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EXTRUSION METHOD FOR MANUFACTURING SMOKELESS POWDER

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This invention relates to gas-producing charges and more particularly to a method for the production of gas-producing grains of small diametric cross section and of relatively large web.

It is well known that colloiding solvent can be removed from large web smokeless powder grains only with extreme difficulty. This difficulty increases in magnitude until it becomes wholly impractical to make grains from solvent powder which have webs greater than about 0.5 inch. Therefore, grains of relatively large web have heretofore been manufactured by solventless extrusion or by a casting process.

The conventional solventless extrusion method essentially comprises mixing the desired powder ingredients in a water slurry or mixing as a wetted mass, drying to the desired moisture content, rolling to a satisfactory colloided sheet by a suitable combination of rolling mills, forming a carpet roll from the rolled sheets, introducing the carpet roll into a hydraulic press and extruding therefrom a strand of desired cross section. As an alternative to forming a carpet roll, the rolled sheet is sometimes divided into shaped pieces and these pieces introduced into the press. This process is hazardous due to possibility of fire during the rolling step. In addition, poor consolidation in the extruded grains is often caused by the air between the rolled powder sheets and from a skin effect on these sheets brought about by surface evaporation or absorption of water, even though the press charge is evacuated prior to extrusion. Also, since considerable manual handling is involved, the overall process is relatively expensive. When demand for large diameter grains of varying webs increased, the solventless extrusion technique was found to be impractical and inefficient and even more hazardous because of the increased size of the press charge. Extrusion of large grains was also undesirable because of the increased cost and size of the necessarily larger presses.

An inherently safe and inexpensive method of fabricating homogeneously consolidated grains of large diameter has now been developed whereby grains of plastic composition, such as propellent grains of smokeless powder, may be cast utilizing inexpensive apparatus. This method and apparatus are disclosed in the copending application of Gordon W. McCurdy, Serial No. 28,218, filed May 20, 1948. Essentially the casting process therein disclosed comprises initially colloiding the nitrocellulose or other high polymer with or without a portion of plasticizer and granulating it, by conventional techniques, in the form of short cylinders or other configurations conducive to good packing. The mass of powder granules is introduced into a suitable casting container and held in place by applying pressure to the upper surface of the powder in the container. The remainder of the composition, consisting of a mixture of essentially nonvolatile explosive and non-explosive plasticizers, is introduced as a liquid into the bottom of the casting container through a manifold base assembly. This base assembly is so constructed that the incoming casting liquid is evenly distributed over the cross-sectional area at the base of the casting container and so that any air entrained in the casting liquid is prevented from rising into the powder granules and causing pitting and poor consolidation. When the powder granules in the casting container are completely covered, the

casting liquid system is disconnected and the unit comprising container and base assembly is stored at a temperature and for a length of time necessary to complete curing and consolidation of the grain. The grain is then removed from the casting container.

This casting process is very satisfactory and completely adapted to the production of grains of more than 6 inches in diameter but it has been found that while grains of lesser diameter may be produced which are of equally high quality, mold and handling costs make their production expensive and inefficient. The expense and inefficiency increases as the size of the cast grain decreases.

It is therefore an object of this invention to provide a process for the production of grains of a combustible, explosive, gas-producing material which at the same time have a small diametric cross section and a relatively large web.

It is a further object of this invention to provide a process for the production of such grains which is economical and relatively nonhazardous.

Generally described, the present invention is a process for the manufacture of grains of gas-producing material which comprises forming a cast mass of combustible, explosive, gas-producing material, curing the mass to insure complete consolidation, conditioning the mass at a suitable temperature for extrusion until its temperature is uniform throughout, charging the cast mass into an extrusion press and extruding the mass to form grains of small cross section. The solid cast mass is preferably formed so that its diameter is only slightly smaller than the diameter of the chamber of the extrusion press used so that a snug, sliding fit may be obtained. The mass may also be formed in oversize molds and then machined to the proper extrusion press dimensions. Both methods are equally operable but the latter obviously involves additional time and expense.

Having now described the present invention, the following examples are given for purposes of illustration.

