This invention relates generally to the manufacture of propellant powder, and particularly to the manufacture of spherical and globular grains of smokeless powder.

In United States Patent No. 2,027,114, granted January 7, 1936, there is disclosed a process of manufacturing smokeless powder wherein droplets of lacquer, composed of smokeless powder base and solvent, are solidified while suspended in a nonsolvent medium. Such a process of manufacturing propellant powder has come to be known among those skilled in the art as the "globular powder" process. The technique of controlling the globular powder process so as to produce grains having various physical and chemical properties is further disclosed in United States Patents Nos. 2,160,626, granted May 30, 1939, 2,213,255, granted September 3, 1940, and 2,375,175, granted May 1, 1945. These patents disclose variations in the basic technique of manufacturing globular powder whereby to control the character, uniformity, and ballistic properties of the powder produced. While the globular powder process has proven eminently satisfactory for the production of powder grains suitable for use in small arms ammunition, i.e., where the diameter of the individual balls of powder does not exceed about ten to twenty-five thousandths of an inch, it was not, prior to the invention disclosed in our co-pending application Serial No. 148,742, filed March 9, 1950 (of which this is a continuing application), possible to produce globular powder grains of larger diameter, i.e., greater than about twenty-five thousandths of an inch, on a mass scale.

While it has heretofore been known, as pointed out in some of the patents above-mentioned, that control of the violence of agitation has its effect upon the granulation of the product—the more violent the agitation the smaller the grains and vice versa—it was not, prior to our invention, possible to produce batches wherein more than a small percentage of the grains were of "large diameter" and well rounded. Efforts to produce large diameter grains by reducing the speed of agitation of the usual globular powder equipment have resulted in batches of grains, practically none of which were sufficiently spherical to meet the requirements of the powder.

The object of the present invention, generally stated, is to provide a process of producing globular powder grains of large diameter and wherein the yield of each batch is predominantly of large diameter.

A further object of the present invention is to provide a process of manufacturing globular powder wherein the granulation may be controlled with greater uniformity than heretofore.

Other objects will become apparent to those skilled in the art when the following description is read:

The invention contemplates the production of globular powder grains, by presizing (as to volume) bodies of powder base lacquer, then suspending the bodies in a non-solvent medium until they become rounded; and then, while in their rounded condition, hardening them. The presized bodies are composed essentially of a solution of the powder base in a solvent which is substantially immiscible with the non-solvent medium, said solution being of a consistency such that the bodies are self-sustaining and self-coherent under the forces to which they are subjected while in suspension.

The presizing of the bodies to the volume which will yield spheres or near-spheres of the desired size, when hardened, may be accomplished in any of a variety of ways. To minimize the tedium of the shaping operation, it is desirable that the presized bodies approach, as closely as practicable, the ultimate shape, and consequently cylinders whose diameters approximate their lengths commend themselves as one of the ideal shapes for the presized bodies. Such cylinders may be produced by extruding the thick lacquer through a round orifice and successively slicing off increments of the appropriate length. Indeed, the technique of presizing the bodies by slicing the extruded lacquer is of advantage in the manufacture of both large grain and the regular sizes, as the volume of the individual soft bodies (and consequently the final grains) may be uniformly controlled within very close tolerances and the lacquer may be of viscosity low enough that the bodies will properly shape without further reduction of their viscosity.

