United States Patent [19]

Abel

[11] **3,769,873**

[45] **Nov. 6, 1973**

[54] **PROCESS FOR UNIFORMLY DEPOSITING RESIN IN COMBUSTIBLE CARTRIDGE CASES**

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[22] Filed: **Jan. 25, 1972**

[21] Appl. No.: **220,642**

[52] **U.S. Cl.**..... **86/1 R,** 102/43 R, 102/DIG. 1, 162/146, 162/164, 162/184, 162/221, 117/63, 117/155 R

[51] **Int. Cl.**..... **F42b 5/26,** D21j 1/08, B44d 1/44

[58] **Field of Search**..... 162/184, 186, 221, 162/222, 231, 218, 183, 146, 164; 86/1 R; 102/DIG. 1, 43 R; 117/63, 155 R, 140 A

[56] **References Cited**

UNITED STATES PATENTS

3,474,702	10/1969	Remaly et al.	86/1 R
3,157,566	11/1964	Brafford	162/183 X
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[57] **ABSTRACT**

A process to control resin migration in combustible cartridge cases composed of nitrocellulose fibers and cellulose fibers is provided by adding to the conventional method the step of dipping the already formed and resin impregnated case into a liquid which is miscible with the solvent used to impregnate the resin into the felted cartridge case, but which is a non-solvent for the resin thereby precipitating the resin in situ and subsequently completing the formation of the case by drying.

7 Claims, 2 Drawing Figures

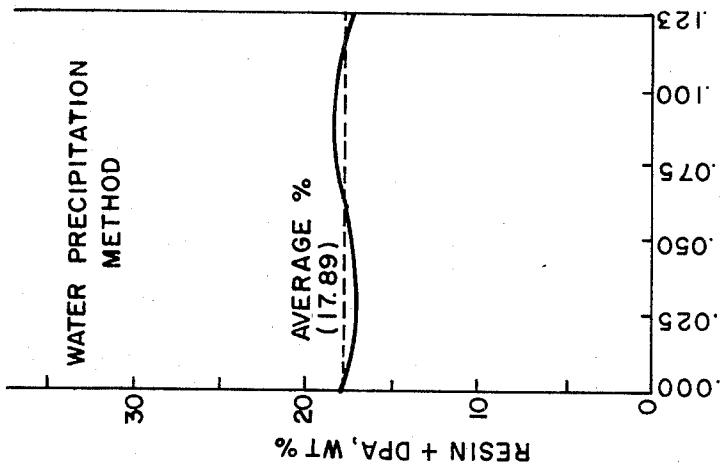

WATER PRECIPITATION METHOD
AVERAGE %
(17.89)
RESIN + DPA, WT %
0
10
20
30
DEPTH FROM SURFACE, IN.
.000
.025
.050
.075
.100
.123

FIG. 2

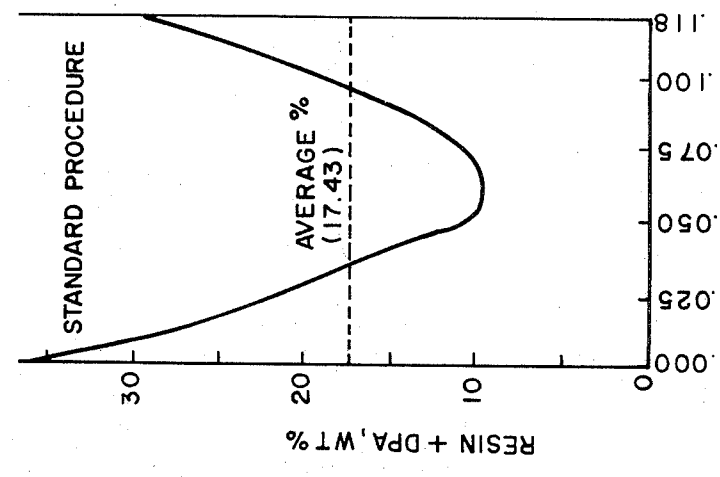

STANDARD PROCEDURE
AVERAGE %
(17.43)
RESIN + DPA, WT %
0
10
20
30
DEPTH FROM SURFACE, IN.
.000
.025
.050
.075
.100
.118

FIG. 1

PROCESS FOR UNIFORMLY DEPOSITING RESIN IN COMBUSTIBLE CARTRIDGE CASES

The invention described herein may be manufactured, used and licensed by or for the Government for governmental purposes without the payment to me of any royalty thereon.

BACKGROUND OF THE INVENTION

Combustible cartridge cases and processes for manufacturing them are well known to the art. A number of U. S. Patents have issued on these inventions, e.g. U. S. Pat. Nos. 3,320,886 and 3,474,702 both of which describe methods of manufacturing such combustible cases.

Generally, prior art processes use a water felting method to combine an energetic propellant component and combustible fibers into a combustible matte to form the overall shape of the cartridge, then treat the formed case with an organic solvent-resin system to impart strength to the case and subsequently remove the solvent and cure the resin by heat. This heating process causes the resin to migrate to the surface of the case during solvent volatilization and produces a case which is not completely satisfactory. Due to this non uniform distribution of resin, the case in certain instances, has a tensile strength and elongation which give poor impact sensitivity.

