Apparatus for processing an elongated continuous green metal component, such as an extrusion, characterized by requiring sintering or reducing and sintering embodying conveyor means for receiving the continuous elongated component having an upper run for supporting the continuous elongated component without imparting force of a character to substantially change the configuration thereof as discharged from an extruder or other means for forming elongated continuous components for conveying through furnace means for sintering or reducing and sintering the elongated component and muffle means encasing the conveyor means affording atmospheric sealing of the protective gas for sintering or the reducing gas for reducing and sintering the elongated component.

15 Claims, 6 Drawing Figures
REDUCING AND SINTERING FURNACE MEANS

The present application is a continuation-in-part of my copending application Ser. No. 858,792, filed Sept. 17, 1969, now abandoned.

BACKGROUND OF THE INVENTION

In the fabrication of elongated articles such as wire, tubing and strip formed, for example, by extruding certain metal compound compacts into a continuous elongated component, it is characteristic that the elongated component has only nominal mechanical strength and great care must be taken to prevent deformation or the creation of imperfections in the elongated component while being sintered or reduced and sintered. After the elongated component has been sintered, it is of good mechanical strength and no longer is it necessary to carefully handle the elongated component. In the furnace means provided for treating the elongated component, a gas must be present to effect the protection or reduction of the elongated components and for economic purposes effective means should be provided to prevent the undue escape of gas from the furnace means.

A typical extruded metal compound compact with which the present invention may be employed was produced in the following manner. The by-product iron oxide, from spent pickling recovery was milled in a conventional manner until 50 percent of the iron oxide particles were less than eight-tenths micron and 50 percent being in a range of from eight-tenths micron to 10 microns. The aforementioned micron sizes were determined by a Coulter counter measurement and it is to be understood that particle size means size as thus determined. A binder was then prepared by adding 15 grams of corn starch to 100 milliliters of water and heating the solution to 160°F until a gel was formed. 4.2 grams of this binder was then added to 22.7 grams of the milled iron oxide powder and the combination was then mixed intimately in the mix-muller. The mixture of iron oxide and binder was then put into the cavity of an extrusion die having an opening of 0.115 inches in diameter and a pressure of 12,000 psi applied which formed an elongated green metal extrusion of nominal mechanical strength. A green metal component of the character noted may be introduced into a furnace having a reducing section providing an atmosphere of hydrogen at a temperature of about 1100°F for a period of 30 minutes, and then a sintering section which at a temperature of 2100°F for about 60 minutes effects sintering of the extrusion.

The present invention also has utility for utilization with tubing formed from a green metal elongated extrusion made from the same formulation of the wire component above described but extruded through a conventional tube forming die to form a tubular green metal component having an outside diameter of 0.115 inches and an internal diameter of 0.065 inches. Such tubing may be reduced and sintered by subjecting the same in a furnace in a hydrogen atmosphere at 1100°F for 30 minutes to effect reduction of the green metal component, and then subjecting the elongated extrusion to sintering in the sintering section of the furnace at a temperature of 2100°F for a period of 60 minutes.

The present invention also has utility for use with elongated continuous green metal components formed by forming means other than an extruder. By way of ex-

ample, elongated components of metal compound compacts may be formed into green preforms for continuous elongated components by depositing slurry-like material compound compacts upon forming means having a surface in which one or more longitudinally extending grooves are formed for receiving the slurry material so that the slurry material is shaped to form one or more elongated components and set therein. The grooves of the forming means are fabricated of material from which the shaped and set material in the grooves may be easily separated from the grooves without fracturing the shaped and set material, and then reducing and/or sintering the shaped and set material as aforesaid.

The slurry material for the purposes last noted may be composed, in part, of a reducible metal compound such as metal oxide powder which may be reduced and sintered to provide metal wire. The metal oxide compound powders of the slurry material may comprise oxide particles of which at least 35 percent are of a particle size of 10 microns or less as determined by Coulter Counter Analysis. Thus the particle size distribution will be considerably below the maximum 35 percent under 10 microns, and may have a mean particle size no greater than about six microns and at least 25 percent by weight, the particles will be below 2.5 microns in diameter. Optimum results are obtained when the apparent average particle of the powder is less than 1 micron in diameter.

