Fig. 3.
This invention relates to a process and apparatus for modifying the physical characteristics of plastic materials, and more particularly, to a process and apparatus for converting plastic materials having a crystalline phase, such as soap, into compacted ultra-microcrystalline material by subjecting them to intense shearing and compacting forces. The conversion operation may coincidentally include cooling of the material while being subjected to such forces and either aerating or de-aerating of the resulting material. Commercial detergent soaps as well as certain other plastic materials have a solid gel structure made up of a network of crystals, usually fibrous in nature, in which a non-crystalline material is included. Thus soap is a two-phase system having a crystalline soap phase and a liquid crystalline or jelly-sol phase. Ordinary cooled neat soap or such soap which has been flaked and partly dried has such a structure. Milling of such soap by conventional roller milling procedures to some extent breaks down the crystal structure and compacts such soap. The present invention is concerned with an entirely new order of conversion of such soap not possible by conventional milling. The extent of particle size reduction required and the compacting forces under intense shearing stresses necessary to produce the conversion of the present invention cannot be obtained with milling rollers because of the large clearances necessitated by mechanical considerations and the inability to hold the material within a confined space where such forces can be developed.

In accordance with the present invention, conversion of the soap or other plastic material is accomplished by employing a rapidly moving carrier band for drawing thin soap films by molecular attraction through narrow apertures or slots under tearing and dispersing conditions to produce intense shearing and compacting. The material is kept in a turbulent condition in thin layers or films on relatively large surfaces on metal members and heat may be rapidly withdrawn through such surfaces to provide effective temperature control. Aeration of the soap during intense shearing and compacting is made possible by using a tightly fitted chamber around the band and introducing air or other gas into the chamber. Likewise, de-aeration is made possible by the exposure of large surface areas of the material in the form of thin ribbons or flakes to negative pressures developed within a chamber surrounding the band by an exhaust.

The present invention employs the extraordinary molecular forces which cause films to adhere to the surface of a metal, such as is revealed in the soap lubrication of cold wire drawing dies, to draw the soap film as a coating on a steel or other metal carrier band through multiple die slots, despite strong particle shearing and compacting forces endeavoring to strip the film off the carrier band. (This surface adhesion phenomenon is fully treated by W. M. Bos in "Petroleum Processing," December 1948.) It enables the use of a high speed lightweight steel or other metal belt or band, as a carrier member for soap films to obtain high compacting pressure and high shearing forces in contrast to the heavy structural arrangements of the prior art for slow moving solid material through the "bite" of rollers. The invention thus eliminates much of the power waste and heat dissipation trouble encountered, for example, when roller milling a relatively thick soap flake (200 microns) through the "bite" of heavy rollers in an endeavor to abrade a crystalline particle to less than 1 micron. By reducing the flake thickness to 5 microns, for example, as can be done by the present invention, the required power is largely utilized for crystal reduction and compacting the soap instead of being wasted by working the bulk of the material unnecessarily.

Air dispersion and retention during the intensified shearing and compacting of the material is obtained in the present invention by the entrainment gas pressures developed within the close fitting die slot, and by the high speed entrainment action of the carrier band at the entrance to the slot and in small air chambers communicating with the die slots. Temperature regulation is easily obtained by spreading the material out to make films measuring about 5 microns thick on elongated heat-transfer surfaces by the use of a high speed carrier band operating in a slot and cooling the walls of the slot or by cooling the band, or both.

As stated above, soap is a two-phase system having a crystalline phase of hard, less soluble material, and a jelly phase of soft, more soluble material. When melted soap is solidified, large crystals form (6 to 15 microns), and these crystals contact each other, or are bonded to each other by crystalline material so that the resulting solid structure is substantially as hard as the crystalline phase and fractures along the crystal shear planes. When soap containing moisture is agitated while cooling until semi-fluid (say at 160° F. for 17% moisture soap), the crystal sizes are reduced and if left to "set," these small crystals bond together with crystalline material.
to form a fine-grained, hard, solid structure (Bodman process). If this same soap is agitated while further solidified (say to 130° to 140° F.) the crystals are still reduced as before, and in addition, the bonds which formed between crystals in Bodman’s soap are broken down, and the jelly-phase material surrounds the fine crystals, so as to give a quicker lather. The cold milling on milling rolls, of soap containing the large crystals above referred to, has long been accepted as the best procedure for high-grade department store tablets. It yields a product with crystals as fine as those of Bodman or Mills (2 to 4 microns) with each crystalline particle surrounded by jelly-phase material to give the quick lather of Mills process soap. However, cold-milled soaps are harder and less wasteful. Roller milling, however, has its limitations, which prevent getting crystals fine enough to produce transparency, welding properties, or the water-penetration of a true jelly (.1 to .5 micron). Even if such fine crystals were produced, roller milling does not provide the shearing compacting of the fine crystals necessary to force the crystals into contact and force the jelly-phase material into the voids between contacting crystals so as to yield a solid structure as hard as the crystalline phase material. To accomplish both the crystal size reduction and compacting of the present invention, the crystal reduction forces must be exerted on very thin layers of soap, measuring only 2 to 5 microns, in order to shear-off particles measuring .1 to .5 micron. Also, the shearing rate must be fast enough to produce fracture of these small crystals, in place of just rearrangement. Furthermore, the compacting of crystals which are smaller than visible light’s wave-length (6,000 A= .6 micron) without having these particles separated from each other by soft jelly-phase material, requires a shearing action under great pressure without at the same time producing localized overheating sufficient to cause crystal size reversion. Certain soap milling apparatus, for example, that known as the Stacom mill, develops crushing pressures which will reduce the size of the soap crystals below that of ordinary roller milling but in a much less efficient manner than in the present invention because much of the power required by such apparatus is not effectively utilized. The Stacom mill, moreover, does not provide the necessary shearing action under the required pressure to produce interlocking of the crystals of the crystal phase.

