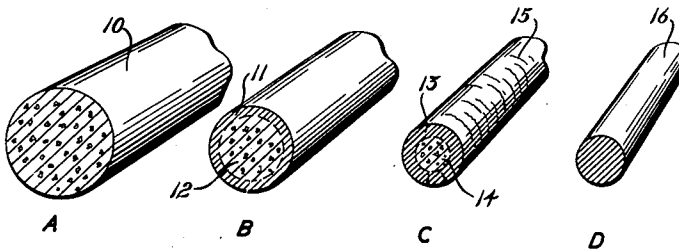
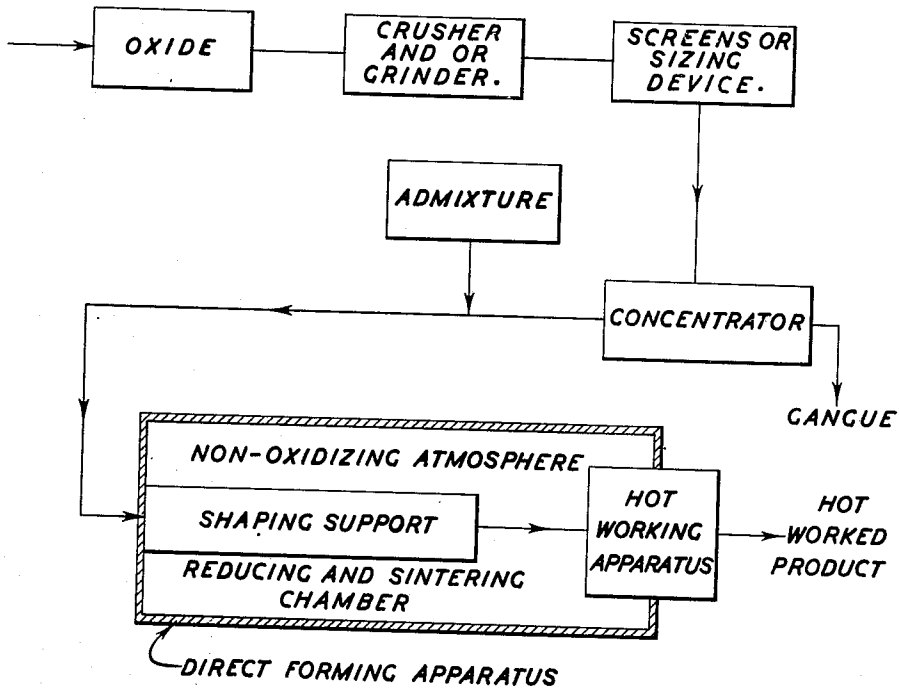


Fig. 2

Inventor  
PATRICK E. CAVANAGH.  
by: *Fetherstonhaugh & Co.*  
Attys.

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3 Sheets-Sheet 2

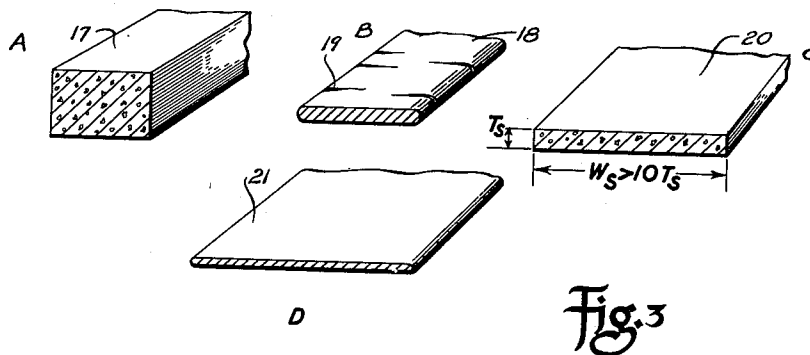


Fig. 3

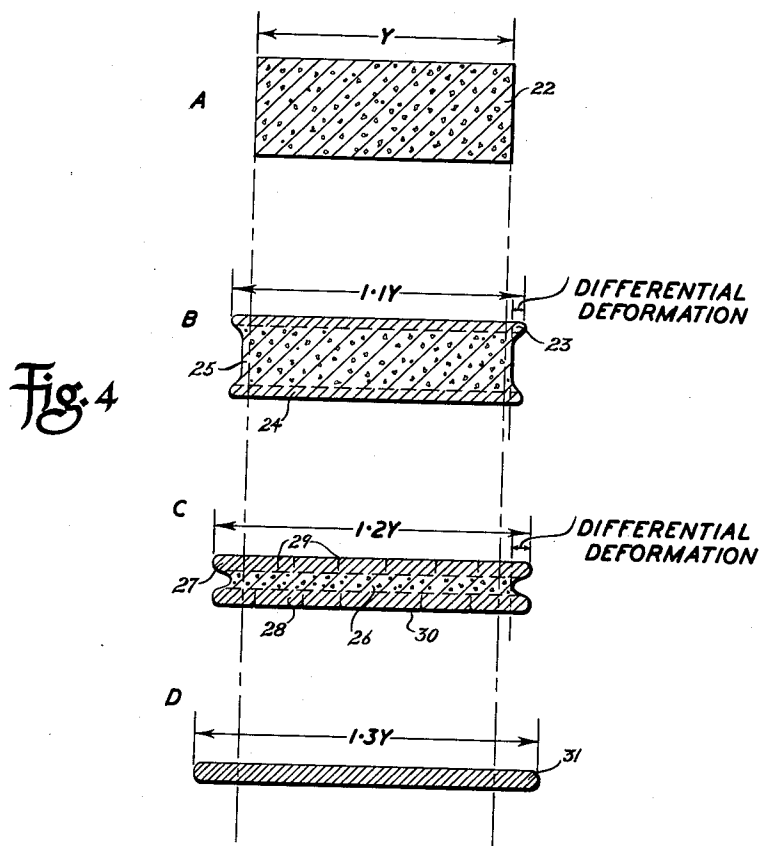


Fig. 4

Inventor  
PATRICK E. CAVANAGH.  
by: *Fetherstonhaugh & Co.*  
Attys.