That the average particle is less than one micron in diameter may be determined by Coulter Counter measurement where agglomeration is not a factor. However, where the particles tend to agglomerate, accurate particle size determination by means such as Coulter Counter measurement is not possible. It has been found, however, that such determination may be made by surface area determination. By determining the total surface area of a given powder one can readily determine the average particle size if one assumes perfectly smooth, spherical particles. Such determination may be made through the utilization of the formula:

\[ D = K(d \times S_A) \]

where,

- \( D \) = average diameter in microns of perfect spheres
- \( K \) = the constant 6
- \( d \) = density in grams per cubic centimeter
- \( S_A \) = surface area of the particles in square meters per gram.

For example, if one determines the surface area of iron oxide (Fe₂O₃) to be 5 m²/g and the density to be 5.24 g/cc then:

\[ D = 6(5.24 \times 5) \]

or

\( d = 0.23 \) microns

There are a number of known means for determining the surface area of powders each differing to some extent in results. It is found that the BET method developed by Dr. Paul Emmet and his associates in the late 1930's for use in measuring the available surface area of catalysts is the most reliable for determining the surface of metal compound powders. In the BET method the surface of the particles is coated with a monomolecular layer of adsorbed gas.
This is accomplished by passing a known quantity of gas, such as nitrogen, through a measured specimen at the boiling point of the gas (−195°C for nitrogen). Under these conditions the gas molecules form a tightly packed monomolecular layer on the surface of the specimen. A determination of the gas consumed by the specimen by monomolecular adsorption as compared to standard specimens readily yields a relatively accurate determination of the surface area of the powder.

For purposes of the foregoing, particle size determination of less than one micron shall be interpreted in accordance with BET measurements.

A suitable slurry material for forming continuous elongated green metal components may be composed of the aforementioned oxide powders together with a binder to provide the slurry material of a consistency to enable the ready deposit of the slurry material onto a grooved belt. A typical binder for iron oxide powder of the particle size range aforementioned may be a PVA-glycerine binder composed of a mixture of polyvinyl alcohol and glycerine in a 80 to 20 ratio mixed with the oxide powder to provide a slurry material of a consistency enabling its deposition onto the forming means as described to fill the grooves in the forming means on the belt.

Upon deposition of slurry material as aforesaid the separated and set green metal continuous elongated components are subjected to reduction and sintering to form elemental wire. Typically, the separated and set green metal continuous elongated components may be suitably passed through a reduction and sintering furnace in which the reduction is effected in an atmosphere of hydrogen at about 1000°F for 5 minutes, and sintering at a temperature of about 2100°F for about 5 minutes. It will be understood that the reduction and sintering time is dependent on the size and cross section of the material being treated.

In addition to the foregoing example of a slurry material suitable for fabricating metal wire, it will be understood that the apparatus of the present invention is applicable to any reducible metal compound, particularly those susceptible to reduction with hydrogen which have standard free energies of reaction with hydrogen less than about +15 kilo calories (per atom) of hydrogen at the reduction temperature. The metal compounds of particular interest in connection with the foregoing are the metal oxides such as the oxides Fe, Co, Ni, Cu, Mo, and W, and combinations thereof.

Although the use of hydrogen to provide the environment for reducing the foregoing slurry materials to elemental metal is preferred, other reducing materials may be employed. For example, the above recited metal compounds, and particularly iron oxide, can be reduced by partially or wholly substituting carbon monoxide for the hydrogen reducing environment.

Any metal compound powders having particles of any general shape (i.e., spherical, oblong, needles, or rods, etc.) and originated from any source (i.e., ore deposits, or concentrates, precipitates, etc.) may be employed in providing the slurry material for the present invention for forming elongated green metal components, for example, in the grooves of an endless belt, and subsequent reduction and sintering.

The sintered elongated components so derived will possess a substantially pore free structure, a smooth surface and can be made to exhibit densities in excess of 90 percent of completely dense material.

