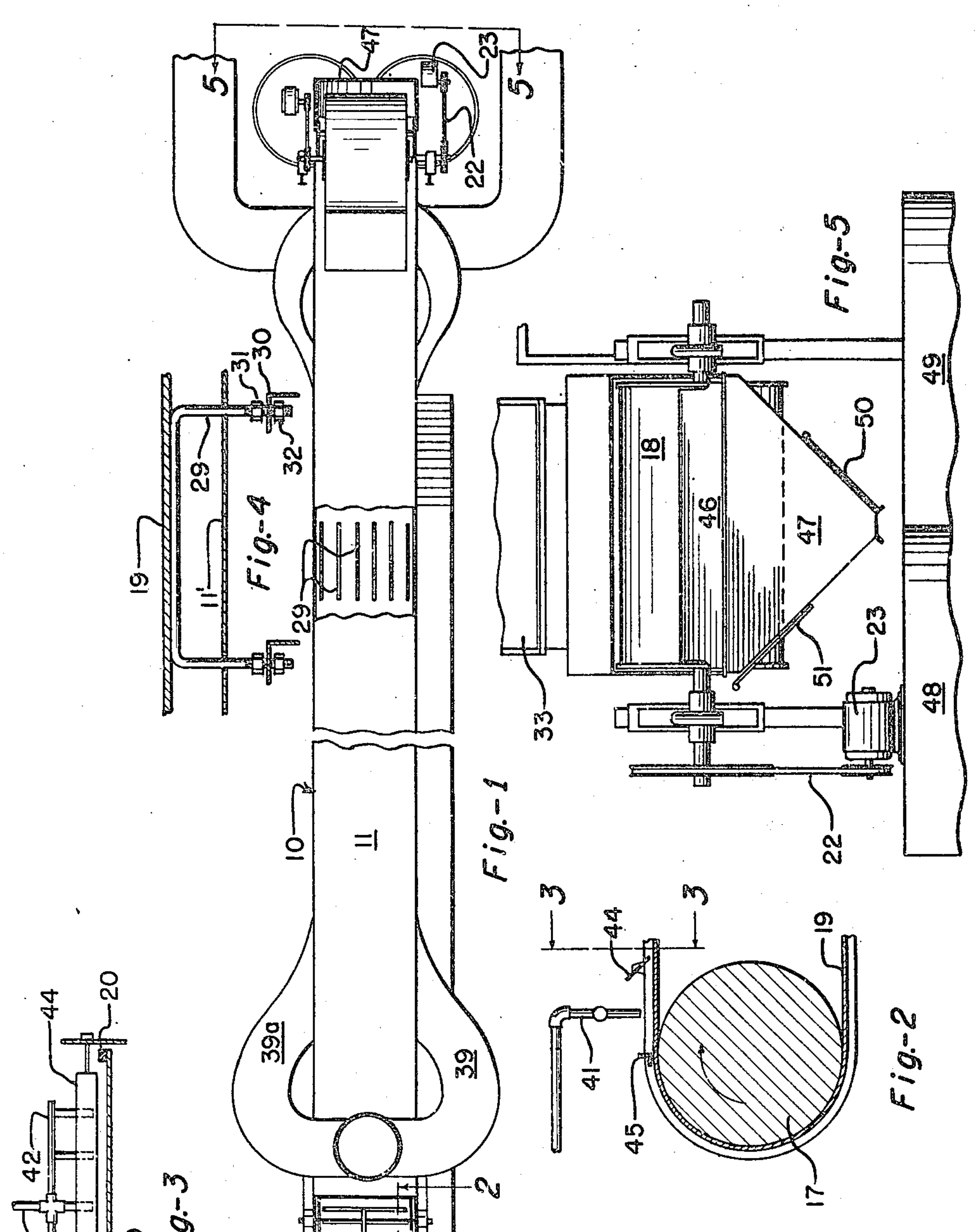
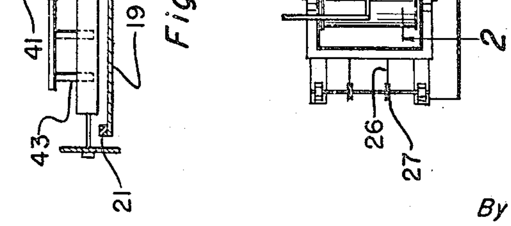
Dec. 20, 1949 A. A. BONDI 2,491,641