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3 Sheets-Sheet 3

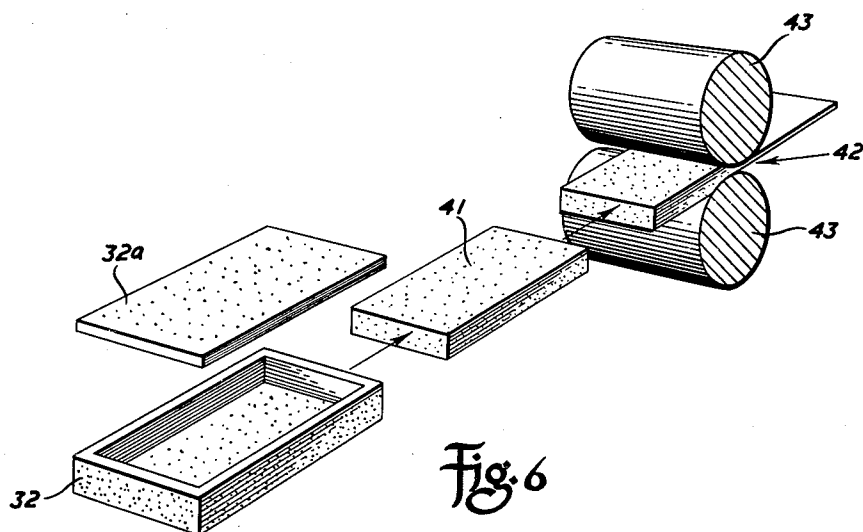


Fig. 6

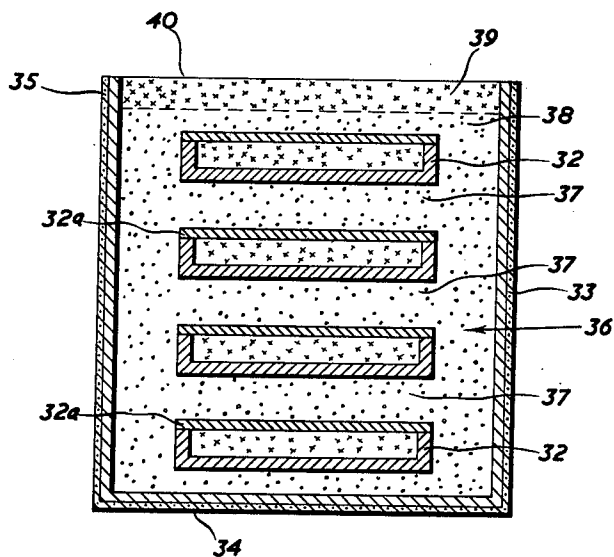


Fig. 5

Inventor  
PATRICK E. CAVANAGH.  
by: *Petherston Haugh & Co.*  
Attys.

## UNITED STATES PATENT OFFICE

2,686,118

METHOD OF MAKING METAL PRODUCTS  
DIRECTLY FROM ORES

Patrick Edgar Cavanagh, Oakville, Ontario, Canada, assignor to Ontario Research Foundation, Toronto, Ontario, Canada, a corporation of Ontario

Application December 23, 1952, Serial No. 327,575

27 Claims. (Cl. 75-211)

1

This invention relates to the manufacture of metal products and particularly to methods adaptable to large scale operations for forming wrought ferrous metal products; for example, sheets, plates, strips, bars, rods and the like, directly from compositions that comprise previously unreduced oxygen bearing metal compounds or from bodies below theoretical full density of the metal thereof, hereinafter defined as sub-density metal bodies. In a more general sense the invention relates to the formation of a metal article of finite shape and of less than solid metal density and also to the working of subdensity metal bodies which may be so formed.

This application is a continuation-in-part of my co-pending applications: Serial No. 231,074, filed June 12, 1951 and Serial No. 232,921, filed June 22, 1951.

By a subdensity metal body, I mean a metal body having a density which is substantially less than the density of the solid material thereof, by reason of voids in the body. It will be understood that such metal body may be formed of a single metallic element or of an alloy or mixture of a number of metals with or without other materials. In an important specific sense, the present invention is concerned with ferrous metal bodies; wherein the predominant component is iron, examples being compositions corresponding to various steels, other ferrous alloys and the like.

While the invention relates to the forming of wrought ferrous metal products from subdensity bodies, it is preferred to practice the invention upon that class of subdensity body which is formed from controlled density metals including techniques associated with manufacture of the latter as set forth in the said co-pending applications. A controlled density metal body may be described as a body produced by complete or partial reduction of oxygen bearing material at temperatures below the melting point of the body, in suitable apparatus whereby the body is produced and proportioned to a predetermined shape of predetermined dimensions during the reducing process; said oxygen bearing material comprising a compound or compounds of one or more metals which, before reduction, may or may not have been mixed with other metallic and/or non-metallic materials; said body having predetermined surface conditions and a predetermined density between 5 per cent and 100 per cent of the theoretical full density of the solid materials of said body.

Prior proposals for forming ferrous products directly from oxides or from subdensity iron particles have involved a complex series of operations and have often been directed to the formation, at the outset, of a large ingot or billet of high density. The ingot or billet is sometimes

2

required to be so compacted in formation as to reach the equivalent of full, solid metal density, or is produced in some other special form of lamination or the like, as heretofore believed necessary for passage of the billet through the rolls of a conventional rolling mill.

In contrast to prior methods, the present invention differs largely in concept in having for a main object, the provision of a direct method adaptable to large scale manufacture for forming hot worked metal products of maximum density, e. g., substantially solid metal density, while eliminating the necessity of forming an ingot of high density or of performing other costly intermediate steps, and wherein the product to be produced may correspond, for example, to the product ordinarily obtained from the final rolls of a rolling mill adapted to work upon an ingot.

Another object of the invention is to provide a method of making sheet steel and similar products of a chosen predetermined thickness directly from a subdensity metal shape proportioned to be reduced to said predetermined thickness when worked by passage through rolls to the point of maximum density. By the term "maximum density" I mean the maximum density attainable in practice by the body.

The first problem encountered is that of providing a subdensity metal shape of sufficient structural unity to endure passage through rolls or other hot working apparatus without disintegration. Three general methods of attempting to provide such a shape are known.

The first class of known method makes use of a pressure technique and a binder for shaping the ore before reduction, the aim being to form a high density sintered product. Three serious disadvantages arise from this approach, namely: it is difficult for reducing gases to permeate through a highly compacted mass and accordingly reduction times tend to be very long; very few iron ores are capable of being reduced after compaction without failure of the compacted shape due to swelling of the ore or undue shrinkage thereof during reduction; the size and proportions of any shape which can be formed by any technique dependent upon relatively large pressures for compaction of the oxide mass are severely limited by both physical and practical conditions as is well known.

It is another object of the invention to provide a non-friable subdensity metal shape of structural unity adapted for hot working operations by aiming preferably at the formation of such a shape at low density for the purpose of achieving rapid reduction.

In a second class of method for forming a shape of structural unity, loose ore is reduced as in a common sponge iron class of process to

provide a mass of metal particles defining no definite shape and of the usual relatively unbonded friable nature and which is highly compacted while hot to form a shape of high density which is subsequently fed to a set of rolls.

A third class of method for forming a shape of structural unity is exemplified by any of the well known powdered metallurgy techniques wherein powdered metal is compacted while hot or before heating and then sintered to obtain a high density spongy metal article or solid metal article.