The extruding and slicing may be accomplished in a variety of ways. For example, the lacquer may be forced under pressure through an orifice, across the exit and (i.e., the end exposed to the non-solvent medium) of which a shear periodically moves. The frequency of movement of the shear across the orifice determines the length of the extruded body, which is severed at each operation of the shear as long as the pressure on, and viscosity of, the lacquer remains constant. Furthermore, the extrusion may be accomplished under centrifugal force as by immersing a peripherally perforated vessel in the non-solvent medium and rotating the same at a speed such as to inject the lacquer from the interior of the vessel into the surrounding non-solvent medium. Where the exterior of such a centrifugal vessel is smooth, so that there is a substantial "slip" (as distinguished from companion movement) at the interface between the perforated periphery of the centrifugal vessel and the contiguous non-solvent medium, the latter will serve to shear the extruded lacquer into bodies whose length depends upon the speed of rotation of the centrifugal, as long as the viscosity of the lacquer, and the degree of "slip," remains constant. In connection with the extruding of lacquers generally, it has been common practice in the past to add a small but material amount of water to the lacquer before extrusion. The water acts as a lubricant as the lacquer passes through the orifice. It has been discovered, however, that the presence of such lubricating water in lacquer used in the process of this invention delays, though it does not prevent, the rounding of the presized bodies.

While the presized bodies are suspended in the non-solvent medium, it is important that the forces of agitation be only sufficient to maintain the suspension, and not of such violence as to subdivide the suspended bodies. Some subdivision and some coalescence of initially separate bodies is almost inevitable, but such are minimized by the precautions followed in the manufacture of globu-
lar powder generally, including in particular the addition of a protective colloid to the suspension.

If the presized bodies, when first suspended, are of consistency such that they do not readily yield under the interfacial tension exerted in the suspending medium, to approach the spherical shape, their viscosity is reduced, as by the introduction of some additional solvent into the suspending medium; said solvent, being not mutually soluble with the medium, is absorbed by the suspended bodies and softens them. Alternatively, the presized suspended bodies may be further softened by heating under pressure sufficient for the emulsionization of the solvent.

After the softening treatment, the bodies begin to yield to the forces of interfacial tension in the medium, and ultimately assume the spherical shape. Thereupon the solvent is removed from the bodies, while moderate agitation continues.

Example 1

As an illustrative example of the process, a powder base lacquer is made from three parts (by weight) of ethyl acetate to each part of nitrocellulose, and containing the usual amount of stabilizer, such as diphenylamine. This heavy lacquer is extruded through an orifice of 0.056 inch in diameter, and sliced off into lengths approximating the size of the final product. The cylinders of lacquer are preferably sliced within a moderately agitated water bath containing a protective colloid, for example, 4.75 per cent of animal glue, and saturated with ethyl acetate. The ethyl acetate content of the saturated bath is about 5 per cent to 6 per cent of the weight of the water.

When the extruded lacquer is substantially anhydrous, as indicated above, the rounding of the cylinders in the bath takes place practically instantaneously. If lubricant water is added to the lacquer before extrusion, the lacquer bodies must be subjected to the moderate agitation of the bath for a time period on the order of four hours. In either event, after the moderate agitation has proceeded for a time such that the bodies of lacquer are sufficiently rounded, the bath and contents are heated (previous operations having been at 68° to 70° C.) to 90° C., and maintained at that temperature until the solvent is sufficiently eliminated to harden the spherical grains.

The resultant grains are practically spherical, about 90 per cent being between .035 and .045 inch in diameter.

If the foregoing process is not carried out in a closed vessel, some ethyl acetate may be lost from the system by evaporation. Under such circumstances, the ethyl acetate removed from the cylinders may cause the viscosity thereof to become so great as to prevent the cylinders from becoming spheres. It is therefore advisable, with such operation, to add sufficient ethyl acetate to the water to maintain the water saturated with ethyl acetate and to maintain about a 3 to 1 ratio of ethyl acetate to nitrocellulose in the lacquer bodies until they have become sufficiently rounded.

With some types of extrusion equipment, it is desirable that the initial lacquer be more viscous than that which results from the 3 to 1 solvent ratio indicated in the foregoing example. In such case, a lesser amount of ethyl acetate is introduced into the initial lacquer, but additional ethyl acetate is added to the water bath to decrease the viscosity of the presized bodies of lacquer sufficiently to permit them to become rounded. A ratio of ethyl acetate to nitrocellulose in the lacquer bodies of about 5 to 1 is usually sufficient with a nitrocellulose of the gun cotton type to permit the extruded bodies to become spherical.