It will be understood that the foregoing examples are only illustrative of the class of elongated continuous metal compound compacts that require reducing and sintering and that the present invention has utility for many other materials including conventional powdered metals which may require sintering only.

THE INVENTION

The present invention comprehends the feeding of continuous elongated components of a metal compound compact onto a conveyor means for conveying continuous elongated components through furnace means having either a sintering section or a reducing section and a sintering section in a manner to prevent disruption of the configuration of the elongated components as discharged from the means for forming the continuous elongated components, and together with muffle means for encasing the conveyor means affording atmospheric sealing of the furnace to prevent the undue escape of protective or reducing gas from the furnace.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a diagramatic longitudinal sectional view of one form of apparatus of the present invention;

FIG. 2 is a diagramatic longitudinal section of a second form of apparatus of the present invention;

FIG. 3 is a diagramatic longitudinal section of a third form of apparatus coming within the invention;

FIG. 4 is a side elevational view of a portion of one form of endless conveyor suitable for use in the apparatus of FIGS. 1, 2 and 3;

FIG. 5 is a detail vertical sectional view taken on the line 5—5 of FIG. 4 looking in the direction indicated by the arrows; and

FIG. 6 is a side elevational view of another form of apparatus of the invention and with which the invention may be practiced.

DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

Referring now to FIG. 1, the apparatus there shown comprises furnace means 12 having a reducing section 13 and a sintering section 14 heated by conventional heaters as diagrammatically illustrated at 15. A plurality of pipes 16 are provided to supply reducing gas such as hydrogen to a gas chamber of muffle means 19 at the reduction section of the furnace means 12. Forming means such as a conventional extruder 17 is disposed adjacent the inlet of the furnace 12 for laying a continuous elongated component or extrusion 18 characterized by requiring drying, reducing, and sintering from a metal compound compact of such material contained within the hopper of extruder 17. An endless conveyor as at 20 supported in a conventional manner on drive and guide rollers 21 and 22 outwardly of the inner and outer ends of the furnace means is suitably driven so that the upper run of the conveyor passes through muffle means 19 mounted in the furnace means 12.

The muffle means 19 encases the upper run of the conveyor 20 and has downwardly curved end portions
23 and 24 having openings lying well below the gas reducing chamber of the muffle. The extruder 17 as shown in this embodiment of the invention is mounted in an air tight sealed relation in housing 25 formed as part of the muffle adjacent the inlet and of the furnace. The reducing gas supply pipes provide for the introduction of a reducing gas into the reducing gas chamber of muffle 19 at the reducing section of the furnace.

In the embodiment of FIG. 1, the upper run of the conveyor 20 moves through the muffle means in a horizontal plane. After laying of an elongated extrusion on the upper run of the conveyor as above noted, the elongated green metal component freely lies on the conveyor in a manner so that no forces are imparted to it which are sufficient to change the configuration of the extrusion as discharged from the extruder. It is important in the present invention that no forces be induced to the elongated green extrusion or component by the path of the conveyor. Once the extrusion has dried sufficiently it will crack if its shape is disturbed. The upper run of the conveyor supports the elongated extrusion in the manner noted while being conveyed through the furnace means. Upon passage of the elongated extrusion through the reducing section 13 of the furnace and the muffle the extrusion is reduced, and upon continued movement of the conveyor the extrusion is sintered as at the sintering section 14. After reducing and sintering of the elongated extrusion, it is of sufficient mechanical strength to pass through the downwardly outer curved end 23 of the muffle means 19.

In the foregoing apparatus it will be observed that the muffle means 19 encloses the upper run of the conveyor in its passage through the furnace means and, that the curved outer ends 23 and 24 have their openings below the reducing gas chamber of the muffle means so as to provide effective atmospheric seals preventing the undue escape of the reducing gas from the muffle.