Attempts to form large ingots by powder metallurgy techniques have not been seriously contemplated for the reason that shape or size factors interfere, limiting practice to the formation of small articles even when using very large compacting pressures.

It is another object of the invention to provide a shaped subdensity metal article formed by reducing ore in a reducing zone without melting while supported to the shape desired and cohering the reduced ore particles to form a unitary mass of subdensity metal by heating the reduced ore particles to the cohering point in a non-oxidizing atmosphere.

I have discovered that a subdensity mass of metal may be successfully reduced to maximum or full density of the metal itself while substantially unconfined by an enclosing die, mold, or the like, by following techniques in accordance with the invention. In the hot working of subdensity ferrous metals, one criterion according to the invention requires that the hot working be limited to that sufficient to develop less than a permissible amount of differential deformation. By the phrase "differential deformation," I mean the difference in elongation in any direction of the core portion of a subdensity metal body compared to the elongation of the surface portions thereof during working. A second criterion requires that if a product is to be wrought so as to have a substantially uniform density throughout, the working, e. g., hot working, should be accomplished by carrying the deformation in one operation (by the application of a continuous force, as in a single rolling pass) from a point where the body has zero differential deformation or less than the differential deformation permissible under the first criterion above, to a point where the density of the body has sufficiently approached maximum density as again to reach a condition of less than a permissible differential deformation. A third criterion requires that the subdensity metal article be proportioned having regard to the nature of the product to be made.

A still further object of the invention is to provide a process for forming hot worked ferrous products wherein the grain size of the resulting ferrous product is substantially determined by the grain size of the particles of iron oxide from which the ferrous product is formed, whereby properties normally attributed only to extensively worked ferrous products can be attained with a small amount of working.

A still further object of the invention is to provide a direct method for obtaining a sheet metal product or the like from ores or oxides comprising a process of interrelated steps in each of which the allowable limits of variations are fixed by the desired characteristics of the product. For example, the desired thickness of the end product will determine the roll setting and the dimensions of the controlled density steel slab for a given density of the slab. The desired

grain size of the finished sheet will determine the average particle size of the ore.

Other objects of the invention will be appreciated by a study of the following specification, taken in conjunction with the accompanying drawings.

In the drawings:

Figure 1 is a flow sheet layout of a preferred process according to the invention;

Figures 2A to 2D illustrate the manner of hot working a subdensity ferrous article according to the invention;

Figures 3A to 3D illustrate the manner of hot working a modified form of slab according to the invention to obtain a clean edge condition on a strip, bar or sheet product;

Figures 4A to 4B illustrate methods of hot working a subdensity ferrous article according to the invention;

Figure 5 is a sectional view of apparatus for forming a shaped cohered subdensity article; and

Figure 6 is a diagrammatic perspective of the formation of a subdensity metal body and a manner of working same.

Referring to the drawings, the concept of the general process will be appreciated from an examination of Figure 1, wherein it will be apparent that an iron oxide is crushed and/or ground, as may be required, and sized by suitable screens or other sizing device to obtain a preferred particle size distribution and bulk density in the oxide to be used in the present process in accordance with the requirements of shrinking and swelling characteristics of the oxide during reduction, grain size in the final product, rate of reduction or permeability of the oxide mass to the passage of reducing gases, and the desired density and strength of the subdensity metal shaped article produced for the hot working techniques disclosed herein. Silica and other gangue constituents may be removed from the chosen form of iron oxide material preferably by dry concentration, for example, by a suitable concentrator such as a magnetic concentrator of the class disclosed in Swedish Patent No. 120,710, granted November 27, 1947, to E. H. H. Eketorp et al.

Following concentration, the sized and concentrated oxide may have admixed therewith, finely divided alloying constituents or foreign bodies in accordance with the requirements of the product to be produced.

The sized and concentrated oxide, with or without admixture, is loaded into a shaping support in the direct forming apparatus, which support is of a nature defining the shape of the subdensity article produced therein during the reduction process. The present process does not rely at this stage upon the application of positive pressure techniques such as extrusion or compaction of the ore by large pressures. The oxide may be freely loaded into the shaping support, this particular technique permitting the present process to be operative with an extremely wide range of ferrous oxide materials which otherwise would react unfavourably if unduly compacted, due to shrinkage or swelling characteristics.

While in the shaping support, the oxide is heated and subjected to a reducing gas in the reducing chamber as indicated and the reduced oxide particles are heated to a sufficient temperature to form a cohered subdensity article of sufficient structural unity permitting removal from the shaping support and of sufficient

strength to be handled as a non-friable cohered body of predetermined dimensional characteristics adapted to be hot worked in the hot working apparatus. It is at present preferred that the strong coherence of the shaped body be achieved solely by heating it to unite its metallic composition, e. g., at least to a temperature for sintering its particles or portions strongly together. It will be understood that in practice, the reducing and cohering operations may be at least in part simultaneously effected; for example, the last stages of a desired extent of reduction may be performed while heating is being continued at a temperature for effectuating or completing the described coherence.

The hot working techniques of the invention may be discussed with reference to Figures 2 to 4. The subdensity ferrous metal shape 10 at Figure 2A may be hot worked to compact the surfaces thereof to maximum density as indicated by the maximum density outer layer 11 at B surrounding the lower density core 12. I have found that it is permissible to so compact the outer regions of such body within the limits of an elongation giving rise to stresses between the outer dense skin and the inner low density core of a limited value. If the structure shown at B in Figure 2 is reduced in diameter by further hot working to a point where the skin 13 is of substantial thickness as compared with the diameter of the core 14 as at C, then the elongation of the skin 13, if in excess of about 10 per cent, may give rise to undue stresses, causing failure imperfections 15 in the skin. It is for this reason that the ordinary swaging machine or rolling mill practice is not operative to form a maximum density rod from a low density subdensity structure unless methods are modified to follow the methods of the present invention.

I have found that a section of entire maximum density as at 16 in Figure 2D having substantially no voids, as would be the case of an ordinary steel rod or the like, may be formed by working the subdensity article shown at 2A to maximum density under the action of a continuously applied force, that is, in one operation through a swaging device.

In forming strip or sheet of maximum density, the subdensity metal slab must be of predetermined shape and dimensions to attain the desired results. For example, a subdensity ferrous article 17 shown in Figure 3A may be reduced in one hot pass through the rolls to form the maximum density product 18 of Figure 3B. If the article 17 is of insufficient "aspect ratio," i. e., the ratio of width to thickness is too small, the product 18 may have edge faults 19. In many cases, small edge faults may be eliminated by edge trimming of the resulting sheet. In general, however, edge faults arising from the proportioning of the subdensity slab may be avoided as indicated in Figure 3C by sizing the slab such as slab 20 to a width greater than ten times the thickness. As indicated in Figure 3D, a maximum density product 21 of more regular edge form will result. A subdensity metal slab aspect ratio of greater than fifteen is preferred as giving satisfactory results in most instances regardless of the particular density of the slab which is to be worked to maximum density.