It is, therefore, an object of the present invention to provide a process to improve the impact sensitivity of combustible cartridge cases.

A further object is to provide an improved method of increasing the percent elongation obtainable from combustible cartridge cases.

Another object is to provide a process to insure homogeneous resin distribution in combustible cartridge cases.

A still further object is to remove organic solvent from the case without permitting migration of the resin by precipitating the resin in situ.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same become better understood by reference to the drawings and following description, wherein it is shown that the above-mentioned objects are attained and the prior art deficiencies are overcome by the additional step, in a conventional method, of treating the preformed resin impregnated case with a system which will precipitate the resin in situ and replace the solvent.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graph showing the resin-diphenylamine distribution in a combustible cartridge case prepared in the manner of the art.

FIG. 2 is a graph showing the resin-diphenylamine distribution in a combustible cartridge case prepared by the improved process of my invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The propellant component, combustible fibers and water are combined in a stock chest and agitated until thoroughly mixed. This slurry is pumped through a closed loop and sampled by a volumetric dispenser. On call, the dispenser releases this make-up slurry to a felting tank with continuing agitation. The fibrous material suspended in the agitated slurry of the felting tank is deposited on the screen surface of a felting die by means of vacuum filtration. The water from the slurry is drawn through the die into a dump tank, where it is measured by a differential pressure gage. When the proper volume of water has been drawn through the die, the die is raised from the tank, and the water is dumped into a white water storage vessel. This vessel supplies make-up water to the felting tank to maintain a constant slurry level in the felting tank. Excess water overflows through a screen catch box for disposal. The felted product is removed from the felting die and placed in a preforming mold. Air pressure on a rubber bag placed into the preformed case in the mold extracts some of the water and serves to compact the preform. The product then is placed in a single-pass hot-air oven and dried. The dry weight of the product is subsequently recorded for calculation of the composition.

After the preform is weighed it is impregnated with a resin solution, which is formulated in a make-up tank by combining an organic liquid solvent system, stabilizer, if necessary, and soluble resin, with continuous mixing. When the solution is clear, indicating complete solvation of all solids, it is pumped into a resin storage drum, which feeds and maintains a constant level in a resin dip tank. The felted, partially dried product is impregnated in this dip tank. The item is submerged below the surface of the resin until it is thoroughly saturated with the resin solution. It is then placed in a mold similar to the preform mold and excess resin solution extracted by inflating a rubber bag; this also serves to size the resin-wet preform.

After the impregnated product is removed from the mold, it is placed in a soak tank containing a liquid in which the resin is insoluble to precipitate the resin. After a specific dwell time the product is removed and placed in a batch oven for a partial dry, from there the product goes to a post forming operation for final mold sizing. The product is then placed in a batch or vacuum oven for final drying and complete solvent removal and then to the finishing stages for trimming to final dimensions.

The improvement of my invention lies in the resin precipitation step, a specific description of which, using water as the precipitating liquid, follows.

The method of water precipitation of resin consists of a normal impregnation of the item by immersion in a resin solution. The solvent system used was 60 wt % of toluene and 40 wt % of ethyl alcohol (2B). The system contained a small amount of water in that the ethyl alcohol was 190 proof alcohol. The solute used in the case application was Formvar 7/95 S resin and diphenylamine stabilizer. The surplus resin solution was expressed through a controlled time-pressure dwell cycle in a closed mold. Upon removal from the mold, the impregnated item was placed into water at ambient temperatures. The water precipitated the resin and replaced some of the solvents. A soaking period of 16 hrs was used. The resin precipitated items were then dried and the resin cured in an air circulating oven. The normal reforming steps were then carried out.

The resin precipitation step is essentially a method of fixing the resin within the case so that migration on solvent removal is prevented. This step is useful in any situation which requires fixing of the resin in a solvent-resin treated item. The advantages of my invention may be obtained by treating the item with a non solvent for the resin thus concurrently precipitating or fixing the resin and displacing a portion of the solvent. If water is

used in my invention as the precipatating agent, it can be used from a mixture of water and ice at about 40°F. up to about 150°F. the limiting factors are a lengthy precipitation time at the lower limit and solvent boiloff which excessively decreases the fiber density of the case, at the higher limit. The time of immersion is generally inversely proportional to the temperature used. Thus at lower temperatures it was found that 16 hrs. produced good results and at higher temperatures as little as 4 hrs. produced a case having the advantages of my invention.

The precipitating agent must be a non-solvent for the resin used and must also be largely miscible with the solvent system used for the resin. The primary criteria for the impregnating resin is that it be compatible with the propellant component and water insoluble when cured. Once the resin is selected then the solvent for the resin and the precipitating agent may be chosen according to solubility of the resin in the solvent, insolubility of the resin in the precipitating agent and substantial miscibility of the precipitating agent with the solvent.

The preferred resin of our invention is a polyvinyl formal resin such as Formvar 7/95 S or 7/95 E (registered trademarks for products marketed by the Monsanto Corp.). Acrylic resins, other polyvinyl resins and phenolic resins may also be used with our invention. The amount of resin used may vary from about 10 to 25% by weight of the case to achieve a good balance of strength and water resistance.