Both reduction of the soap crystals to an ultramicrocrystalline size and the application of intense shearing and compacting forces sufficient to cause interlocking of the fine crystals are required to produce a hard bar structure of ultramicrocrystalline soap. Ultra-fine crystals give bar soap better texture, transparency, toughness, and water-welding properties as well as rapid solubility to avoid overuse. Compactness gives soap a hardness which prevents disintegration into wasteful soap-dish flakes, sloughing off of undissolved material during the washing manipulation. That is to say, the greater solubility of the jelly phase will cause the bar to rapidly disintegrate if the crystals of the crystal phase are not interlocked.

In accordance with the present invention, the formulation employed for hard bar soaps is less critical because those animal fats, resin, vegetable and marine oils which yield the most soluble soaps and the type of lather desired, can be used without having a soft, wasteful jelly. The crystalline phase materials in various fatty acid soaps varies in hardness but substantially all such crystalline phase material is harder than necessary to give the required body to the bar, providing these crystals are interlocked, instead of being impacted in the foaming bar, but a much softer and more wasteful bar (Mills process).
Fig. 3 is an isometric detail of the feeding, shearing, compacting, aerating and discharging elements of the milling apparatus partly in section and having certain of the parts removed; Fig. 4 is a fragmentary vertical section through a band cooling device which may be employed in the device of Figs. 1 to 3; and Fig. 5 is a horizontal section taken on the line 5—5 of Fig. 4.

As shown in Fig. 1, the apparatus may include a band mill structure similar to a conventional band-saw and provided with driven band wheels 1 and 2. For propelling a suitable metal band member 3. The wheels may be mounted on driven shafts 4 provided with crowned rims 5 carrying the band 3. Plopper-type flake feeding devices 6 may supply the material to be processed to feed chambers later described although the feeders may be suitable pumps if the material being converted is pumpable.

The band wheels 1 and 2 are separated sufficiently to accommodate the feeding devices 6 and die slot members 7 and 8, each surrounding one half of the band. The members 7 and 8 may be jacketed or channeled as later described to distribute a coolant introduced at inlet 9 and removed at outlet 10. Hoppers 11 and 12 receive the material to be processed, and hoppers 13 and 14 receive and mix the products which have been processed. The hoppers 11, 12, 13 and 14 are shown as being open, but can be made air-tight and can be insulated, if necessary, to prevent loss of heat, volatile peroles, or moisture. A gas feeding or exhausting means is illustrated at 15 and 16, and may be used when desired to either augment or remove the entrained gas unavoidably brought into the apparatus with the fed material into hopper 11 or by the carrier band 3.

Fig. 3 illustrates in more detail the elements of the die slot members 7 and 8 and associated elements. The plopper 8 may include a worm 17 within a barrel 18. The worm 17 may be driven from any suitable variable speed source of power, not shown, connected to a shaft 19 so as to develop the proper hydraulic feeding pressure on the material in a feed chamber 20 of the die slot member 8. The die slot member 8 is made of a suitable metal, such as stainless steel, and is suitably constructed so as to contain a precisely machined slot 21 adjacent one face and extending longitudinally through the member. The slot 21 closely fits the band carrier 3. For example, on a model this slot was fitted to within 1/6 inch of the width of the carrier band 3 and to within 1/4 inch of the thickness, for a band dimension of approximately 3/4 inch x 3/4 inch. The band 3 was made of mild steel such as that used for band sawing wood. Special clearances for this slot, at certain critical points, will be explained later. Cover plate 22 forms an air-tight wall on one side of the slot 21, and is arranged so that by removal, the band 3 can be replaced and the various chambers in the slot member 8 cleaned or examined with ease. At the entrance of the band carrier 3 the slot member 8 is provided with a labyrinth seal 23 closely fitting the band carrier to prevent the escape of material fed into chamber 20, under pressure. The labyrinth seal providing clearance between the inner clamping strips in conjunction with the inward travel of band 3 prevents any leakage from chamber 20. Revolving disc guides 24, shown in Figs. 1 and 2, bear against one edge of the band 3 to center the band in slot 21 and thereby eliminate any abrasion points where bare metal parts have rubbing contact, thereby avoiding discoloration of the material being processed.

Feeder 6 is bolted in a suitable air tight manner to die member 8, for example, by flange 25 and delivers the material to be milled into the chamber 20 and into contact with the band 3. The die slot member 8 may have coolant channels 26 so as to provide external metal walls and metal walls surrounding the die slot 21 and the various chambers in the die slot member. Coolant connections 9 and 10 communicate with the coolant channels and the coolant channels may be provided with interconnections and suitable baffles, not shown, to provide for proper flow of coolant through the die slot member. Carrier band 3 bisects the chamber 20 so that its edge is presented to the incoming flow of compressed material. The hydraulic pressure of this material, in chamber 20, is equally divided on the flat surfaces of band 3 as well as the opposite edges thereof. This balancing the pressure on the band to allow the band to take the forces of the feed without being displaced in the die slot.

The clearance of slot 21 at the feed point 26 at the lower portion of chamber 20 may be increased and made slightly wedge shaped to facilitate adequate input to slot 21 but not sufficient to overfeed and cause the material to disgorge around band 3 at outlet 21 of the slot 21. The flat sections of the slot, such as 26 and 28, between small expansion chambers 29, will have various lengths according to the type of material being handled. The criterion is to determine by observation and test the maximum length which these flat sections may have in order that the material will not remain stationary on the walls of the slot, but will crawl along the surface thereof, for example, from one expansion chamber 29 to the next chamber during properly adjusted operation. In general, the last flat section 35 should be somewhat longer than the other flat sections so as to withstand its coating. These flat sections, such as section 28, in conjunction with the traveling carrier band constitute an improved counterpart of the “bite” between two mill rollers operating at a differential in surface speed.