Edge fault problems derived from slab proportions will not ordinarily arise in the practice of the invention to form a wide sheet of desired thickness at the point of maximum density of the slab. Accordingly, the proportions of the slab

will primarily be determined by other considerations. I have ascertained that slab proportions for purposes of the invention are derived from a consideration of relative volumes according to the relation

$$\frac{V_s}{V_{\min}} = \frac{D_{\max}}{D_s}$$

where

- 10  $V_s$  is the volume of the subdensity slab;  
 $D_s$  is the density of the subdensity slab;  
 $V_{\min}$  is the volume of the maximum density product, as it comes from the rolls;  
 $D_{\max}$  is the density of the maximum density product.

Assume a desired sheet volume of 5 cubic units of 10 units in length, 5 units in width and  $\frac{1}{10}$  of a unit in thickness and of a density of say 8. Assume a subdensity slab of density 2 to be reduced in one hot pass of the rolls to  $\frac{1}{10}$  of a unit in thickness. The volume of the slab should be

$$V_s = \frac{D_{\max} \cdot V_{\min}}{D_s} = 20 \text{ cubic units}$$

- 25 The elongation in the direction of rolling will be say 30 per cent. The elongation in the lateral direction will be say 10 per cent.

The length of the slab may then be

$$L_s = \frac{100}{130} \times 10 = 7.7 \text{ units}$$

The width of the slab may be

$$W_s = \frac{100}{110} \times 5 = 4.54 \text{ units}$$

The thickness of the slab may be

$$T_s = \frac{V_s}{W_s \times L_s} = \frac{20}{7.7 \times 4.54} = 0.57 \text{ units}$$

Slab dimensions 7.7 x 4.54 x 0.57.

Although the aspect ratio of such a slab is less than the preferred minimum, there is a possibility of obtaining satisfactory edge conditions assuming regular slab edges, good rolling practice and the usual amount of edge finishing or trimming in the final product.

While proportioning techniques set forth herein apply largely to methods of forming a product of maximum density material in its entirety, the same techniques generally apply to portions of a slab which may be reduced to maximum density while the remainder is substantially unworked. For the purpose of calculation of general slab proportions, the unworked portion may be assumed to retain its original density.

Special techniques also arise in the control over other faults which may occur in the formation of strip, bar or sheet. For example, in Figures 4A to 4D, the longitudinal section of ferrous metal bodies is illustrated. In Figure 4A, a subdensity ferrous metal article 22 is shown of a specific length  $y$ . As in the technique discussed with reference to Figure 2, the subdensity article 22 may be rolled or otherwise compacted to form skins of maximum density 23 and 24 on either side of a low density core 25 such as by a single pass through the rolls while the article is hot, wherein a differential deformation in the case of ferrous products will be of the order of about 5 per cent. As indicated, the skin portions may undergo an elongation of about 10 per cent or less during a rolling operation; however, as shown in Figure 4C, if the structure of 4B is subjected to further passes through the rolls, undue differential deformation may occur and the skin portions may elongate to an extent setting up

stresses during rolling in excess of the strength of the skins. Thus one may obtain a core 26 of low density having thereover the skins 27 and 28 having faults 29 and 30. After one pass through the rolls to form a product such as in Figure 4B, one risks the condition of Figure 4C upon attempting to roll further to maximum density.

It will thus be evident that elongations between about 10 per cent and about 25 per cent represent a substantially difficult range to be avoided in the working of subdensity ferrous materials in particular. While I believe that differential deformations should not exceed about 5 per cent for many ferrous metal products, this will not be found inconsistent by skilled persons employing these general limits in conjunction with factors of safety assuring desirable results in any circumstance. The degree of perfection required will be dictated by specifications for the product.

If a product of maximum density throughout is required, I subject a subdensity metal article such as the article 22 of Figure 4A, to a continuously applied force such as by a single pass through the rolls, while hot, to reduce it to maximum density, at which an elongation of about 30% or more will be experienced, as indicated in the case of the product 31 of Figure 4D. As before mentioned, in a more general sense an important criterion of working requires that if a product is to be wrought so as to have a substantially uniform density throughout, the working, e. g., hot working, should be accomplished by carrying the deformation in one operation (by the application of a continuous force, as in a single rolling pass) from a point where the body has zero differential deformation or less than the differential deformation permissible under the first criterion above, to a point where the density of the body has sufficiently approached maximum density as again to reach a condition of less than a permissible differential deformation. It will therefore be understood that the single application of continuous force is exerted to carry the body to a relatively high preferably uniform density and to not more than a predetermined differential deformation. In the formation of a sheet, strip or bar, this technique must be taken into account, along with the technique disclosed in the discussion of Figure 3.

I have successfully formed products according to the invention by employing a reheating technique wherein the subdensity ferrous article is allowed to cool and is then reheated in a reducing or non-oxidizing atmosphere before working.

Thus Tables I to IV list information pertinent to examples of practice of the invention applied to three different iron oxide materials.

**TABLE I**  
**Mill scale**

|                                                    |                                              |
|----------------------------------------------------|----------------------------------------------|
| Total Fe.                                          | 73.6%.                                       |
| Silica                                             | 0.48%.                                       |
| Bulk density                                       | 200 lbs./ft. <sup>3</sup><br>or 3.2 gms./cc. |
| Sieve analysis:                                    |                                              |
| +60                                                | 0.0 gms.                                     |
| -60 +80                                            | 1.5 gms.                                     |
| -80 +100                                           | 5.0 gms.                                     |
| -100 +150                                          | 14.0 gms.                                    |
| -150 +200                                          | 18.0 gms.                                    |
| -200 +270                                          | 38.0 gms.                                    |
| -270 +325                                          | 17.0 gms.                                    |
| -325                                               | 5.0 gms.                                     |
|                                                    | 98.5 gms.                                    |
| Reduction temperature                              | 1980° F.                                     |
| Holding time (reducing and cohering time in hours) | 20.                                          |
| Slab density                                       | 3.0 gms./cc.                                 |
| Slab thickness                                     | 0.516 inch.                                  |
| Single hot pass reduction                          | 75.7 percent.                                |
| Sheet thickness                                    | 0.125 inch.                                  |
| Sheet density                                      | 7.5 gms./cc.<br>(computed).                  |
| Cold rolling reduction                             | 70 percent.                                  |
| Cold rolled sheet thickness                        | 0.038 inch.                                  |
| Annealing temperature                              | 1400° F.                                     |
| Annealing time                                     | 30 min.                                      |