Example 1

Cast powder masses were prepared by the casting process described herein with a composition as follows:

	Percent
Nitrocellulose (12.6% N)	56.4
Nitroglycerin	25.1
Ethyl centralite	0.9
Dimethylphthalate	11.0
Dinitrotoluene	6.5
Carbon black	0.1

These masses were machined on a lathe to solid cylinders 4¾ inches in diameter by 12 inches long. The solid cylinders were then extruded into perforated cylinders of small diameter and considerable length in a hydraulic press with conditions as follows:

Press temperature	°F.	139–144
Powder temperature	°F.	140
Pressure	p.s.i.	4400–5500
Die inside diameter	inches	1.165
Pin diameter	do	0.40

The extruded perforated cylinders were then cut to predetermined length to form grains which were of excellent quality and were perfectly consolidated. A number of tests were made using one of these extruded grains 1.20 inches in outside diameter by 0.4 inch inside diameter by 8 inches long in each test, in a 2 inch diameter by 12 inch long test chamber. The nozzle diameter was varied in each round so as to obtain a measurement of the burning rate at a variety of pressures. The results of these firings showed that all grains were of good quality and gave bal-

listic performance similar to unextruded cast grains of the same composition and size.

Example 2

Cast powder masses were prepared by the casting process described herein with the following composition:

	Percent
Nitrocellulose (13.15% N)	52.3
Nitroglycerin	30.3
Ethyl centralite	4.0
Triacetin	8.1
Dinitrotoluene	4.0
Potassium sulfate	1.3
Carbon black (add.)	0.02

These masses were machined on a lathe to solid cylinders 4⅜ inches in diameter by 12 inches long. The solid cylinders were then extruded into perforated cylinders of small diameter and considerable length in a hydraulic press with conditions as follows:

Press temperature	° F.	150–155
Powder temperature	° F.	132–140
Pressure	p.s.i.	3200–3800
Die inside diameter	inches	1.165
Pin diameter	do	0.40

The extruded perforated cylinders were then cut to predetermined length to form grains which were of excellent quality and were well consolidated. Firing tests showed that these grains gave ballistic performance similar to unextruded cast grains of the same composition and size.

Example 3

Cast powder masses were prepared by the casting process described herein with a composition as follows:

	Percent
Nitrocellulose (13.15% N)	58.5
Nitroglycerin	22.1
Ethyl centralite	8.0
Triacetin	8.4
Dinitrotoluene	2.5
Lead stearate	0.5

These masses were machined on a lathe to solid cylinders 4⅜ inches in diameter by 12 inches long. The solid cylinders were then extruded into perforated cylinders of small diameter and considerable length in a hydraulic press with conditions as follows:

Press temperature	° F.	151–154
Powder temperature	° F.	140
Pressure	p.s.i.	3200–4100
Die inside diameter	inches	1.165
Pin diameter	do	0.50

The extruded perforated cylinders were then cut to predetermined length to form grains which were of excellent quality and perfect consolidation. Firing tests showed that these grains gave ballistic performance similar to unextruded cast grains of the same composition and size.

The cast powder masses or charges to be extruded in accordance with this invention may be cast by any process which produces homogeneous, well-consolidated grains. The process disclosed in the application of Gordon W. McCurdy, Serial No. 28,218, filed May 20, 1948, is preferred because of the superior quality of the grains produced. It is highly desirable and of utmost importance that the matrix of the cast charges be well consolidated and completely free from pits formed by air bubbles in order to insure that the grains extruded therefrom are well consolidated and of satisfactory quality. The cast charges are preferably cured by storing them at a temperature of from about 30° C. to about 80° C. for the length of time necessary to complete their consolidation. Depending on the composition and the size of the charges produced by this casting process, suitable curing times have been found to vary from as little as one to as many as six hundred hours.

The cured cast powder masses or charges should be conditioned at a suitable temperature for extrusion prior to being charged into the press. The length of the conditioning time naturally depends on the size of the charge. For the 4⅜-inch charges produced and employed in Examples 1, 2 and 3, a conditioning time of approximately 4 hours is satisfactory while with larger charges correspondingly longer conditioning times are required to obtain a uniform temperature throughout. After the charge reaches the desired temperature, it is inserted into the chamber of the extrusion press and extruded. The press temperature and pressure are functions of each specific composition and optimum conditions must be determined empirically. Otherwise, standard extrusion techniques may be followed.