The expansion chamber 29 preferably extend at an angle to the line of travel of band 3 that is, generally crosswise of the band, and this angle will vary according to the consistency of the soap or other material so that the bulged-out coating on the band 3, as it emerges from compression in a flat portion of the slot, is trimmed upon its entrance to the next flat portion of the slot and the trimmings travel along the expansion chamber 29. Under these conditions, the material in the expansion chamber does not cake but is kept in a turbulent condition and is redistributed over the surface of the band in the next flat slot portion. The proper number of flat slot zones, such as the portions 28, and expansion chambers 29, is dependent upon the properties of the particular material being processed.

The die slot member 8 is also preferably provided with one or more stripping chambers 30 interrupting the slot 21. The stripping chamber 30 contains a pair of stripping knives 31 secured to the cover plate 22 and engaging opposite sides of the band carrier 3 to remove the film of soap from the band carrier. The stripping chamber 30 is also connected with the gas conduit 15 by.
orifices 32 for introduction or withdrawal of gas or air. The functions of the stripping chamber will be discussed more in detail below. The soap or other material from the stripping chamber is then carried through the remainder of the slot 21 so as to pass through the additional expansion chambers 33 and the straight portion 34 of the slot. The milled material is then removed from the band carrier 3 by a pair of knives 33 as it emerges from the straight portion 36 of the slot.

Soap milling has heretofore involved the use of a series of sets of rollers each having two heavy mill rollers spaced approximately .001 in. apart to make a flake .008 inch thick. The rolls are driven so as to have their surfaces traveling in the same direction. Various speeds are employed but one roller usually has a surface speed of about 1,300 ft. per minute with the pick-up roller of the pair running approximately 7% to 8% faster, so as to develop a shear at the rate of approximately 100 ft. per minute on the soap passing through. This shear is exerted on a layer of soap approximately equal to the clearance between rollers, or .007 in., which is approximately 176 microns and represents about the lower practical limit of clearances for roller mills. It is an extremely inefficient operation for abrading particles to a size of say 10 microns, for milled soaps or 1/2 micron for transparent-phase soaps. It means handling, at heavy squeezing pressures (600 lbs. per square inch), a soap layer that is 17 to 350 times as thick as the particles being developed by shear and pressure.

The apparatus of the present invention has a "bite" that is much more efficient for either milled soaps or ultramicroncrystalline soaps. The apparatus is operated so that no material discharges around the band at the outlet 27, but instead all of the material is carried out as a coating on the band. The coating on the band is approximately .0008 inch in thickness. This has been found by test wherein 1 ounce of soap is carried out as a coating on an 1/8" inch band, coated on both sides, running 3,125 ft. per minute, every 21/2 hours. This means that soap at a density of approximately .70, which is 3.47 cubic inches to the ounce, is being spread over 22,300 square inches of band area, and therefore is approximately .0008 inch in thickness. This may vary up to .002 inch, depending on the soap moisture, but it usually runs at an approximate dimension of 2 to 5 microns. Transparent-phase soap requires much smaller particles than the figure of 10 microns used for milled soaps. It must be converted to 1 to 5 micron, depending on the material. The "bite" in the present apparatus, which works on a carrier band coating of 2 to 5 microns thickness, at a shearing differential in speed of 3,125 ft. per minute, efficiently reduces the material into particles which will measure 5 micron. The apparatus of the present invention employs substantially all of the input power in using the shearing action in a confined space under high compacting forces. In contrast, the majority of the work done in passing soap through a roller mill is wasted in working the mass of soap in a manner not reducing the particle size even so far as developing 10-micron particles is concerned, and such a mill is not at all suitable for producing the much smaller particles of the transparent-phase soaps, namely, 1 to .5 micron. The crushing pressure of the rollers is not an important factor, because the small particles escape being crushed at such large clearances.

There is no direct method of calculating the compacting and shearing forces in the present apparatus but it is apparent that they are very high as shown by the translucency of transparent and transparancy and hardness of the resulting soap. The translucency is an indication of a particle size less than 2 microns. A length of light and the hardness is an indication of intense compacting. These forces are produced by developing very high wedging pressures with the soap material itself as it is carried along by molecular adhesion through the slot 21. The wedging effectiveness of slot 21 depends upon the crowding or compacting of the material in the straight or flat sections, such as sections 26 and 28, by an overdose from the previous feed chamber 20 or expansion chambers 29. So long as the coating on band 3 is active, rather than set into a layer that polishes to a hardness that escapes the drag of the expansion chamber 25 or the close-fitting straight sections 25, this effective wedging action is developed all along the length of slot 21. It is the purpose of the stripping chamber 30 and its knives 31 to keep an inactive coating from developing. The number of stripping chambers, such as chamber 30, which must be employed as well as the distances between stripping and expansion chambers to keep the straight sections of the slot at their peak of effectiveness will vary with different materials. Interchangeable die slot members which are easy to mount in the apparatus can be provided so that the proper die slot member can be employed for a particular material or the die slot member can be made in sections to enable various combinations or sections containing straight sections and chambers to be employed.

The stripping knives 31 within the stripping chamber 30 remove all of the band coating and allow this stripped material to pass around the knives 31 and again feed onto the cleaned band as a new coating. The material in stripping chamber 30 may feed out as fast as it is stripped or it may pile up and develop a considerable hydraulic pressure, before the material will feed out as a new coating. In any case, since the soap films adhere to the metal band tenaciously, the stripping chamber 30 will operate at some developed pressure.