**TABLE II**  
**"Old Bed" concentrate**

|                                                    |                             |
|----------------------------------------------------|-----------------------------|
| Total Fe.                                          | 70.2%.                      |
| Bulk density                                       | 3.5 gms./cc.                |
| Sieve analysis:                                    |                             |
| +60                                                | 1.0%                        |
| -60 +100                                           | 22.7%                       |
| -100 +150                                          | 20.3%                       |
| -150 +200                                          | 15.6%                       |
| -200 +325                                          | 12.7%                       |
| -325                                               | 27.7%                       |
|                                                    | 100.0%.                     |
| Reduction temperature                              | 1980° F.                    |
| Holding time (reducing and cohering time in hours) | 12½.                        |
| Slab density                                       | 2.1 gms./cc.                |
| Slab thickness                                     | 0.410 inch.                 |
| Single hot pass reduction                          | 79%.                        |
| Sheet thickness                                    | 0.085 inch.                 |
| Sheet density                                      | 7.7 gms./cc.<br>(computed). |
| Cold rolling reduction                             | 40%.                        |
| Cold rolled sheet thickness                        | 0.046 inch.                 |
| Annealing temperature                              | 1150° F.                    |
| Annealing time                                     | 30 min.                     |

**TABLE III**  
**"Mag Iron" concentrate**

|                                                    |                                              |
|----------------------------------------------------|----------------------------------------------|
| Total Fe.                                          | 71.4%.                                       |
| Silica                                             | 0.44%.                                       |
| Bulk density                                       | 194 lbs./ft. <sup>3</sup><br>or 3.1 gms./cc. |
| Sieve analysis:                                    |                                              |
| +60                                                | 0.0 gms.                                     |
| -60 +80                                            | 4.0 gms.                                     |
| -80 +100                                           | 9.0 gms.                                     |
| -100 +150                                          | 23.5 gms.                                    |
| -150 +200                                          | 21.5 gms.                                    |
| -200 +270                                          | 20.5 gms.                                    |
| -270 +325                                          | 7.0 gms.                                     |
| -325                                               | 14.0 gms.                                    |
|                                                    | 99.5 gms.                                    |
| Reduction temperature                              | 1980° F.                                     |
| Holding time (reducing and cohering time in hours) | 10.                                          |
| Slab density                                       | 2.1 gms./cc.                                 |
| Slab thickness                                     | 0.410 inch.                                  |
| Single hot pass reduction                          | 80.7%.                                       |
| Sheet thickness                                    | 0.079 inch.                                  |
| Sheet density                                      | 7.7 gms./cc.<br>(computed).                  |
| Cold rolling reduction                             | 46%.                                         |
| Cold rolled sheet thickness                        | 0.040 inch.                                  |
| Annealing temperature                              | 1150° F.                                     |
| Annealing time                                     | 30 min.                                      |

**TABLE IV**  
**Properties of rolled sheet (test data)**

|                         | Mill Scale                    | Old Bed                      | Mag Iron                     |
|-------------------------|-------------------------------|------------------------------|------------------------------|
| Hot Rolled:             |                               |                              |                              |
| Rockwell B Hardness     | 60.                           | 70.                          | 67.                          |
| Bend Tests              | 180° on ½"                    | 180° on ¾"                   | 210° on ¾"                   |
| Ult. Tensile            | 52,000 p. s. i.               | 54,300 p. s. i.              | 59,000 p. s. i.              |
| Elongation (tested)     | 19%                           | 12%                          | 16%                          |
| Grain Size              | 7-8.                          | 7-8.                         | 7-8.                         |
| Cold Rolled:            |                               |                              |                              |
| Rockwell B Hardness     | 103.                          | 98.                          | 99.                          |
| Bend Tests              | 45°                           | 46°                          | 50°                          |
| Ult. Tensile            | 102,000 p. s. i.              | 96,200 p. s. i.              | 99,000 p. s. i.              |
| Elongation (tested)     | 1%                            | 2%                           | 1%.                          |
| Annealed:               |                               |                              |                              |
| Rockwell B Hardness     | 60.5.                         | 55.                          | 56.                          |
| Bend Tests              | bent on self, surf. openings. | bent on self, tears through. | bent on self, tears through. |
| Ult. Tensile            | 49,000.                       | 46,300.                      | 47,000.                      |
| Erickson Ductility Test | 4.8.                          |                              |                              |

In the foregoing examples, it was proposed to form a 3 inch wide strip of hot rolled steel about 12 inches long and less than  $\frac{1}{8}$  inch thick. Accordingly, a subdensity metal slab was made from each of the three iron oxide materials noted, 3 inches wide and 12 inches long and of a low density of about 2 to 3 gms./cc. since such low density would offer a low resistance to the passage of reducing gases. Assuming a slab density of 3 and having regard to volume-density relations as set forth herein, the slab thickness required to produce a  $\frac{1}{8}$  inch sheet was about  $\frac{1}{2}$  inch, giving a very low aspect ratio for the slab of 6 to 1.

The Mill Scale selected was obtained from a continuous hot strip mill and accordingly was relatively low in residual alloys and carbon content. Upon analysis, the Mill Scale showed 73.6% iron and 0.48% silica.

Since it was desired in all of the above examples to predetermine the grain size of the resulting rolled product to a conventional magnitude, a substantially corresponding sizing of the Mill Scale was carried out by grinding in a ball mill to minus 60 mesh screen size. The ground Mill Scale was screened to provide the particle size distribution shown in Table I of relatively low bulk density consistent with the delivery of a relatively low density metal slab and accordingly conducive to rapid reduction. The bulk density of the ground and sized Mill Scale was determined by pouring it into a graduated flask vibrated for thirty seconds to constant volume after which the volume and weight of the contents were measured and the density calculated.

Permeable molds were then made by mixing 3% of a thermo-setting plastic binder with foundry sand as in well-known shell molding techniques. A shell mold was formed with this mixture over a metal pattern heated to 500° F. The finished mold was removed from the pattern and after cooling, was loaded with the prepared Mill Scale. The loosely filled mold was then vibrated to settle the loose oxide as much as possible and a shell mold material cover was placed over the upper end of the loaded mold.

A convenient way of performing the formation of the subdensity article within this mold is illustrated in Figure 5. A plurality of loaded permeable molds 32, having covers 32a were placed, one above the other, in a cylindrical container 33. A metal sagger of 15 inches inside diameter and 18 inches in height, was used. The saggars were fabricated from sheet alloy steel (15 per cent chrome, 35 per cent nickel). The bottoms 34 of the saggars were formed of cast alloy, each of the saggars being coated with a suitable high temperature enamel 35 to reduce oxidative tendencies. In loading the molds into the saggars, a reducing mixture 36 was first placed on the bottom of the saggars and then the molds 32 were placed thereabove spaced each from the next by a layer 37 of the reducing mixture with a final layer 38 of the reducing mixture to a level within 2 inches of the top of the sagger. A one inch layer 39 of loose iron ore of finely divided form was placed over the topmost layer of reducing mixture to form a seal over the open end 40 of the saggars and to prevent reoxidation of any iron produced in the sagger.

