June 15, 1943. G. B. TAYLOR 2,321,635

TREATMENT OF POLYAMIDE FILMS

Filed Nov. 26, 1940

Fig. 1.

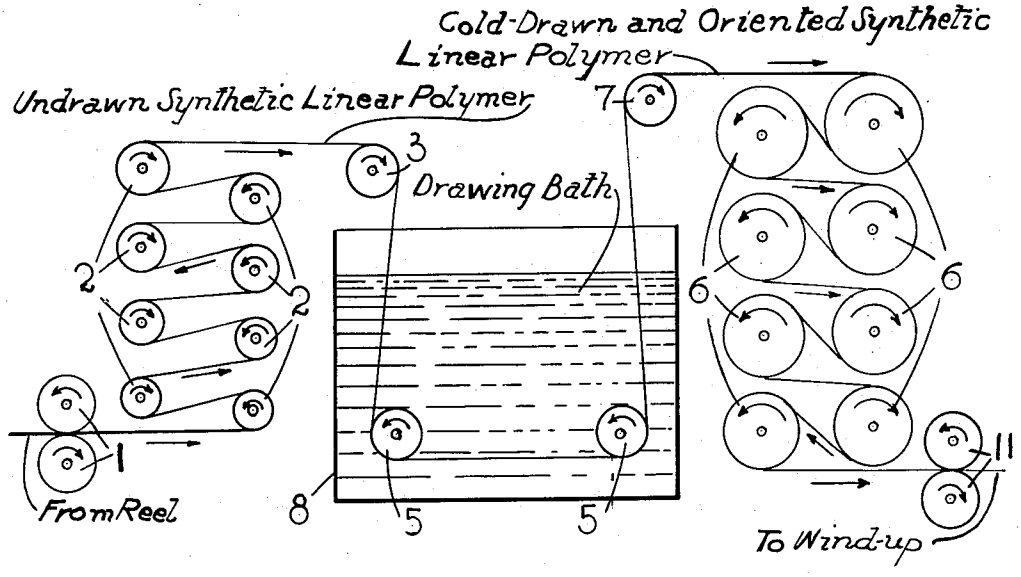
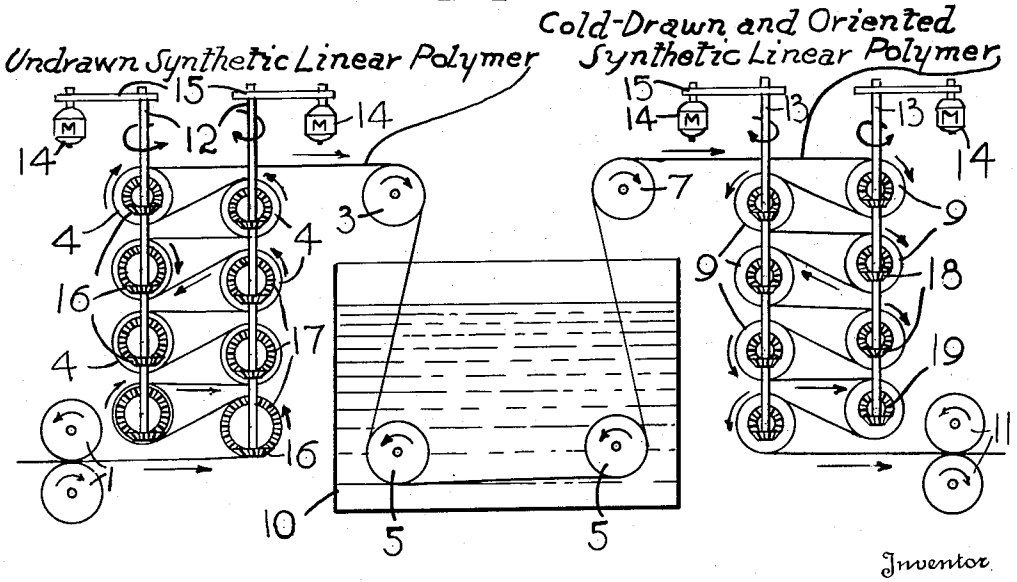


Fig. 2.



Inventor

Guy B. Taylor

By R. F. Miller.

Attorney

Patented June 15, 1943 2,321,635

UNITED STATES PATENT OFFICE

2,321,635

TREATMENT OF POLYAMIDE FILMS

Guy B. Taylor, Wilmington, Del., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

Application November 26, 1940, Serial No. 367,289

10 Claims. (Cl. 18—15)

This invention relates to the treatment of orientable sheets, films, and ribbons and more particularly to improvements in the production of oriented films from synthetic linear polymers.