The stripping chamber 30 serves another purpose, namely, for the aeration or de-aeration of the material. Gas supply means 15 or 16 can be any suitable non-fouling arrangement of calibrated orifices 32, such as are described in my copending application, Serial No. 791,610, filed December 13, 1947, Figs. 7, 8, 9, 10, 17 and 18. A high gas pressure may be required with certain soaps, or especially synthetic detergent materials, and at the same time in relatively meager amounts, so that a minute, non-fouling, calibrated orifice means is required for orifices 32.

The conditions in the stripping chamber 30 are about the same as in the feed chamber 20 where the entrainment of gas normally takes place. However, certain soaps, such as milled soaps which are de-aerated and passed through small chambers, such as a microcrystalline state, may not entrain sufficient air in the feed chamber 20 alone to make them into a floating flake that can be integrated into a floating bar. Additional aeration may be accomplished in one or more stripping chambers 30, supplied with measured and pressure-regulated gas or air.

De-aeration, if desired, may be accomplished in one or more stripping chambers 30 by exhaust-
ing the accumulated gases therefrom through non-fouling, extremely fine orifices 32 with a vacuum pump attached to line 16. The stripping chambers 30 or the preceding orifices of the non-fouling orifices 32 are so arranged that the gas issuing from the stripping chamber 33 is moderated by their elongation, so that no gassing takes place at the discharge point 27. A test model, with dimensions described later, gave such results on several of the soap tested. If the straight sections 34 and 36 are elongated, a point will be reached, as is the case with any labyrinth seal, where no material can make its way out except as a coating on the band, and this is the preferable operation.

The final stripping knives 33 are shown in Fig. 3 as not being enclosed, so as to facilitate adjustment and renewal of the knife edges. A dust cover only will usually be employed and the knives can be constructed of a solid mounting 25 which takes a renewable blade. The test model, described later, used a flexible safety razor blade in a suitable mounting and was very satisfactory on a 1 1/4 inch band 2. By staggering the blades so that they contact spaced portions of the band and adjusting the tension on the band, a resilient arrangement is provided which minimizes the wear and abrasion of either the knife blade or the carrier band and provides a delicate running contact efficiently removing the coating from the band.

As a specific example, approximately 4 ounces of 17% moisture soap, made by the process of Mills Patent No. 2,205,594 was processed in 10 minutes with the carrier band being properly coated and no gassing around the band occurred at exit 27, using a 1 1/2 inch band (3), at 3,125, ft. per minute.

Excessive feeding pressure on chamber 20 causes soap to disgorge around the band 3 at exit 27 in addition to that adhered to the band. This is not a desirable operating condition, since best results are obtained when all of the discharge is carried out of the die slot by adherence to the band 3. The material will not be uniform in texture if part of the material is disgorge around the band, since disgorge material is not sheared or compacted to the same extent as that which is coated on the band 3. Furthermore, excessive power is consumed when disgorge takes place; due to overheating and jamming of the band. Properly adjusted feed will give a very uniform power demand.

Soap should be softened by heating, if possible, to feed at hydraulic pressure of 5 to 15 lbs. per square inch, within the chamber 20. Cooler and harder soap will, of course, feed at higher hydraulic pressures, but these pressures may be avoided by softening the soap with heat before feeding, without incurring any loss in milling effectiveness, since the cooling of the slot 21 and the band 3 restores hardness to the soap before it is actually milled. Thus, good hard milling will occur despite a deliberate tendency of the soap to be fed into the chamber 20. Reversion of crystal structure in the soap does not usually occur at the slightly elevated temperatures needed to get a good feed at 5 to 15 lbs. gauge. This lower feed pressure shortens the labyrinth gland 23 needed to seal the feed chamber at the blade entrance zone and places less strain on the band 3.

In addition to the cooling of the walls of the band as described above, the band 3 may also be cooled. Some cooling of the band 3 can be accomplished by such expedients as supplying a coolant to the rims of the band wheels 1 and 2 through channels in the wheels and shafts 4 or by directing an air blast against the band before it enters the feeding chamber 20. However, it is advantageous in many operations to drastically cool the band. This may be accomplished by surrounding the band with cooled metal walls, for example, as shown in Figs. 4 and 5. As shown in these figures, cooling chambers 31 made for example of sheet metal, may be positioned on opposite sides of the band 3. Any suitable coolant may be introduced through inlet pipes 38 and withdrawn through outlet pipes 39. That is to say, the cooling chambers may be the evaporator of a refrigeration system so as to produce any low temperature desired. The cooling chambers 31 may replace one of the die slot members 7 or 8 in Figs. 1 or 2 or may be positioned adjacent the entrance of the band into a feeding chamber on the same straight section of the band 3 as a die slot member.

Even though die slot members 7 or 8, wherein conversion takes place, are cooled and the temperature of the soap coatings on the walls of these slots is well regulated, it is usually desirable to cool the band 3 itself. The thin film which carries through on the band despite the congestion in the slot and chambers absorbs the heating and compacting energies more than the thicker coatings on the walls of the slot and the band 3 itself produces a major temperature regulating effect on this thin active film.

Test runs, for example, on 17% moisture soap fed at 100°F. both with and without band cooling showed that cooling of the band more than doubled the conversion attained in the absence of such cooling. This improvement in conversion also provides a verification of the fact that the conversion of soft uncompact soap to extremely hard compact structures is not a moisture-reduction effect. Some moisture loss can be expected from a coating only 2 microns thick riding on a warm carrier band at high speed. Only nominal evaporation can occur in the short exposed zone around knives 35 if not enclosed by a vapor proof cover and this nominal amount will be greater with a warm band than with a cold band, and therefore it is impossible to explain the much greater hardness and transparency gained when a colder carrier band is employed except by conversion of the soap structure. The moisture reduction in the totally enclosable system can be held to a negligible point and the great changes in hardness and transparency are obtained in tests run at 20% moisture, 17% moisture or 13% moisture and lower for the soap fed into the apparatus.