The molds or casting containers used to cast the charges may be made from any suitable material to which the grain will not adhere such as aluminum, brass or steel. Removal of the cured charge from the container is facilitated if the interior of the container is coated with a thin film of lubricant, such as silicone grease, prior to the introduction of the casting powder. The use of silicone-type lubricant has been found to have no effect on the ballistics of the grains made from the cast charges by extrusion according to this invention. Accordingly, silicone-type lubricant is preferred although other mold lubricants may be utilized providing that they do not materially affect the ballistics of the end product. Moreover, where it is desired to employ an external lubricant in conjunction with the extrusion step, the silicones are preferred. Generally, however, it is preferred to incorporate the extrusion lubricant in the cast charge and materials such as lead stearate or candelilla wax may be used for this purpose.

The powder compositions which have been set forth in the examples indicate the flexibility of the process of this invention and represent preferred embodiments but are not intended to restrict its scope to those specific formulations.

In general, the powder composition will comprise casting powder and casting liquid including the usual other ingredients, such as stabilizer, opaquing and/or coloring agents, and lubricants. If it is desired, a pulverulent burning rate control material may be included. In such case, the rate control powder and casting powder must be in the form of a heterogeneous mixture in the casting container prior to introduction of the casting liquid in order to prevent undesirable localization of the rate control material within the casting or extruded grains. The following is a generalized formula of a composition suitable in accordance with this invention:

	Percent
Nitrocellulose (11.2–13.4% N)	50–60
Plasticizers	35–47
Other ingredients	0–3.0

It will be understood that by plasticizers is meant either heating or explosive plasticizers and/or cooling or non-explosive plasticizers and in either or both cases, substances which are substantially nonvolatile. Such substances should be so chosen as to satisfy the calorific requirements of the powder desired and should be compatible with nitrocellulose and other constituents of the powder as well as stable.

It is therefore apparent that the process of the present invention presents may advantages over prior art methods of producing large web grains of gas-producing material. Grains of combustible, explosive, gas-producing material having small diameters and relatively large webs may now be produced safely and economically. Of equal importance is the fact that grains produced by the extrusion of cast powder according to this invention are superior in quality to grains produced by the conventional

solventless extrusion process and give enhanced ballistic performance.

What I claim and desire to protect by Letters Patent is:

1. In the manufacture of gas-producing charges, the method which comprises uniformly combining combustible propellant composition comprising nitrocellulose with substantially nonvolatile plasticizer for nitrocellulose in a mold, curing the contents of the mold at substantially atmospheric pressure until a solid mass is obtained, and extruding the mass to form grains of smaller diameter than the mass.

2. In the manufacture of gas-producing charges, the method which comprises uniformly combining granular smokeless powder comprising nitrocellulose with substantially nonvolatile plasticizer for nitrocellulose in a mold under substantially atmospheric conditions, curing the contents of the mold at substantially atmospheric pressure until a solid mass is obtained, and extruding the mass to form grains of smaller diameter than the mass.

3. In the manufacture of gas-producing charges, the method which comprises uniformly combining granular smokeless powder comprising nitrocellulose with substantially nonvolatile plasticizer for nitrocellulose in a mold under substantially atmospheric conditions, curing the contents of the mold at substantially atmospheric pressure and at a temperature of from about 30° to about 80° C. until a solid mass is obtained, and extruding the mass to form grains of smaller diameter than the mass.

4. In the manufacture of gas-producing charges the method which comprises uniformly combining granular smokeless powder comprising nitrocellulose and nitroglycerin with substantially nonvolatile plasticizer for nitrocellulose in a mold, curing the contents of the mold at substantially atmospheric pressure until a solid mass is obtained, and extruding the mass to form grains of smaller diameter than the mass.

5. In the manufacture of gas-producing charges, the method which comprises uniformly combining granular smokeless powder comprising nitrocellulose and nitroglycerin with substantially nonvolatile plasticizer for nitrocellulose in a mold, curing the contents of the mold at substantially atmospheric pressure and at a temperature of from about 30° to about 80° C. until a solid mass is obtained, and extruding the mass to form grains of smaller diameter than the mass.

6. In the manufacture of gas-producing charges, the method which comprises introducing granular smokeless powder comprising nitrocellulose into a mold, introducing nonvolatile plasticizer for nitrocellulose into the base of the mold until the powder grains in the mold are covered with the plasticizer, curing the contents of the mold at substantially atmospheric pressure and at a temperature of from about 30° to about 80° C. until a solid mass is obtained, heating the mass at substantially the predetermined extrusion temperature until the temperature of the mass is uniform throughout, and extruding the mass to form grains of smaller diameter than the mass.

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