Examples of synthetic linear polymers which are capable of being molecularly oriented and to which this invention is advantageously applied are those described in U. S. Patent 2,071,250. Since the most valuable of these polymers are the polyamides disclosed in U. S. Patents 2,071,253 and 2,130,948, this invention is described more particularly in connection with the polyamides. These polymers are capable not only of being formed into filaments which can be cold drawn (permanently elongated, often as much as 400% or more, by application of tensile stress below the melting point of the polymer) into oriented fibers, but also of being formed into films, sheets or ribbons (hereinafter referred to as films) which likewise exhibit upon cold drawing molecular orientation in the direction in which the film has been elongated. The cold drawing of the film results in an increase in stiffness, a decrease in sensitivity to distortion, and an improvement in other properties which enhance their utility for various purposes.

Cold drawing of filaments by feeding the filaments to a revolving spool or bobbin, wrapping the filaments several times around the spool, and then drawing the filaments around a second spool revolving at a greater speed than the first spool is comparatively simple. But apparatus which successfully draws filaments is not applicable to the continuous drawing of films because one cannot wrap film several times around a rotating spool and at the same time continuously feed on and take off film. Films of the type used in this invention can be oriented by cold rolling but this procedure is disadvantageous for certain purposes because of the tendency of the film to become distorted by buckling in the process.

This invention has as an object a new and improved method and apparatus for cold drawing films which are susceptible to cold drawing.

It should be understood that the expression "cold drawing" is not limited to drawing in the cold. It signifies that the drawing of the film is carried out in the solid state, i. e., below the melting point of the film.

The above objects are accomplished by the means described in connection with the accompanying drawing in which

Fig. 1 is a diagrammatic view in elevation of a form of apparatus suitable for carrying out my invention, and

Fig. 2 is a similar view of a modified form of apparatus.

In either of the methods for carrying out the invention by the apparatus shown in the drawing the film is passed over a first series of rolls (2 in Fig. 1 and 4 in Fig. 2) where the tension is built up by a slightly greater peripheral speed of each succeeding roll until the tension on the film at the last roll is sufficient under maintenance of this tension by the second series of rolls 6 or 9 to cold draw the film during its passage through the hot bath contained in tanks 8 or 10 but not at any other point. The bath of hot fluid within which the film is cold drawn is preferably a mild swelling agent such as water. The bath functions as a focal point for the cold drawing because the draw point is reached only at this point by reason of the fact that hot film draws more easily than cold film. From the draw bath the film passes to the second series of rolls 6 or 9 which have a greater average peripheral speed than the first series of rolls. The succeeding rolls in the second series of rolls have slightly decreasing peripheral speeds. The present process comprises, therefore, continuous cold drawing of the film by moving it under successively increasing tension built up at a plurality of first tension stages along the path of travel of the film until the tension built up is sufficient to draw the film at a higher temperature, cold drawing the film by passing it under said tension through a zone of higher temperature, and then successively diminishing the tension on the cold drawn film at a plurality of second stages along the path of travel of the film.

The film or sheeting passes between the pinch rolls 1 from the casting drum on which it is produced or from a reel not shown around the series of driven rolls 2 in Fig. 1 or 4 in Fig. 2. These rolls lead the film into the drawing bath over guide roll 3 and under guide rolls 5 contained in the bath, from where the film is drawn from the bath over guide rolls 7 by means of the second series of rolls 6 in Fig. 1 and 9 in Fig. 2. The film then passes through the pinch rolls 11 to the final wind up roll not shown. The pinch rolls 1 are braked or driven to establish a small tension on the film between the pinch roll and the first of the series of rolls 2 or 4.