PRODUCTION OF AN ALUMINUM BASE GREASE CONTAINING ORGANIC AMINES

Filed June 27, 1946 3 Sheets-Sheet 1





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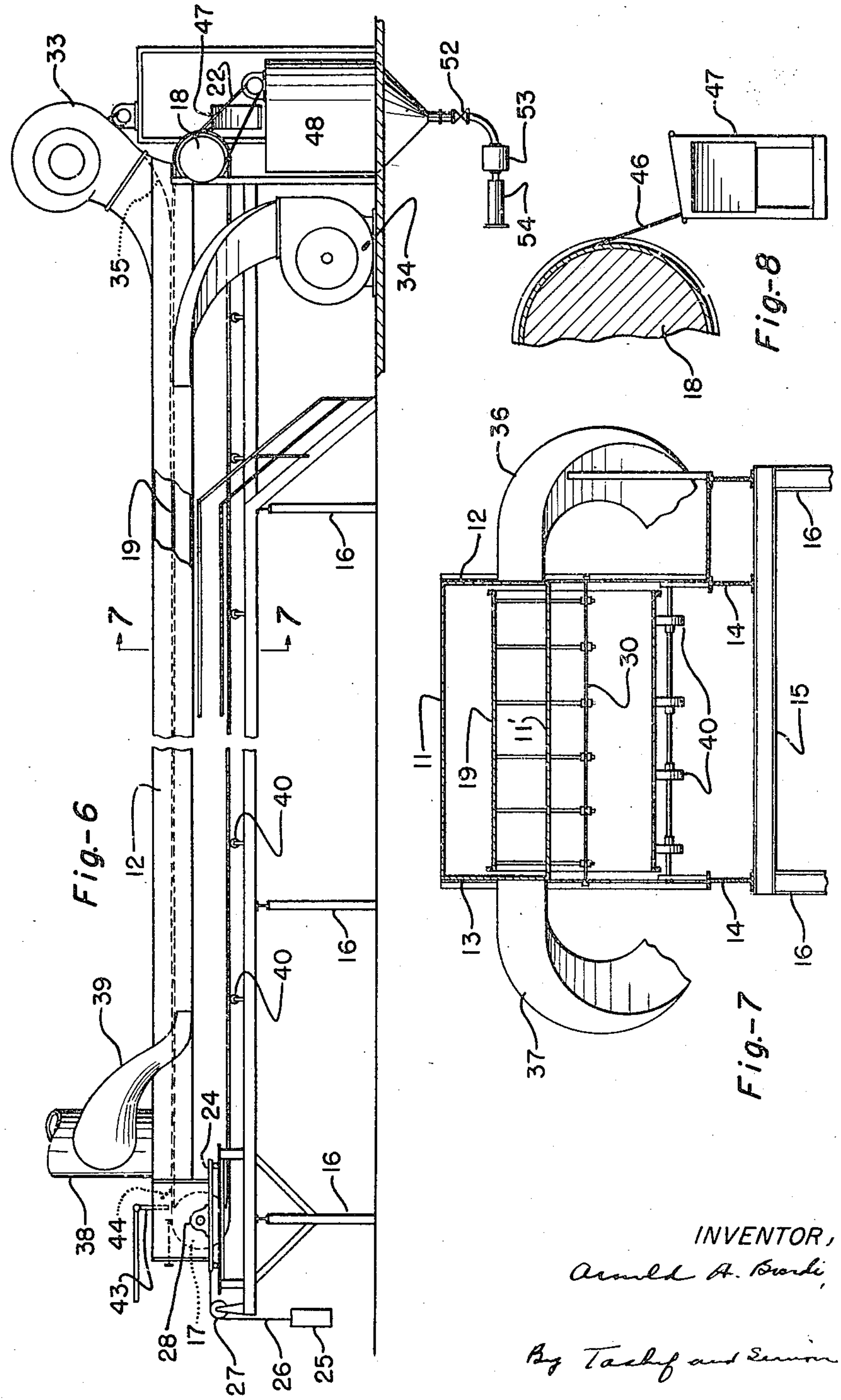