The reducing mixture 36 was thus placed entirely outside the material loaded into the molds and comprised a mixture of coke breeze and limestone wherein the coke breeze was of at least 80%

carbon, completely dry and sized to minus 8 mesh screen size; 15% by weight of minus  $\frac{1}{4}$  inch limestone was added to the coke breeze to act as a desulphurizer to take up the sulphur from the coke and prevent it from going into the iron particles as they were formed from the oxides being reduced in the sagger. The ratio of coke to oxide in the saggars was of the order of 10 of coke to 1 of oxide by weight.

The loaded saggars were heated to a reducing temperature by passing them through a tunnel kiln at sufficient speed so that the time at a temperature of 1980° F. was 20 hours. This period of time enabled the reduced oxide particles to cohere together to form a non-friable cohered mass which was allowed to cool over a period of twenty-eight hours to 200° F. The time at temperature during the reducing and cohering period was of a time period which, in conjunction with the average size of the oxide, the particle size distribution thereof, the temperature and the amount of carbon available from the reducing gas for solution in the iron particles produced, delivered a carbon content of 0.12% and a density of 3.0 gms./cc. All of these factors were adjusted to produce a controlled density steel slab, in accordance with the teachings of the disclosure of my prior applications, Serial No. 231,074, filed June 12, 1951, and Serial No. 232,921, filed June 22, 1951.

The reduction of the oxide within the molds and the saggars could have been carried out in any batch type furnace such as a heat treating furnace. Any furnace which will sufficiently heat a number of such saggars to a temperature between 1900° F. and 2200° F. at the rate of about 100° F. per hour and which will hold the saggars at this temperature for a considerable period of time, would be suitable for processing of the type indicated.

In accordance with the invention and as indicated in Figure 6, a subdensity slab 41 of controlled density, predetermined shape and dimensions, is removed from a mold 32 after formation therein by the reducing and cohering process and while heated to a working temperature, may be wrought as at 42 to a uniform density of substantially a maximum value by applying a continuous force such as by the rolls 43 set substantially to the thickness of the product desired.

In the present example, subdensity metal slabs conforming to the shape of the molds were removed from the saggars after cooling and were reheated in an atmosphere of nitrogen containing about 30% hydrogen in an electric furnace to a temperature of 2100° F. and held for ten minutes.

The rolls of a small mill were set to produce a sheet in one pass of the slab to a thickness of 0.125 inch. After passing the heating slabs through the rolls so adjusted, a reduction of 75.7% was effected in one pass delivering a density in the final product of 7.5% being substantially the point of maximum density of the material being worked upon. The value for density was calculated from the dimensions of the product.

In forming slabs from magnetite concentrates, two representative ores were selected. The concentrate designed "Old Bed" in the Tables II and IV is a New York State magnetite. The concentrate designated "Mag Iron" in Tables III and IV is a Canadian magnetite, these concentrates being well known to persons familiar with the production of iron and steel.

Procedure followed with Mag Iron and Old

Bed ores was the same as with Mill Scale. The screen size distribution and bulk density and analysis of the ores are shown in the tables. In both cases, the ore was magnetically concentrated after grinding to remove most of the gangue.

The slab sizes were the same and the coke to ore ratio the same as for Mill Scale. The holding time at temperature was 10 hours for Mag Iron and 12½ hours for the Old Bed. As a result, the density of the slabs produced was 2.1 in both cases and the carbon content was 0.27% for the Mag Iron slab and 0.08% for the Old Bed slab.

The slab thicknesses were 0.410 inch and after rolling reduction of about 80% in a single pass after reheating to 2100° F. in hydrogen plus nitrogen atmosphere, the resulting slabs had a computed density of 7.7. Primarily, due to the low aspect ratio of the slabs before rolling, edge faulting in the final products occurred.

As indicated in the foregoing tables, all of the sheets obtained by hot rolling in one pass to maximum density, were then subjected to a cold rolling reduction and annealed. Mechanical tests were carried out for each of the hot rolled, cold rolled and annealed products as outlined in Table IV. Observe that the grain size of the hot rolled products was between 7 and 8 after one hot pass. Average numbers for grain size for most hot rolled steels range between about 4 and 8, after a large number of hot passes. These numbers for grain size are in accordance with the classification of the American Society for Testing Materials, denoting the number of grains per square inch at 100 magnification. It is noteworthy that a comparable grain size to that accomplished in ordinary steels, was obtained by the present process by a control exercised on the sizing of the ore. The structural properties listed in Table IV illustrate the accomplishment of desirable structural characteristics by one hot pass of the subsdensity metal structure through the rolls to maximum density and also show satisfactory properties after cold rolling and annealing.

The subsdensity articles formed for the purposes of forming the maximum density sections, need not be of a high density themselves. This invention specifically contemplates the advantage to be gained by making these subsdensity articles of a low density (i. e., less than half the value of maximum density, in some cases as low as one-fifth; but sufficient to accomplish a cohered body) so that the oxide mass may offer relatively low resistance to the passage of reducing gases there-through, thus permitting very short reduction times. Space is provided in the voids of the mass to accept swelling and shrinking characteristics of the oxide particles during reduction. Such characteristics are discussed in some detail in a paper by the present inventor entitled "Pelletizing of Iron Bearing Fines by Extrusion" and reported in 1950 Proceedings, volume 9, page 54 of The American Institute of Mining and Metallurgical Engineers.

While the space provided by the voids generally assists in controlling shrinking and swelling characteristics, it is particularly contemplated according to the present invention, to mix shrinking and swelling ores to cancel out such characteristics to a degree permitting a closer dimensional control than has heretofore been possible with most iron ores. The problems are not critical in the preferred practice of my invention wherein the shape of the subsdensity article

is determined during the reducing process. However, where subsdensity metal shapes are formed directly from the oxide by prior methods, the proportioning of the shapes taught herein for working purposes may not be realized or such prior methods may not be operative with many ores unless a blend of a plurality of ores of different dimensional change during reduction is used in accordance with the present invention.

While certain limits of differential deformation have been discussed herein, it must be appreciated that the permissible differential of deformation between the core and skin portion depends upon the material of the subsdensity article and the hot working temperature, as well as the nature of the hot working apparatus. It is conceivable that a permissible differential deformation of 5 per cent or more may be achieved under special conditions.

On the other hand, it will be apparent that if the subsdensity product is worked to maximum density while confined to limit the elongation and hence the differential deformation of the material upon being compressed to maximum density, then the many faults described herein may not arise.