One suitable type of apparatus for accomplishing the extrusion and slicing, together with concomitant suspension of the lacquer bodies, is illustrated in United States Patent No. 2,027,114 to Olsen, wherein a still is shown having an agitator device which may be operated at speeds slow enough to maintain the plastic globules in suspension without subdividing them. To such a still may be added an input pipe whose exit end is situated below the level of the non-solvent medium in the still, and is there provided with an orifice of the desired size through which the lacquer may be forced; and across that orifice there may be arranged to rotate on a shaft concentrically with the agitator shaft therein, and provided with a moving blade or more blades, the frequency of whose movement across the orifice may be controlled so that the lacquer bodies are sliced off to the desired length.

An alternative arrangement for accomplishing the extrusion and slicing may consist of the addition to the still, in the aforesaid Olsen patent, of a centrifugal cup arranged concentrically with the agitator shaft therein, and provided about its outer periphery with a plurality of perforations of the desired size. The perforations should be located below the level of the non-solvent medium within the still, and the cup should be driven at a rotational speed considerably in excess of the speed of the agitator shaft. The lacquer may be introduced into the rotating cup through a conduit which extends from the exterior of the still, and terminates within the cup, so as not to interfere with rotation of the latter. Preferably the cup has a centrifugal cup and is concentric with the shaft on which it rotates, so as to minimize the tendency to induce concomitant swirl in the bath of non-solvent medium. In this case, where there is substantial slip at the interface between the perforated periphery of the centrifugal cup and the cup not limited by the non-solvent medium, the latter operates to shear lengths of lacquer as they are extruded through the perforations in the cup. The non-solvent medium is, however, agitated moderately during such extrusion and slicing so as to maintain the cut-off sections of extruded lacquer in suspension therein until they become rounded and hardened.

From the foregoing description, those skilled in the art should understand that the invention accomplishes its objects and provides not only a process whereby globular powder grains of large diameter may be made with facility and on a mass scale, but also provides a process whereby globular powder grains of any granulation may be manufactured so as to produce grains within narrow limits of diameter. While a complete embodiment has been disclosed in detail and modifications therein suggested, it is to be understood that the example given is for the purpose of illustrating the invention and is not to be construed in any way of limitation. It is recognized that those skilled in the art will make appropriate adjustment of the several variables in the process in order to adapt it to the peculiarities of any given operation, and consequently it is to be distinctly understood that the examples given, save as indicated in the appended claims.

Having thus described the invention, what is claimed and desired to be secured by Letters Patent is:

1. In the process of manufacturing globular powder grains wherein a lacquer is made of propellant powder base and solvent therefor which is substantially immiscible with water, the lacquer is subdivided into small bodies, the small bodies of lacquer are suspended in an aqueous medium, and such suspension is maintained until the small bodies of lacquer have assumed a spherical shape and are then hardened by removal of solvent from the lacquer, the improvement which comprises, presizing the bodies by extruding the lacquer through an orifice, cutting successive uniformly sized sections from the extruded lacquer as it emerges through the orifice, and collecting the cut sections in the aqueous medium.

2. The process of claim 1 wherein the cut sections are suspended in the aqueous medium immediately as they are cut.

3. The process of claim 1 in which the cut sections are of substantially the same diameter in each direction, std.

4. In the manufacture of globular powder grains by the process wherein a lacquer is made of propellant powder base and solvent therefor which is substantially immiscible with water, and the lacquer is subdivided and
suspended in an aqueous medium while the subdivided lacquer bodies are shaped and hardened, the method of controlling the size of the grains, comprising, extruding the lacquer through an orifice and into the aqueous medium, severing successive sections from the extruded lacquer, suspending the sections in the aqueous medium until they approach spherical shape, all without solidifying the lacquer, and then removing solvent from the shaped sections to solidify the same while the suspension is maintained.

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