The apparatus of FIG. 2 is particularly suited for extrusions of conventional powdered metals. It embodies furnace means 29 having a lubricant evaporating section 26 and a sintering section 27 with protective gas supply means 28 being arranged to provide an atmosphere for the gas chamber of the muffle. Again, suitable heaters 30 are embodied in the furnace to provide heat to effect the sintering of an elongated extrusion. Muffle means 43 is arranged with a portion thereof extending angularly upwardly in the furnace from the inlet 35 of the furnace to its outlet 36. An endless conveyor 38 supported on suitable drive and guide rollers 39 and 40 provides an upper run 42 extending on the same axis as that of the portion of muffle 43 within the furnace. The muffle 43 encases the upper and discharge run portions 42 and 43 of the endless conveyor and has inlet and outlet openings 44 and 45, respectively, effective to function to provide atmospheric sealing of the gas contained in the muffle 43 at the reducing section 26 of the furnace means 29. Forming means of the form of an extruder 50 is disposed adjacent the inlet opening of muffle means 43 and as before the extruder provides for the disposal of a continuous elongated extrusion of the characteristics aforediscussed onto the conveyor. In this embodiment of the invention the upper run 42 of the conveyor again freely supports the elongated extrusion in a manner so that no forces are imparted to the extrusion to alter its configuration or to impart imperfections to it. After passage of the extrusion through the sintering area, it is of sufficient mechanical strength to permit its path of movement to be changed in a direction around the curved portion 65 between the upper run 42 and discharge run 43 of the muffle and then outwardly of the discharge end of the muffle.

In the apparatus of FIG. 3 furnace means 70 having a reduction section 72 and a sintering section 73 like that of the furnace of FIG. 1 is provided and again with the reduction section having pipe means 75 for admitting a reducing gas, such as hydrogen, to the reducing gas chamber of muffle 76 at the reducing section of the furnace.

An endless conveyor 77 is supported and driven by known drive and guide rollers 78 and 79. The upper run of the conveyor and the intermediate lower surface of the muffle 76 within the furnace lie on a substantially constant radial lengthwise of and between the inlet and outlet of the muffle of an amount so that an elongated extrusion laid on the upper run of conveyor from the extruder 80 passes through the muffle without distortion or disruption.

The muffle 76 is provided with an inlet 82 and an outlet 83 with their being a curved portion 84 connecting the intermediate section 76 of the muffle with the downwardly extending portion 85. Again in the form of apparatus last referred to, the elongated extrusion is laid on the upper run of the conveyor at the opening 82 of the muffle, and with the upper run of the conveyor being of large radius for the purpose indicated, and which after the extrusion has been reduced and sintered by reason of its substantially increased mechanical strength passes around the curved end 84 and out the outlet 83 of the muffle. Again, the muffle 76 by disposal of its inlet and outlet 82 and 83 as above described afford atmospheric sealing for preventing undue escape of reducing gas from the muffle.

Also preferably in the apparatus shown in FIGS. 1 and 3 valve controlled piping 95 provides for the continuous escape of small amounts of hydrogen gas at the exit ends of the muffles, and which escaping hydrogen is consumed by a continuously burning flame.

The several conveyors of the aforedescribed apparatus may be of serially connected links of heat resistant material such as shown at 90 in FIGS. 4 and 5. The links at one end are formed with spaced apart lugs as at 91 between which a lug 92 of an adjacent link fits. A pin 93 extends through the several lugs to provide a pivotal connection at the ends of adjacent links to enable the links to pass around the drive and guide sprockets of the described apparatus. The several links are formed with a U-shaped groove as at 94 which when the links are abutted as when passing through the reducing and sintering sections of the furnaces form a continuous groove to receive and support the material from the extruder in the form of an elongated extrusion. The drive and guide sprockets are formed with suitable spaced apart transverse grooves to receive the several pins 93 for driving and guiding the endless conveyor through the described apparatus.