By way of recapitulation with respect to rolling and like operations, it has been indicated above to be preferable that in rolling a subsdensity slab to form a product having solid metal density the slab be hot rolled with sufficient reduction and elongation in a single pass to carry the body directly from its unworked subsdensity state to substantially maximum density. It will be understood, however, that a rolling pass which in the above general fashion effects a substantial and preferably the major part of the deformation necessary to reach maximum density may sometimes be safely accompanied (in the course of achieving maximum density) by preliminary or subsequent passes or both; e. g., hot rolling passes preferably representing only a minor part of the total density change in reaching the solid-metal (or otherwise substantially uniform) density state.

For instance, in some cases it has been found possible to carry a subsdensity slab of ferrous metal, having a density of about 2 to 3 gms./cc., to a density of about 6 gms./cc. in a single, major, hot rolling pass, without impairing the structural integrity of the body. In such operation, the working is found to be carried (in the single pass) beyond the range of elongations wherein the differential deformation is excessive, the body being thus brought to a point where the differential deformation is again safely small and where a substantial density increase has presumably occurred throughout the body. Thickness reduction to maximum density, say about 7.5 gms./cc. can then be completed with one or more further passes; e. g., hot rolling passes. It also appears possible in some cases to employ a preliminary hot pass or passes on the unworked subsdensity slab before proceeding to reduce it throughout to substantially uniform, high density as explained in the foregoing or elsewhere herein, but such preliminary operation (which will provide dense skins on a low density core) should preferably keep below the limit of permissible differential deformation (i. e. the limit explained in connection with Figure 4B) and most advantageously well below such limit.

In other words, each rolling pass or other single continuous application of deforming force on a body constituted wholly or partly of subsdensity

metal should preferably be controlled or designed to avoid leaving the body in a state illustrated in Figure 4C. Specifically, each single pass (depending on its purpose or its position in a sequence of passes) should preferably be such as either (a) to avoid carrying the first stage of thickness reduction beyond the point of permissible differential deformation (Figure 4B) or (b) to carry the thickness reduction to a much further point, apparently represented by attainment of increased density throughout the body where there is likewise no excessive differential deformation. In a more general sense, an important feature of the invention is the thus exemplified concept that in rolling a subdensity metal body, the characteristics of the rolling pass or passes should be coordinated with the shape, size and density of the body to avoid excess differential deformation, such coordination being achieved by proper control of one or more of the stated characteristics or factors, indeed in effect of all of them.

Methods of the invention are adapted to the formation of a wide variety of products different in structural character and composition from products obtainable from conventional metal forming arts. The subdensity article formed according to the methods set forth herein may be of varied composition and/or density before being wrought. The composition is determined by the materials loaded into the shaping support for reduction to form the subdensity metal body. Such materials are loaded into the shaping support in zones or layers according to the composition desired in corresponding zones or layers in the final product. The density of such layers is individually controlled generally by sizing of the material therefor. The resulting subdensity body of varied metallic composition is wrought in the manner set forth herein to provide what may be termed a sandwich structure such as a low carbon steel or other ferrous metal subdensity core having integrally joined thereto, thin or thick skins or surface portions of maximum density stainless steel or other ferrous metal composition. The resulting subdensity body may also be wrought to uniform or entire maximum density to provide, for example, a stainless steel sheet having a mild steel core inherently bonded thereto by reason of formation therewith during the reducing process. A wide variety of compositions and densities is thus afforded in metal products of the invention. In all such products, the metals are contemporaneously formed in, and/or derived from, the same reducing and working processes.

In the rolling reduction of subdensity structures, a faulty or inoperative range may be experienced which can be overcome by working through the difficult range with a continuous force. The explanation of the difficult range of working by reference to a proposed theory of excessive differential deformation appears to satisfy the results of many experiments. While a differential deformation theory of explanation may be helpful to an understanding of practice of the invention, I do not wish to exclude some other explanation for the phenomena encountered in the working of subdensity bodies. Accordingly, regardless of how these phenomena may be explained, I overcome undesirable conditions by working with a continuous force through and thus traverse the difficult range of rolling reduction.

I claim:

1. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing meal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body having voids defined by integrated wall structure in the form of interconnected particles; and proportioning said body substantially to said predetermined width and to a thickness adapted to deliver said product upon rolling said body to substantially maximum density.

2. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body having voids defined by integrated wall structure in the form of interconnected particles; and in preparing said material obtaining a more uniform rather than less uniform particle size to provide a large percentage of voids in said material from which a subdensity body of low rather than high density may be formed of low resistance to gas passage and of low energy requirement for rolling.

3. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body having voids defined by integrated wall structure in the form of interconnected particles; and rolling said body by the application of a continuous force of one rolling pass to substantially maximum density.

4. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body having voids defined by integrated wall structure in the form of interconnected particles; traversing a difficult range of rolling reduction by rolling said body in substantially one pass to maximum density; and

15

in preparing said material obtaining a more uniform rather than less uniform particle size to provide a large percentage of voids in said material from which a subdensity body of low rather than high density may be formed of low resistance to gas passage and of low energy requirement for rolling.

5. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size; introducing alloying material into said prepared material; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompact metal body having voids defined by integrated wall structure in the form of interconnected particles; and proportioning said body substantially to said predetermined width and to a thickness adapted to deliver said product upon rolling said body to substantially maximum density.

6. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompact metal body having voids defined by integrated wall structure in the form of interconnected particles; and proportioning said body substantially to said predetermined width and to a thickness adapted to deliver said product upon rolling said body to substantially maximum density of a portion thereof.

7. The method of forming a metal product of predetermined width, thickness, and metal grain size comprising: preparing oxygen-bearing metal-containing material to an average particle size substantially corresponding to said grain size, reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompact metal body having voids defined by integrated wall structure in the form of interconnected particles; and proportioning said body to an aspect ratio greater than ten to one and substantially to said predetermined width and to a thickness adapted to deliver said product upon rolling said body to substantially maximum density.

8. The method of making a metal sheet directly from oxygen-bearing metal-containing material and equivalent in thickness, width, and metal grain size to a conventional rolled metal sheet and comprising: preparing said material to an average particle size substantially corresponding to said grain size; reducing said prepared material to metallic form at less than melting temperatures; cohering said material upon reduction at a temperature sufficient only to cause coherence of substantially all of the particles thereof

16

to provide an uncompact metal body of finite shape proportioned substantially to said width and to a thickness adapted to deliver said sheet upon rolling said body to substantially maximum density, said body having voids defined by integrated wall structure in the form of interconnected metal particles; and hot rolling said body to substantially maximum density.

9. The method as claimed in claim 8 and the step of reducing said material in layers of different metal content to provide a metal sheet of different metal strata bonded in situ.

