

1

2,780,997

SMOKELESS POWDER GRAINS AND METHOD OF MAKING SAME

William O. Baker, Morristown, N. J., assignor, by mesne assignments, to the United States of America as represented by the Secretary of the Navy

No Drawing. Original application January 11, 1950, Serial No. 138,073, now Patent No. 2,668,132, dated February 2, 1954. Divided and this application May 22, 1953, Serial No. 358,216

1 Claim. (Cl. 102—98)

The present invention relates to the manufacture of smokeless powder grains and more particularly to the manufacture of such grains having greater mechanical strength than has heretofore been attained, making them more suitable for use as a rocket fuel. This application is a division of application Serial No. 138,073, now matured into Patent No. 2,668,132, filed January 11, 1950, for Smokeless Powder Grains and Method of Making Same.

A fuel commonly employed for the propulsion of rockets is smokeless powder, one of whose chief constituents is cellulose nitrate. The usual method of preparing the fuel charges for rocket propulsion consists of forming a plurality of grains of desired size and shape from a plastic mass of cellulose nitrate in a suitable solvent, said mass including the various other constituents of the charge, such as a nitrate salt and/or nitroglycerine or the like, dissolved therein or in suspension. In order to determine the ballistic characteristics of a rocket, it is necessary that each charge be controlled and uniform. Heretofore such determinations have been unreliable chiefly because of the fragmentation and fracturing of the grains during the burning of the fuel charge resulting in non-uniform combustion thereof.

It is therefore one object of this invention to provide smokeless powder grains that are superior to those heretofore produced, in that they offer greater resistance to fracture and fragmentation during ignition thereof, yielding determinable and reproducible ballistic characteristics of a rocket when employed as a propellant therefor.

Another object of the present invention is to provide a smokeless powder grain of superior mechanical strength than has heretofore been known.

Another object of the present invention is to provide a smokeless powder grain comprising a plurality of laminae having oriented cellulose nitrate crystallites or molecules in each lamina, and alternatively having laminae with non-oriented crystallites interdispersed among the laminae having oriented crystallites.

A further object of the present invention is to provide a smokeless powder wafer having uniaxial and/or uniplanar orientation of its cellulose nitrate crystallites.

A still further object of the present invention is to provide a smokeless powder grain comprising a mat of smokeless powder filaments, each filament having oriented cellulose nitrate crystallites, and alternatively being interwoven with other filaments whose crystallites are not so oriented.

A still further object of the present invention is to provide a process for producing uniaxial and/or uniplanar orientation of the molecules or crystallites of cellulose esters such as cellulose nitrate, acetate, butyrate, acetate butyrate, acetate propionate and the like and other cellulose compounds.

A still further object of the present invention is to provide a grain comprising a plurality of sections of a cellu-

2

lose compound, each section having uniaxial and/or uniplanar orientation of its cellulosic crystallites or molecules, and alternatively having sections with non-oriented cellulosic crystallites or molecules interdispersed among the sections having oriented crystallites or molecules.

Smokeless powder is usually formed of cellulose nitrate or a like cellulosic ester having suspended or otherwise contained therein various other constituents, such as nitrate salts and/or nitroglycerine or the like. According to the present invention grains of such a smokeless powder are made by forming a plastic composition of the cellulose nitrate by dissolving it in a suitable solvent such as acetone, alcohol, ether, or the like and introducing into said plastic composition the other desired constituents of smokeless powder. This composition is then extruded from a die or the like, the extrusion being carried out at high shear. The extrusion may be in the form of a sheet, ribbon, or filament or any other desired shape provided a high shear is maintained at the extrusion nozzle. The extruded form is then dried, while being maintained under tension along the line of extrusion during the drying step. This process results in substantially uniaxial orientation of the cellulose nitrate crystallites or molecules. The above process is equally applicable to other cellulose compounds such as acetate, butyrate, acetate propionate, acetate butyrate and the like.

In the above process, when substances whose structure includes crystallites are used, both crystallite and molecular orientation is obtained, but in the case of cellulose compounds not possessing crystallites, such as the mixed esters and highly plasticized cellulose esters such as compounded cellulose nitrate, the same desired properties are obtained by the molecular orientation in the absence of crystallites.

To obtain suitable orientation of the crystallites or molecules of the cellulose compound, the extrusion of the plastic mass in the above described process should be carried out at rates of shear greater than 100 second⁻¹ and at shearing stresses greater than 100 p. s. i. through orifices of 1/2 inch or less when the viscosity of the material being extruded is greater than 10⁵ poises. The temperature in the drying step should be such that the extrusion rapidly attains a value of greater than 10⁸ poises. The tension maintained on the extrusion during the drying step is critical only for the brief time in which the viscosity is low and during this time should be relatively great, preferably near the breaking strength of the hot ribbon. The sufficiency of the shear during the extrusion process to produce the desired orientation can be determined by X-ray examination of the product, due to the X-ray scattering effect caused by the orientation of the crystallites or molecules.