The base material of the case is usually formulated from about 65 to 90 percent by weight nitrocellulose, about 2 percent of a stabilizer for nitrocellulose such as diphenylamine or other stabilizers such as methyl centralite, ethyl centralite or 2 nitro diphenylamine and combustible fibers such as Kraft pulp fibers or sisal fibers. Other cellulosic fibers may also be used.

In the process of my invention, when nitrocellulose is utilized, the stabilizer is incorporated during the resin impregnation step. However, this is done as a matter of convenience and the stabilizer might be added at any earlier stage in the process.

The preferred method of use of my invention has been previously set forth. Several compositions used with my invention to achieve the desired properties are described in the following examples together with certain physical parameters, measured by testing the final cartridge case. It is, of course understood that these examples are meant to be illustrative and not restrictive of my invention.

EXAMPLE 1

FORMVAR 7/95 S RESIN CARTRIDGE CASE DATA

Material Composition	Wt %	Condition	Physical Parameters Tensile psi	Elongation %	Young's Modulus psi
Resin	19.5	77°F-50% RH	2130	3.8	89600
Nitrocellulose, 12.6% N	67.4	160°F-20% RH	1840	4.4	83600
Kraft	8.9	−65°F-100% RH	—	—	—
Sisal Fiber	3.2	77°F-98% RH	1940	5.2	86500
Diphenylamine	1.0	Water Wet			
Density, g/cc	0.718				

Resin deposition method: Solvent impregnation
Precipitation agent: Water
Drying Method: Air circulation oven, 150°F

EXAMPLE 2

FORMVAR 7/95 S RESIN CARTRIDGE CASE DATA

Material Composition	Wt %	Condition	Physical Parameters Tensile psi	Elongation %	Young's Modulus psi
Resin	17.4	77°F-50% RH	1100	5.1	52000
Nitrocellulose, 12.6% N	69.3	160°F-20% RH	911	6.4	46800
Kraft	12.3	−65°F-100% RH	1490	5.2	43900
Diphenylamine	1.0	77°F-98% RH	800	6.8	43800
Density, g/cc	0.565				

Resin dposition method: Solvent impregnation
Precipitation agent: Water
Drying method: Air circulating oven, 150°F

A comparison of the resin distribution throughout the finished case can be seen in the drawings. The percentage distribution at different levels from the surface of the case may be observed in FIG. 2 for the water precipitation method of my invention as compared with the distribution of a typical prior art cartridge in FIG. 1. The very uniform resin distribution of the samples prepared by the water precipitation method eliminated the high resin content evident on the surface of the prior art cartridge case. While this even resin distribution did not improve the tensile strength of low density cases, the percent elongation was improved, thus improving impact resistance of the material. Additionally, prior art cartridges of 0.718 g/cc density have a tensile strength and a modulus of 1510 psi and 76,000 psi respectively. Examples 1 and 2 present these physical parameters for a similar material made by the water precipitation method. A 41 percent increase in tensile strength over the prior art is obtained at 50 percent RH and 77°F.

Thus it is evident that the use of my invention, wherein a resin precipitation step is used to prevent resin migration, forms the basis for an advance in the art.

I wish it to be understood that I do not desire to be limited to the exact details shown and described for obvious modification will occur to a person skilled in the art.

I claim:

1. The method of making a combustible cartridge case which comprises the following steps:

depositing an aqueous slurry of fully combustible fibers consisting essentially of a mixture of nitrocellulose fibers and cellulose fibers on a cartridge shaped felting die to make a preform;

drying the preform;

impregnating the dried preform with a solution of a water-insoluble, non-elastomeric organic resin in a liquid organic solvent;

contacting the resin impregnated preform with a liquid, which is substantially miscible with said organic solvent but in which said organic resin and said fibers are insoluble, whereby the resin is precipitated from said solvent solution; and

drying the preform containing said precipitated resin to obtain a combustible cartridge case, wherein said resin is distributed essentially uniformly throughout said case.

2. The method of claim 1, wherein the resin is deposited in amount between about 10% and 25% by weight of the cartridge case.

3. The method of claim 1, wherein the resin is selected from the group consisting of polyvinyl-formal resin, acrylic resin and phenolic resin.

4. The method of claim 1, wherein the liquid employed to precipate the resin is water.

5. The method of claim 4, wherein the resin is polyvinyl-formal and the organic solvent therefor is a mixture of toluene and ethyl alcohol.

6. A felted article for use as a combustible cartridge case, consisting essentially of a resin bonded, felted fibrous matte of combustible fibers consisting essentially of a mixture of between about 65% and 90% nitrocellulose fibers and between about 35% and 10% cellulose fibers, wherein said resin is a water-insoluble, non-elastomeric organic resin distributed essentially uniformly throughout said matte and is not chemically bonded to said fibers.

7. The felted article of claim 6, wherein the resin is selected from the group consisting of polyvinyl-formal resin, acrylic resin and phenolic resin present in amount between about 10 and 25 percent by weight of the case.

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