10. The method of making a steel sheet directly from oxygen-bearing iron-containing material of equivalent thickness, width and metal grain size to a conventional rolled steel sheet and comprising: preparing said material to an average particle size substantially corresponding to said grain size; reducing said prepared material to metallic form at less than melting temperatures; cohering said material upon reduction at a temperature sufficient only to cause coherence of substantially all of the particles thereof to provide an uncompact metal body of finite shape proportioned substantially to said width and to a thickness adapted to deliver said sheet upon rolling said body to substantially maximum density, said body having voids defined by integrated wall structure in the form of interconnected metal particles; and hot rolling said body to substantially maximum density.

11. A metal product made by the method set forth in claim 1.

12. A steel sheet made by the method set forth in claim 10.

13. The method of forming a metal article of finite shape and of less than solid metal density from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure adapted to form said finite shape while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompact metal body of said finite shape having voids defined by integrated wall structure in the form of interconnected particles; and freeing said body from said shaping structure.

14. The method according to claim 13 in which the density of the article to be produced is substantially controlled by controlling the particle size and particle size distribution of said material during preparation thereof.

15. The method of forming a metal article of finite shape and of less than solid metal density from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size and to a particle size distribution providing a volume of voids in a mass thereof in excess of that required to compensate for dimensional changes in the particles of the mass while in a reducing atmosphere; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure adapted to form said finite shape while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompact metal body of said finite shape having voids defined by wall structure in the form

of interconnected particles; and freeing said body from said shaping structure.

16. The method of forming a metal article of finite shape and of less than solid metal density from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size; disposing a loose mass of said prepared material in supporting structure adapted to form said finite shape; converting the loose mass of material to a cohered metal body having said shape by reducing the material to metal particles with reducing gas and cohering substantially all of the metal particles together to form a unitary uncompacted metal body having said finite shape, while maintaining the mass, during said reducing and cohering, within said supporting structure, said cohering being effected by heating the mass to a temperature sufficient only to cause coherence of substantially all of the metal particles and forming integrated wall structure defining voids in said body.

17. The method of forming a metal article of finite shape and of less than solid metal density from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size and to a particle size distribution providing a volume of voids in a mass thereof in excess of that required to compensate for dimensional changes in the particles thereof while in a reducing atmosphere; disposing a loose mass of said prepared material in supporting structure adapted to form said finite shape; converting the loose mass of material to a cohered metal body having said shape by reducing the material to metal particles with reducing gas and cohering substantially all of the metal particles together to form a unitary uncompacted metal body having said finite shape, while maintaining the mass, during said reducing and cohering, within said supporting structure, said cohering being effected by heating the mass to a temperature sufficient only to cause coherence of substantially all of the metal particles and forming integrated wall structure defining voids in said body.

18. The method of forming an article of finite shape and of less than solid metal density from oxygen-bearing metal-containing material subject to substantial dimensional change in a reducing atmosphere, comprising: preparing the material to relatively fine particle size and to a particle size distribution affording a volume of voids in a mass thereof in excess of that required to compensate for dimensional changes in the particles thereof while in a reducing atmosphere; supporting said prepared material to the shape of the said article; and while so supported, reducing said material to an uncompacted metallic form having voids and of said shape in a reducing atmosphere at least than melting temperatures.

19. The method of forming a metal article of finite shape and of less than solid metal density from oxygen-bearing metal-containing material subject to substantial dimensional change in a reducing atmosphere, comprising mixing with said material another material having a different characteristic of dimension change in a reducing atmosphere to substantially compensate for the said dimensional change of the first-mentioned material; supporting said mixed material to the shape of the desired article; and while so supported reducing said material to uncompacted metallic form having voids and of

said shape in a reducing atmosphere at less than melting temperatures.

20. The method of forming a metal article of finite shape and of less than solid metal density from an iron oxide material comprising: preparing the material to relatively fine particle size; disposing a loose mass of said prepared material in supporting structure adapted to form said finite shape; converting the loose mass of material to a cohered metal body having said shape by reducing the material to metal particles with reducing gas and cohering substantially all of the metal particles together to form a unitary uncompacted metal body having said finite shape, while maintaining the mass during said reducing and cohering within said supporting structure, said cohering being effected by heating the mass to a temperature sufficient only to cause coherence of substantially all of the metal particles and forming integrated wall structure defining voids in said body.

21. A metal article made by the method set forth in claim 13.

22. A metal article made by the method set forth in claim 20.

23. The method of forming a metal article from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure adapted to form a finite shape while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body of said finite shape having voids defined by wall structure in the form of interconnected particles; freeing said body from said shaping structure; and working said body of finite shape to substantially maximum density.

24. The method of forming a metal article from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size and to a predetermined particle size distribution providing a volume of voids in a mass thereof in excess of that required to compensate for dimensional changes in the particles of the mass while in a reducing atmosphere; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure adapted to form a finite shape while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to thus form a unitary uncompacted metal body of said finite shape having voids defined by wall structure in the form of interconnected particles; freeing said body from said shaping structure; and working said body to substantially maximum density.

25. The method of forming a metal article from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size; disposing a loose mass of said prepared material in supporting structure adapted to form a finite shape; converting the loose mass of material to a cohered metal body having said finite shape by reducing the material to metal particles with reducing gas and cohering substantially all of the metal particles together to form a unitary uncompacted metal body having said finite shape, while maintaining

## 19

the mass, during said reducing and cohering, within said supporting structure, said cohering being effected by heating the mass to a temperature sufficient only to cause coherence of substantially all of the metal particles and forming integrated wall structure defining voids in said body; freeing said body from said shaping structure; and working said body to substantially maximum density.

26. The method of forming a metal article from an oxygen-bearing metal-containing material, comprising: preparing the material to relatively fine particle size and to a predetermined particle size distribution providing a volume of voids in a mass thereof in excess of that required to compensate for dimensional changes in the particles thereof while in a reducing atmosphere; disposing a loose mass of said prepared material in supporting structure adapted to form a finite shape; converting the loose mass of material to a cohered metal body having said shape by reducing the material to metal particles with reducing gas and cohering substantially all of the metal particles together to form a unitary uncompact metal body having said finite shape, while maintaining the mass, during said reducing and cohering, within said supporting structure, said cohering being effected by heating the mass to a temperature sufficient only to cause coherence of substantially all of the metal particles and forming integrated wall structure de-

## 20

fining voids in said body; freeing said body from said shaping structure; and working said body to substantially maximum density.

27. The method of forming a metal article from an iron oxide material, comprising: preparing the material to relatively fine particle size; reducing a loose mass of said material to form a mass of metal particles at less than melting temperatures; supporting said mass of metal particles in shaping structure adapted to form a finite shape while heating said mass to a temperature sufficient only to cause coherence of substantially all of the particles thereof to form thus a unitary uncompact metal body of said finite shape having voids defined by wall structure in the form of interconnected particles; freeing said body from said shaping structure; and working said body of finite shape to substantially maximum density.

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