In FIG. 6 there is illustrated an apparatus embodying forming means 96 disposed and claimed in the copend-
The invention claimed is:

1. Apparatus of the character described comprising furnace means having a sintering section, muffle means extending through said furnace means having an inlet at one end of said furnace means and an outlet at the other end of said furnace means, conveyor means having a run thereof disposed for movement through said muffle means from the inlet to the outlet of the latter, extruder means for extruding a continuous elongated extrusion characterized by requiring sintering onto the run of said conveyor at the inlet of said muffle means, said run of said conveyor means defining for the support of the extrusion thereon without imparting force of a character to substantially change the configuration of the extrusion as discharged from said extruder and while being conveyed through said furnace means, said muffle means having sealing means affording atmospheric sealing of the inlet and outlet thereof to prevent escape of gas from said muffle means.

2. The apparatus of claim 1 in which said run of said conveyor means lies in a horizontal plane.

3. The apparatus of claim 2 characterized by the provision of means for mounting said extruder in air tight relation in said muffle means.

4. The apparatus of claim 1 in which said run of said conveyor means lies in a plane inclined upwardly from the inlet of said muffle means.

5. The apparatus of claim 1 in which said run of said conveyor means lies in a plane inclined upwardly from the inlet of said muffle means.

6. The apparatus of claim 1 in which said run of said conveyor means is curved on a radius between the inlet and outlets of said muffle means.

7. Apparatus of the character described comprising furnace means having a sintering section, muffle means extending through said furnace means having an inlet at one end of said furnace means and an outlet at the other end of said furnace means, conveyor means having a run thereof disposed for movement through said muffle means from the inlet to the outlet of the latter, extruder means for extruding a continuous elongated extrusion characterized by requiring sintering onto the run of said conveyor at the inlet of said muffle means, said run of said conveyor means providing for the support of the extrusion thereon without imparting force of a character to substantially change the configuration of the extrusion as discharged from said extruder and while being conveyed through said furnace means, said muffle means having sealing means affording atmospheric sealing of the inlet and outlet thereof to prevent escape of gas from said muffle means, said furnace means having a reducing section, and said muffle means having a reducing gas chamber at the reducing section of said furnace means.

8. The apparatus of claim 7 in which said run of said conveyor means lies in a horizontal plane.

9. The apparatus of claim 7 characterized by the provision of means for mounting said extruder in air tight relation in said muffle means.

10. The apparatus of claim 7 in which said run of said conveyor means lies in a plane inclined upwardly from the inlet of said muffle means.

11. The apparatus of claim 7 in which said run of said conveyor means is curved on a radius between the inlet and outlets of said muffle means.
12. Apparatus of the character described comprising furnace means having a sintering section, muffle means extending through said furnace means having an inlet at one end of said furnace means and an outlet at the other end of said furnace means, conveyor means having a run thereof disposed for movement through said muffle means from the inlet to the outlet of the latter, means for forming a continuous elongated component characterized by requiring sintering onto the run of said conveyor at the inlet of said muffle means, said run of said conveyor means defining a path extending along a line of continuous constant configuration so as not to impart force of a character to substantially change the configuration of the elongated component as deposited from said forming means and while being conveyed through said furnace means, and said muffle means having sealing means affording atmospheric sealing of the inlet and outlet thereof to prevent escape of gas from said muffle means.

13. Apparatus of the character described comprising furnace means having a sintering section, muffle means extending through said furnace means having an inlet at one end of said furnace means and an outlet at the other end of said furnace means, conveyor means having a run thereof disposed for movement through said muffle means from the inlet to the outlet of the latter, forming means for forming a continuous elongated component characterized by requiring sintering onto the run of said conveyor at the inlet of said muffle means, said run of conveyor means providing for the support of the elongated component thereon without imparting force of a character to substantially change the configuration of the elongated component as deposited from said forming means and while being conveyed through said furnace means, said muffle means having sealing means affording atmospheric sealing of the inlet and outlet thereof to prevent escape of gas from said muffle means.

14. The apparatus of claim 12 in which said run of said conveyor means lies in a plane inclined upwardly from the inlet of said muffle means.

15. Apparatus of the character described comprising furnace means, muffle means for containing a gaseous atmosphere extending through said furnace means, means for forming a continuous elongated component for passage through said muffle means, and sealing means for affording an atmospheric sealing between said muffle means and said forming means.

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