Band speed is an important factor governing the rate of feed, the particle size developed, the hardness resulting from the compacting action, the amount of aeration, and the thickness of the film or coating on the band 3. Band speeds ranging from approximately 3,000 to 8,000 ft. per minute are contemplated, a suitable speed being about 6,000 ft. per minute. It will be apparent that the greater the speed the greater the capacity of the apparatus. Band width also increases the capacity proportionately, and the
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The greatest practical band width consistent with such limiting factors as problems of stripping knife performance, traction of driving pulleys, etc., will usually be employed. Both the down-travel of the band and the up-travel on the straight-away sections, if the structure is vertical, may be utilized. It is desirable to drive both the upper and lower band pulleys 1 and 2 to get sufficient traction as well as to hold the band 3 in line so as to rub the edge of the slot 21. The pulleys may be crowned in the conventional manner used for band-saws. A horizontal structure also works satisfactorily and offers more accessibility in a battery unit, made up of several bands operating on one shaft.

The present process involves the intensification of shearing and compacting of materials by developing a very thin coating of the input material on the large surface afforded by a high speed carrier band and the shearing of this coating against the coating developed on the surface of a close fitting die slot through which the carrier band is caused to travel. The coatings are subjected to large wedging forces developed by the momentum of the coating on the band due to the high speed of the carrier band and the crowding of the slot with input material moved into the die slot and carried from stage to stage by the molecular forces of surface adhesion which are of a very high degree.

Soap introduced into the feed chamber can be made to adhere to the carrier band by regulating the thickness with heat, and by the hydraulic pressure of the fed material in the chamber 20 forcing the soap against the surfaces of the carrier band 3 as it passes through the feed chamber. Melted soap requires little or no pressure to make it adhere to the carrier band. Soap heated as high as possible without reversion of the crystal formation will feed usually at 5 to 10 lbs. pressure in the feed chamber. The lower the feed pressure, the smaller the labyrinth 23 need be around the carrier band at the entrance to the slot, and the less power required by the feeding means 6. Special texture and material considerations may require less heat and more hydraulic pressure to coat the carrier band satisfactorily. There is no mechanical limitation within reason on this feed pressure. By making the chamber 20 smaller the drag on the carrier band 3 is minimized. The edge thrust on the carrier band may be offset by rolling guides 24 outside the feed chamber 20 so that the cantilever beam strength of the blade edgewise can withstand large hydraulic pressures in the chamber 20, if necessary. The labyrinth seal at the band entrance can be made extensive enough to step down the pressure and thereby seal and contain a material being fed at very high pressure, if necessary.

Once the carrier band is coated, that coating will carry into the die slot entrance stages, due to molecular forces of surface adhesion phenomena, and this coating will crowd the material ahead of it through the die slot 21, stage by stage, so as to crowd against the carrier band within the slot and be carried through by the band, despite the shearing and compacting strength of the soap structure which resists it. Since the clearance are small and the gap is kept closed by the coatings themselves, there is developed a tearing and compacting action on even the smallest of the crystals. The transformation of the soap texture in tests of the model indicates that particles of approximately 3-3.4 micron can be developed.

If the initial coating on the surfaces of the die slot straight section 28, etc., is allowed to cake and become polished by the fast moving coated band 3, the shearing and compacting action on polished surfaces only and the balance of the material back of these surfaces escapes attrition. Also the throughput becomes very small. Such a condition is prevented by three factors.

The thickness of the coating on the band 3 may be controlled at the entrance to the slot than the thickness farther along the slot, thereby crowding the slot from the feed end all along and nearly to the exit 27. This crowding disrupts the coatings on both the surfaces of the slot 21 and the surface of the band 3. This may be accomplished by slitting the entrance section of the slot, in contrast to the rest of the slot length, as well as by the hydraulic pressure in the feed chambers 29 or stripping chambers 30.

The expansion chambers 29 allow the compacted coating on the carrier band to bulge at the entrance into such chambers and thus a portion of the coating is trimmed off and churned along the slanted expansion chamber. This portion is forced back again onto the carrier band 3 as a new layer on the coating, thus disrupting the balance of the coating by crowding and cross flow.

The knives in the stripping chambers 30 positively remove the coating from the carrier band 3 by mechanical means. The crowding action of the remnant material again develops a hydraulic pressure in the enclosed stripping chamber 30 to cause the carrier band 3 to be recoated for the next pass into the slot 21.

The number and spacing of the expansion chambers 29 and stripping chambers 30 is determined by the nature of the material and its consistency at the temperatures required to preserve the crystallization. On soap made according to Mills U. S. Patent No. 2,295,594, (Ivory) excellent conversion to the transparent phase was obtained by having 13 expansion chambers, followed by a stripping chamber, and 13 more expansion chambers, while on soap made according to Bodman Patent No. 2,215,539, (Swan) two interposed stripping chambers with fewer expansion chambers worked better.

The die slot member 3 can be opened and by carefully examining the material in the slot 21 and in the expansion chambers 29, it can be determined whether the coating is being sufficiently disturbed, or whether it is tending to cake and clog such that the action of the band 3 is a surface polishing action only. Under balanced operation when material is not being disgorged from the die exit 27, the amount of power required is a useful index in arranging the number and spacings of the expansion chambers 29 and the stripping chambers 30. If no more power is required when the number of expansion chambers 29 is increased, their effectiveness in diminishing and stripping chamber 30 should be interposed to strip and recoat the carrier band 3.