In Fig. 1 each succeeding roll 2 is driven at the same angular speed and is of slightly greater diameter than the preceding roll with consequent progressive increase in peripheral speed and ten-

sion on the film. Although cold drawing takes place only in the path, the film whether drawn or undrawn like all film materials under tension exhibits some elastic stretch which is taken care of in the feed set of rolls by a slight increase in diameter from roll to roll. The number and size of the rolls are such that at roll 3 the tension on the film is sufficient to draw the film in the bath. The series of rolls 6 have a substantially greater average peripheral speed, usually at least twice, and generally three to four times, that of the rolls 2 in order to take up the increased length of the film after cold drawing. The tension thus built up on the film between the guide rolls 3 and 7 causes cold drawing of the film in the bath. Each of the rolls 6, driven at the same angular speed, progressively decrease in diameter from the largest roll receiving the film from guide roll 7 to the smallest roll of the series from which the film passes to the pinch rolls 11. This set of rolls operates in the same principle described in connection with rolls 2 but in reverse order. In this manner the belt tension of the film around the rolls decreases in small amounts from its maximum at the first of the rolls 6 to substantially zero at the last of these rolls.

The apparatus shown in Fig. 2 operates in the same manner with the exception of the means used for varying the peripheral speed of the rolls. As in Fig. 1 the first or feed set of rolls 4 is essentially a brake device while the second or take off set 9, which runs at a higher speed, provides the pull and lets down the tension. The rolls 4 and 9 are all of the same diameter but are driven at different angular speeds by means, which for simplicity in illustration, consists of shafts 12 for rolls 4 and shafts 13 for rolls 9 driven by motors 14 through belts 15. The shafts 12 have rigid therewith equal sized pinions 16 meshing with gears 17 which are provided on the rolls 4 and which progressively decrease in size from the gear on the roll receiving the film from the pinch roll to the gear on the roll from which the film passes to the guide roll 3. The rolls 9 are driven at a greater average peripheral speed than that of the rolls 4 either by means of increased speed in the driving motors, or by appropriate ratio of the pinions and gears 18 and 19 with respect to that of the gears and pinions 17 and 16. In either case the rolls 9 are driven at successively diminishing peripheral speed by pinions 18 of the same diameter meshing with gears 19 of increasing diameter.

Although it is usually preferred to operate with six or more rolls in each series, the process may be carried out with a lesser number, and a different number of rolls may be used in the two series. Also it is possible to include in each series idler rolls having the same peripheral speed as the preceding roll. The force required to cold draw the film will determine the number of rolls necessary to build up the necessary tension and this in turn will depend upon the thickness of the film and the nature and temperature of the liquid or vapor in the drawing bath. It should be observed, however, that the contact angle made by the film on the rolls is also a factor in determining the number of rolls required. The greater this angle the greater the tension built up and consequently the fewer the number of rolls required. Angles of contact between 150° and 230° are most practical. The tension built up is independent of the diameter of the rolls and the width of the film but increases with the coefficient of friction of the surface of the rolls. Polished steel rolls are satisfactory.

With increase in temperature the films draw more uniformly and at lower tension. The temperature in the bath must of course not be so high as to melt the film. Hot water or steam is used to advantage in the draw bath in the case of polyamide films, although it is not necessary that the liquid or vapor have a swelling action on the film. Other useful drawing media are hot alcohols, e. g., methanol, ethanol, or benzyl alcohol, or in fact any liquid which does not degrade the film, e. g., mineral oil. By way of example, it may be observed that with water at 95° C. in the bath the force required to draw polyhexamethylene adipamide films of 10'' width and 10 mils thickness is 250 lbs. To build up this tension requires about six rolls.

The successive increase in peripheral speed from roll to roll in the first series of rolls and the successive decrease in peripheral speed from roll to roll in the second series of rolls depends on the elasticity of the film in use. In general, however, the total change in peripheral speed from the first to the last roll in each series of rolls will vary from 5 to 25%. The value may differ between the two series of rolls since the elasticity of the undrawn film and the drawn film may differ. The difference between the average peripheral speed of the first and second series of rolls will depend upon the extent to which the film can be cold drawn and the extent to which it is desired to elongate it. By effecting the cold drawing at elevated temperature, e. g., about 75–150° C., it is possible to obtain a uniform product even though the film is not cold drawn to its maximum extent. This is frequently not the case if drawing is effected at ordinary temperatures.

The present invention provides a practical and economical method for cold drawing continuous films which are capable of being oriented by cold drawing. Such films include those made from the synthetic linear polymers described in U. S. Patent 2,071,250, polymerized ethylene of the type described in U. S. Patent 2,153,553, and various vinyl polymers. The process is particularly well adapted to the cold drawing of films made from synthetic linear polyamides of the types described in U. S. Patent 2,071,253 and 2,130,948.