It has been found that cellulose nitrate and other cellulose compounds, having their crystallites or molecules oriented along an axis, present greater mechanical strength along the axis of orientation than can be obtained from commensurate samples of cellulose esters having random orientation of their crystallites or molecules. Since it is desirable, as discussed above, to have grains of smokeless powder of high mechanical strength for the fuel charge for rockets, the present invention contemplates in addition to the foregoing process, the advantages to be derived therefrom in producing smokeless powder grains from the product of the above process.

In one embodiment of this invention derived from the above described process, a ribbon of smokeless powder, or other mixture containing a cellulose ester or other cellulose compound amenable to the above process, is formed according to the foregoing process to produce uniaxially oriented crystallites or molecules therein. The dried ribbon is then divided into particles, sections or wafers of uniform size. Several of these wafers are then

laminated together such that the axis of crystallite or molecular orientation of each lamina is preferably in a different direction from the axis of each other lamina. The resultant grain then has greater mechanical strength along the lines of these various axes than can be obtained by a random orientation of the crystallites or molecules.

The lamination step may be carried out by any conventional process such as by merely applying pressure to the stacked particles or by coating the surfaces of each particle with a small quantity of solvent, stacking the pieces, and then drying the product. Unoriented laminae may, if desired, be interdispersed among the oriented laminae, sufficient mechanical strength being supplied by the latter to insure a sufficiently strong grain.

In another embodiment of the present invention a filament of smokeless powder, or other mixture containing a cellulose ester or other cellulose compound amenable to the above process, whose crystallites or molecules have been oriented according to the process described above is produced, and then the filament is divided into a plurality of shorter fibers or sections which are then formed into a mat by any conventional process, the various fibers or sections being placed in the mat to provide for axes of orientation in a plurality of directions. The grain formed thereby is afforded greater mechanical strength than can be obtained from random orientation of the crystallites or molecules as in the previously described embodiment. Also, if desired, fibers whose crystallites or molecules are randomly oriented may be intermingled with the uniaxially oriented fibers. This embodiment may also be applied to ribbons having oriented crystallites or molecules, as well as fibers, to produce a mat as here described.

A still further embodiment of the present invention involves an additional step to the above described process for producing uniaxially oriented crystallites, namely that a ribbon, sheet, or filament of cellulose nitrate, or other cellulose compound such as acetate, butyrate, and the like, having uniaxially oriented crystallites may be given a uniplanar orientation of said crystallites in addition to uniaxial orientation by rolling or pressing the uniaxially oriented product to from one-third to one-fifth its original thickness or less. This process, however, is limited to cellulose compounds which possess crystallites in their structures and is not generally applicable to molecular orientation as in the preceding process. Thus a charge of smokeless powder may be formed having great mechanical strength along the axis and in the plane of orientation of the crystallites thereof.

As in the case of the above described embodiments, the ribbon or sheet or the like of smokeless powder, or other cellulose compound amenable to the latter process, having its crystallites thus oriented may be divided into particles, sections, wafers, or fibers or the like, and if

desired various wafers may be laminated together or various fibers or ribbons may be matted together as above described, thereby forming a smokeless powder grain having the axis of orientation of various wafers or fibers on different lines from those of other wafers or fibers, and thereby imparting to the wafer great mechanical strength along each of the said axes and along the plane of orientation.

The uniplanar orientation above discussed may be accomplished independently of the uniaxial orientation, and when so done is not dependent upon the existence of crystallites as in the combined uniplanar and uniaxial orientation. The uniplanar orientation process may, therefore, be applied to cellulose nitrate and other cellulose compounds such as acetate, butyrate, acetate butyrate, acetate propionate, and the like to produce grains thereof, as above described, having great mechanical strength due to the uniplanar orientation of the molecules or crystallites thereof.

It is thus apparent that by use of any of the foregoing embodiments of the present invention a smokeless powder grain may be formed suitable for use as a motive fuel charge for rockets, and having such mechanical strength as substantially to eliminate the fragmentation or fracture of the grain due to the shock waves that traverse the charge on ignition, thereby imparting reproducible and predictable ballistic characteristics to the rocket flight.

The embodiments of the present invention herein described are only illustrative thereof. Other embodiments and modifications of the present invention within the spirit and scope of the appended claim will be apparent to those skilled in the art.

What is claimed is:

The process of producing a smokeless powder grain of great physical strength consisting of extruding a cellulose nitrate composition under high shear sufficient to produce detectable orientation of the molecules from X-ray scattering of the extruded composition to form a ribbon, drying said ribbon extrusion, maintaining said ribbon extrusion under tension while drying thereby imparting a uniaxial orientation to the cellulose nitrate molecules thereof, dividing the ribbon thus formed into wafers, and laminating said smokeless powder wafers to form said grain with diversified axes of uniaxial molecular orientation such that said grain has the characteristics of uniform combustion without fracture and fragmentation.

References Cited in the file of this patent

UNITED STATES PATENTS

1,784,758	Sheppard et al. _____	Dec. 9, 1930
1,808,998	Sheppard et al. _____	June 9, 1931
2,000,013	Dreyfus et al. _____	May 7, 1935
2,655,694	Piper _____	Oct. 20, 1953