The carrier band 3 itself may be made of metal alloys which give better coating adherence with. Also, the flat surface of the carrier band 3 may be shaped to increase its pick up effect. These markings may, however, produce rapid wear of the stripping knives to shorten the life of the knife blade, so that if good adhesion can be gained by hydraulic feed pressure alone, that means is preferable.
In addition to the fact that crystal or texture refinement is carried to a greater extent and accomplished at higher efficiency, should the process, in any prior art process, the resulting ultra-microcrystalline soap can be uniformly aerated during the process to make it float. This aeration is done on extremely thin coatings so that the dispersion of air or gas is uniform. The shearing and compacting, which ordinarily might be expected to squeeze out all of the gas cells, cannot do so under the pressure developed in the slot by the rapidly moving band. The heating acts as a gas dispersion means and the gas cells are retained despite the compacting. Should the speed of the carrier band not be sufficient, or the hydraulic pressure on the feed be too great, or the temperature of the soap too high to permit entrainment of all the air which is necessary at the entrance to the feed chamber, then additional aeration with exact conditions for regulation is accomplished in the totally enclosed stripping chambers 30 along the length of the member 8.

The feeding of this air into the chamber is done at the most effective pressure by the mounting of feeding orifices 32 with non-fouling openings, such as those disclosed in my copending application Serial No. 791,610, supra, wherein the orifice calibration is regulated by a restricting pin, and the orifice around the pin is kept clean and unplugged by imparting motion to this pin in relation to the orifice by means of any suitable reciprocating drive. Such means can be used to introduce small regulated amounts of gas at high pressure as is needed. Gas introduced in a regulated manner to the stripping chamber 30 will be incorporated in the coating in addition to any gas entrained with the feed in chamber 20 until the correct finished density is reached to make a floating soap chip which later can be integrated into a floating bar by several processes, such as those disclosed in my copending application Serial No. 791,610, supra, or Serial No. 128,693, filed November 23, 1949, which latter process involves reducing the material to granular powdered form and then compacting the same into bars.

De-aeration is accomplished by reversing the process. Whatever air is unavoidably entrained with the feed may be removed by placing a vacuum pump in communication with a stripping chamber or chambers 30 in a suitable arrangement in the entrance section of the member 8. Complete de-aeration depends upon the exposure of very thin films of aerated soap to the exhausted stripping chamber 30 and also upon the stripping of these films from the carrier band 3. Air which has been impacted in an ultra-microcrystalline, rubbery soap film is released mechanically by the spreading and stripping action of the band 3 in the slot 21 and knives 31. This enables the air to be removed from the resulting soap flake when it is finally stripped from the carrier band 3. For de-aeration, non-fouling and extremely small restricted orifices 32 may be employed to prevent clogging of the gas withdrawal channels or removal of soap with the gas. Such orifices are afforded in accordance with my copending application Serial No. 791,610, referred to above.

Soap crystal reduction and compacting of the soap is best accomplished at the maximum carrier band speed possible within practical limits and with minimum coating thickness on the surfaces of the slot 21 and on the band 3. In using the present process for developing a desired soap flake with respect to dimensions and glossy finish, it is preferable to use two stages in the operation. These stages may be a crystal reduction and compacting stage using a very high speed band and close clearances and then a second stage for flake forming using a much slower speed and larger clearances in the discharge end of the slot 21. The latter stage may be conducted in apparatus in which the slot clearance is gradually increased. A stripping chamber 30 may then be employed to produce a new coating on the band 3 which is thicker than the desired flake. The last portion of the die slot may then be employed to reduce the thickness of the coating and polish the surface thereof rather than to disrupt the coating. The angle of knife 35, providing for the removal of the flake without causing it to be upset or wrinkled, also may be made smaller in the latter stage than is desirable for a high speed conversion operation. It is extremely difficult to avoid wrinkling of the flake unless the coating is increased to at least 0.001 inch.

One very advantageous factor in the finishing of flakes by the present process is the refined texture imparted in the shearing and compacting stage. The soap becomes waxy after processing through the die slot member 8 and does not resist removal from the carrier band 3 by the knives 35. The surface of the flake next to the band 3 as well as the surface next to the slot 21 are both ironed to a sheen not possible heretofore, and the waxy and tough character of the refined soap leaves it dust-free. The flakes do not easily break down into "fines" because of their rubbery nature.

Another important factor in the present process is the close temperature regulation which may be obtained. Heat is rapidly conducted through the walls surrounding the die slot and the various chambers therein and such walls may be cooled throughout their length by any suitable cooling medium. It is apparent that the channels for the cooling medium may be baffled or sectionalized so as to enable any desired temperature gradient to be maintained. The die slot. Also, the band may also be cooled as described above. Since the soap or other material being milled is formed into very thin coatings on the band and on the surfaces of the die slot and these coatings are kept in a turbulent condition, heat may be rapidly extracted therefrom.

While the present process is primarily a conversion operation, it lends itself to producing desirable solidification effects by heat regulation. For example, it is known that intense stirring during cooling of soap and other similar materials to produce crystals results in a different texture than cooling without agitation. Thus ultra-fine crystals can be produced by feeding the material at a temperature just above its solidification temperature and cooling and simultaneously working the material in the die slot. By simultaneously introducing air or other gas, aeration during solidification can also be accomplished. The process can therefore be employed as an ultra-fine crystallizer for such materials as soap, ice-cream, shortening, etc., either with or without aeration. Other materials such as face creams, synthetic detergents or greases, etc., in semisolid form can be subjected to a conversion operation or they can be
soldified in ultra-fine crystalline form with advantageous results.