Orientation by cold drawing is advantageous over orientation by cold rolling, especially in thin films, in that the material is much less subject to distortion during the process. Variations in thickness cause no distortion in the drawn films but are quite serious in rolled films. The necessary tension is obtained by the arrangement of multiple rolls described herein without slip of the film on the rolls which would tend to mar and scratch the film. The stepwise raising and lowering of the tension aids in eliminating distortion tendencies. The drawing which takes place in the heated medium is uniform to a high degree.

Films prepared according to the process of this invention are useful as photographic film bases, wrapping film, safety glass interlayers, leather substitutes, and various other uses to which films are put. The films can be prepared in transparent or opaque form. They may contain modifying agents such as plasticizers, pigments, dyes, and the like. In general the presence of a plasticizer decreases the force required to draw the films.

As many apparently widely different embodi-

ments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that I do not limit myself to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A continuous process for cold drawing film which is susceptible to cold drawing, said process comprising moving said film through a zone, which is heated above the temperature of the film before it enters the zone but below the melting point of the film, under sufficient tension to cold draw the film in the heated zone, successively building up the tension on the film entering the heated zone at a plurality of tension stages along the path of travel of the film, and then successively decreasing the tension on the cold drawn film at a plurality of second tension stages which are positioned along the path of travel of the film and which draw the film from the heated zone.

2. A process for cold drawing a continuously moving film capable of being cold drawn, said process comprising moving said film under successively increasing tension applied at a plurality of tension stages along the path of travel of the film until the tension built up is sufficient to draw the film on its subsequent passage through a heated fluid, cold drawing the film by moving it through a hot fluid medium above the temperature of the film before it enters the bath but below the melting point of the film and at a speed substantially greater than that of the film after it has passed over said tension stages and before it has reached the heated fluid, and then successively decreasing the tension on the cold drawn film over a plurality of second stages.

3. A process for cold drawing a continuously moving film of synthetic linear polymer capable of being oriented upon cold drawing, said process comprising moving said film under successively increasing tension applied at a plurality of tension stages along the path of travel of the film until the tension built up is sufficient to draw the film when immersed in a hot liquid, passing the film through a bath of liquid heated to about 75° to 150° C., and cold drawing the film in said bath by moving the film at a speed at least twice that of the film after it has passed over said tension stages and before it has reached the hot liquid, and then successively decreasing the tension on the cold drawn film over a plurality of second stages along the path of travel of the film.

4. The process set forth in claim 3 in which said polymer is a polyamide and said liquid is water.

5. A process which comprises permanently elongating a film susceptible to cold drawing, said process comprising passing the film over two series of rolls, drawing the film between the two series of rolls, the succeeding rolls in the first series having slightly greater peripheral speeds from roll to roll and the succeeding rolls in the second series having slightly smaller peripheral speeds from roll to roll but having an average peripheral speed substantially greater than the first series of rolls, and each series of rolls having a sufficient number of rolls to build up sufficient tension on the film between the two series to draw the film and having sufficient angular contact with the film to prevent slippage of the film on the rolls.

6. A process for cold drawing films of synthetic linear polymer capable of being oriented upon cold drawing, said process comprising passing said film over a series of driven rolls in which succeeding rolls have a peripheral speed slightly greater than that of preceding rolls and thereby building up the tension until it reaches the draw point of the film on its subsequent passage through a draw bath, then passing the film through a draw bath containing a mild swelling agent for the film and there cold drawing the film by passing it over a second series of driven rolls in which succeeding rolls are driven at a peripheral speed slightly less than that of preceding rolls, said second series of rolls having an average peripheral speed substantially greater than that of the first mentioned series of rolls.

7. Apparatus for drawing films of polymeric material capable of being cold drawn to permanent and substantial increase in length, said apparatus comprising a vessel adapted to contain a fluid medium to facilitate drawing of the film, two series of driven rolls arranged to convey the film through said vessel, one of said series of rolls being adapted to lead the film into said vessel, means for driving said last mentioned rolls at such successively increasing speeds which will build up the tension of the film passing through the rollers and into the vessel to the extent required to draw the film in the vessel, the other of said series of driven rolls being adapted to lead the film from said vessel, means for driving said last mentioned rolls at successively decreasing speeds and at an average peripheral speed substantially greater than that of the first mentioned series of rolls.

8. The process set forth in claim 1 in which said film is a polyamide film.

9. The process set forth in claim 5 in which said film is a polyamide film.

10. The process set forth in claim 5 in which said film is a polyhexamethylene adipamide film.

GUY B. TAYLOR.