The extent of intensification of the shearing and compacting effects by this process is indicated in the following table, comparing the essential factors of conventional soap milling, done with water-cooled rollers, with the present process:

<table>
<thead>
<tr>
<th>Thickness of soap flake sheared and discharged from process</th>
<th>rollers</th>
<th>present</th>
<th>comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.006&quot;*</td>
<td>0.0006&quot;</td>
<td></td>
<td>yes</td>
</tr>
<tr>
<td>Number of pipes through &quot;bite&quot;</td>
<td>12</td>
<td>25-50</td>
<td>16-4 times</td>
</tr>
<tr>
<td>Distance of flake travel between passers</td>
<td>2/&quot;</td>
<td>11/8&quot;</td>
<td>yes</td>
</tr>
<tr>
<td>Shearing rate in &quot;bite&quot;</td>
<td>100 fl. per min.</td>
<td>500-4,000 fl. per min.</td>
<td>50-60 times</td>
</tr>
</tbody>
</table>

The following data on test runs of a model of this process will serve to evaluate its great effectiveness as a crystal reducer and compactor, despite the presence of air cells:

Material fed: 17°% moisture, aerated. A test process of un-aerated, 13°% moisture milled soap having an 80°% tallow and 20°% coconut oil base (Camay) yielded a product with texture comparable to double-milling of aerated, 17°% moisture Mills-process soap (Ivory). Soap was fed at 100° F. and discharged at 120° to 130° F., and the products were run through a carrier and extruded and some additional material to make the finished flakes float.

This new process is particularly applicable to the ultra-refinement of soaps. However, many other applications are possible using the same general principles which govern its effectiveness on soap. For example, tooth pastes can be reduced to a colloidal and de-aerated state in this band mill.

Shaving creams or powders, made from microcrystalline soap bases, can be refined into products with better lathering properties or better textures. Also, face creams of superior texture can be obtained.

Synthetic detergent chips, both those containing the salts incident to their chemical processing and without these salts, can be aerated and compacted. Band cooling is particularly important when low melting point synthetic detergent bases are being aerated. Tests on a sulfated mono-glyceride synthetic without band cooling gave poor conversion as measured by transparency and hardness of the processed material. However, with a cooled band entering a cooled die slot chamber the conversion was very pronounced.

Translucency of a high order developed and the converted material was 3 to 7 times harder than the original material. Subsequent integration into aerated bars may be accomplished by the process disclosed in my co-pending application, Serial No. 129,093, filed November 23, 1949, now Patent No. 2,554,956 issued April 29, 1951.

Special greases, requiring a blend of converted soaps with petroleum products, can be benefited by the present process, this being a typical application in which the soap ingredient is assisting another ingredient with less molecular surface adhesion to metal.

It will be apparent that the present apparatus constitutes an efficient collision dispersion mill useful for many applications where intense shearing and compacting forces are required, particularly where controlled temperature conditions are also required.

I claim:

1. The method of converting soap by mechi-
2,619,680

17. The method of mechanically refining and aerating soap and similar material, which comprises: forming the material into a plurality of film-like sheets not more than on the order of one sixty-fourth of an inch thick; passing the ribbon of soap lengthwise through a shearing and compacting zone; imparting motion to the surface layer of soap at one side of the ribbon as it moves through said zone, relative to but parallel with the surface layer of soap at the opposite side of the ribbon, to effect subdividing and compacting of the individual particles in the mass of the soap comprising said ribbon; successively and repeatedly turbulently spreading and congesting the ribbon of soap as it moves through the shearing and compacting zone to thereby intensify the conversion; and maintaining the temperature of the soap undergoing conversion below the crystalline reversion point of the soap.

2. The method of mechanically refining and aerating soap and similar material, which comprises: forming the material into a plurality of film-like sheets not more than on the order of one sixty-fourth of an inch thick; force feeding a plurality of such sheets edgewise in one direction through a shearing and compacting zone with the sheets in side-by-side parallel relationship; introducing air into said zone; in said zone imparting high speed motion to the surface layer of the material at the inner sides of the sheets relative to and substantially parallel with the surface layers of the material at the outer sides of the sheets, and simultaneously effecting high speed internal churning of the mass of material comprising each of said sheets so as to effect subdividing and compacting of particles throughout the mass of material comprising each sheet, and occlusion and fine dispersion of air throughout the mass of material comprising the sheets; and maintaining the temperature of the material undergoing refinement below the crystalline reversion point of the material.

3. An attrition mill, comprising: an endless movable metal band having opposite substantially flat sides; means constraining the band for lengthwise travel of a stretch thereof in a straight line path; a stationary member at each side of said straight stretch of the band having a milling surface spaced slightly from the adjacent flat side of the band; means for feeding material to be milled edgewise against the band and into said spaces between said surfaces of the stationary members and the sides of the band; and means for driving said band at high speed.

4. The apparatus set forth in claim 3 further characterized by the fact that said stationary milling members each have a series of spaced apart congestion grooves therein opening to and extending across their milling surfaces generally crosswise of the path of travel of the movable milling element.

5. An attrition mill, comprising: an endless movable band constrained to lengthwise travel in one direction; means forming a milling chamber through which the band travels, said chamber having walls embracing the stretch of the band traveling through the chamber and provided with elongated milling surfaces between which the band moves with very close clearance at each side, which clearances together constitute the straight stretch of the mill, said chamber having an inlet positioned to direct material against the edge of the band and an outlet downstream therefrom with respect to band movement; means for forcibly feeding material to be milled into the milling chamber through the inlet thereof to fill said attrition zone and means for driving the band at high speed in a direction to cause it to travel through the inlet of the milling chamber toward the outlet thereof and whereby the travel of the band and the force feeding the material into the milling chamber cause to effect movement of the material through the attrition zone and to subject the material to intense shearing and compacting forces before it reaches the outlet of the milling chamber.

6. The attrition mill of claim 5 further characterized by the fact that said chamber walls have grooves therein opening to their milling surfaces and extending obliquely across said surfaces.

7. The attrition mill of claim 5 further characterized by the provision ofscraper means acting upon the band as it leaves the milling chamber for removing milled material from the band.

8. The attrition mill of claim 5 further characterized by the fact that said endless band is relatively thin and flexible, has flat opposite sides, and is constrained to travel with a stretch thereof moving in a straight line path; and further by the fact that the milling surfaces between which the band travels are flat and parallel with the stretch of the band, said surfaces having a length several times the width of the band so that particles in the mass of material moving through said attrition zone are subjected to shearing and compacting forces a plurality of times before reaching the outlet of said chamber.

9. An attrition mill, comprising: an endless movable metal band; means constraining the band for lengthwise travel along a straight line path; means defining an attrition chamber having side walls spaced apart a distance slightly greater than the thickness of the band; means mounting said attrition chamber across said straight line path of the band with the band extending through the chamber and positioned between the side walls thereof; means for feeding material to be milled into said chamber; means for driving said band at high speed; and means for cooling the band.

10. Milling apparatus of the character described, comprising: a flexible carrier band; means mounting the band for movement thereof along a straight line path; a movable chamber embracing the band as it travels along said straight line path, said milling chamber being closed except for inlet and outlet openings in opposite walls thereof through which the band passes, and an inlet opening for the material to be milled; the interior of said chamber being shaped to provide a feeding zone communicated with the inlet opening and a milling zone, the walls of the feeding zone being spaced from the edges as well as the sides of the band, and the milling zone having side walls closely fitting the opposite sides of the band; and means for feeding the material to be milled under pressure into said feeding zone.

11. The method of mechanically refining solidified soap to raise its viscosity and at the same time increase its solubility, which comprises: moving a pair of coating wall surfaces across one another with substantial areas thereof at all times in opposition and substantially uniformly spaced apart a distance on the order of not more than one sixty-fourth of an inch to define a thin shearing and compacting zone having relatively moving walls of substantial area; applying pressure upon the soap in the solidified state and thereby forcing the soap as a film-
like sheet into the shearing and compacting zone with the pressure on the soap sufficient to have it fill said zone; by said pressure and the relative movement between the opposed wall surfaces forcing the film like sheet of material through and from the shearing and compacting zone and thereby effecting relative movement between that portion of the film like sheet of soap contiguous to one of the wall surfaces and that portion thereof contiguous to the other wall surface; so regulating the speed at which the opposing wall surfaces move with respect to one another that said relative movement between the portions of the film like sheet of soap at the opposite sides thereof effects intensified shearing and compacting of the soap manifested by increased viscosity and solubility; and maintaining the temperature of the soap undergoing refinement below the crystal line reversion point of the soap.

12. The method of mechanically refining solidified soap and similar plasticizable material which is capable of being softened by heat and has a crystalline phase capable of being broken down to an ultra-microcrystalline particle size, which comprises: moving a pair of coacting wall surfaces across one another at a speed of at least on the order of 3000 feet per minute with substantial areas of the wall surfaces at all times in opposition and substantially uniformly spaced apart a small fraction of an inch to define a thin shearing and compacting zone having relatively moving walls of substantial area; applying pressure upon the material in its solidified state and thereby forcing the same as a film-like sheet into the shearing and compacting zone with the pressure upon the material sufficient to have it completely fill said zone; by said pressure in coaction with the relative movement between the opposed wall surfaces forcing the film-like sheet of material through and from the shearing and compacting zone and thereby effecting relative movement between that portion of the material at one side of the film-like sheet and that portion thereof at the other side of the sheet, which relative movement effects an intensified shearing and compacting of the material; and maintaining the temperature of the material undergoing refinement below the crystalline reversion point of the material.

13. The method of claim 11 further characterized by feeding an ingredient compatible with soap into the shearing and compacting zone along with the soap.

14. The method of mechanically refining solidified soap and similar plasticizable material which is capable of being softened by heat and has a crystalline phase capable of being broken down to an ultra-microcrystalline particle size, which comprises: passing a carrier element having opposite surfaces of substantial area between a pair of facing wall surfaces which also have substantial area, with a substantial area of each of said surfaces of the carrier element at all times opposite and substantially uniformly spaced from the adjacent one of said pair of facing wall surfaces a distance on the order of not more than one sixty-fourth of an inch to define a pair of side-by-side thin shearing and compacting zones each having relatively moving walls of substantial area; applying pressure upon the material in its solidified state and thereby forcing the same as film-like sheets simultaneously into both shearing and compacting zones with the pressure upon the material sufficient to have it completely fill both zones; by said pressure and the movement of the material leaves the shearing and compacting sheet of material through and from the shearing and compacting zones and thereby effecting relative movement between the surface layers of material at opposite sides of each film-like sheet, which relative movement effects an intensified shearing and compacting of the material; and maintaining the temperature of the material undergoing refinement below the crystalline reversion point of the material.

15. The method of claim 14 further characterized by the step of removing the material adhering to the surfaces of the carrier element as they pass from between the pair of facing wall surfaces.

16. The method of claim 14 further characterized by the fact that the maintenance of the temperature of the material undergoing refinement is effected by cooling the carrier element outside the shearing and compacting zones.

17. The method of claim 14 further characterized by the step of balancing the speed of the carrier element and the pressure upon the material that the material leaves the shearing and compacting zones along with and substantially at the speed of the carrier element.

18. Soap which has been mechanically refined by the method set forth in claim 11.

DONALD E. MARSHALL.

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The following references are of record in the file of this patent:

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<table>
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