Fig-6

Fig-7

Fig-8

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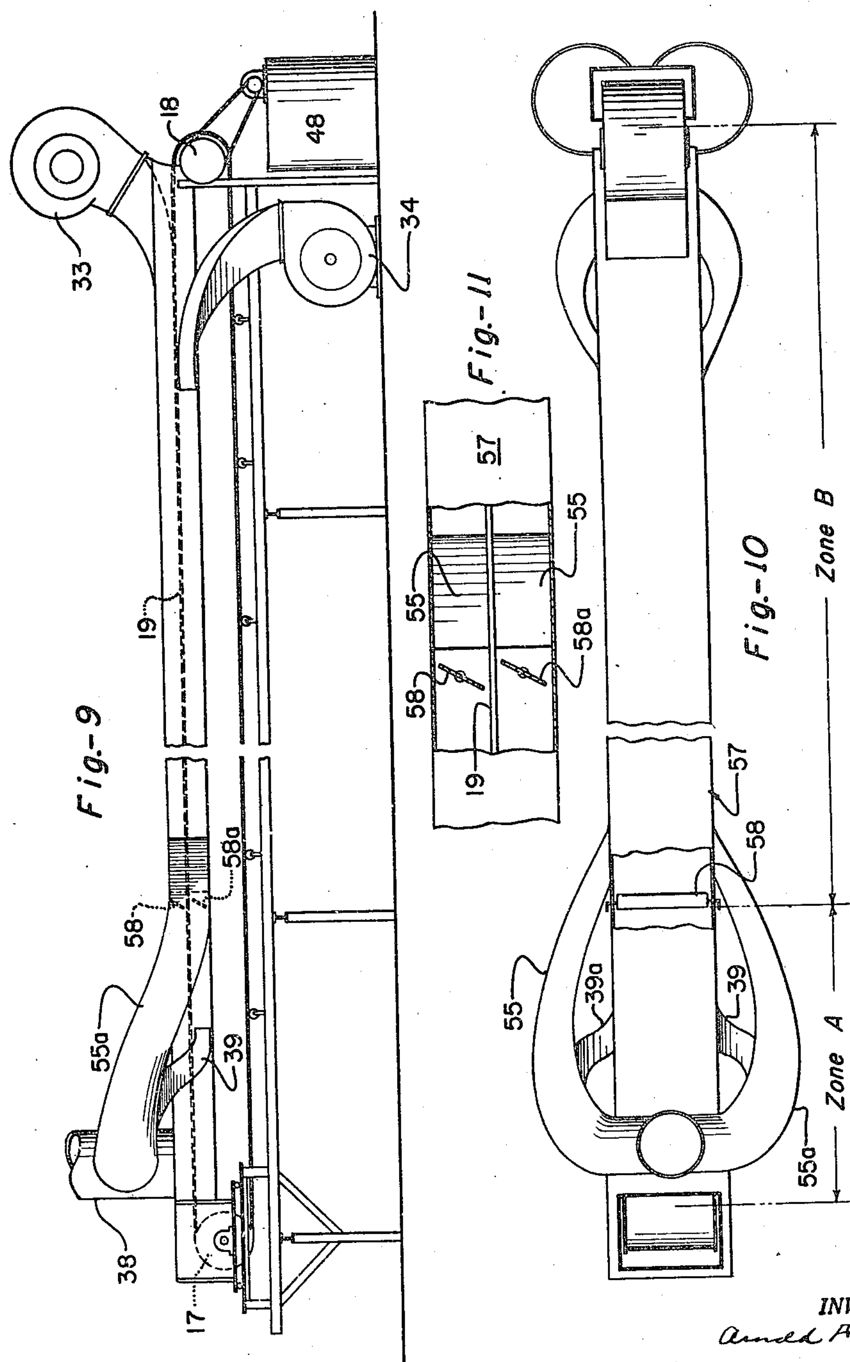

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2,491,641

PRODUCTION OF AN ALUMINUM BASE GREASE CONTAINING ORGANIC AMINES

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Application June 27, 1946, Serial No. 679,727

9 Claims. (Cl. 252—35)

The present invention relates to the production of lubricating greases containing aluminum soap of the higher fatty acids and particularly aluminum stearate, said grease also having present an aliphatic amine which functions under the conditions under which the grease is cooled to produce a smooth homogeneous grease gel which is non-bleeding.

In accordance with the present invention, a non-bleeding and non-graining aluminum base grease, that is a grease with a buttery texture is produced by incorporating in the aluminum base grease from about .05 to about .3% of an aliphatic amine and feeding the grease in a fluid condition in a relatively thin layer on to a heat conducting surface which passes through a single or plurality of cooling zones, the grease while being moved being maintained in a quiescent state and being subject to the continued action of a cooling medium, as for example air, said grease being cooled in a single continuous operation to preferably below the transition point of the grease in a time period of less than one hour and usually in a time period varying from three minutes to fifteen or twenty minutes, there being produced a smooth homogeneous grease gel.

It is desired to point out that this method of cooling contrasts with the prior art method of cooling an aluminum base grease, said prior art method comprising cooling the greases in relatively thick layers in a pan or cooling the grease in a helical screw conveyor where the grease is subjected to a shearing stress.

In accordance with the present invention, the aluminum base grease containing an aliphatic amine as an inhibitor of crystallization is fed to a steel belt or the like so as to form a relatively thin continuous layer on the steel belt, and is there subjected to a current of air or other cooling medium so that the grease while being carried along with the belt is cooled in a uniform fashion free from shearing stress.

In accordance with the present invention, there is provided an aluminum soap grease which has the desired consistency, and this is obtained using less aluminum soap than has been customary in the prior art. Previously, to obtain the desired consistency, it was necessary to use 6.5% to 7.5% of aluminum distearate or an equivalent fatty acid soap to obtain a grease having a worked penetration of 330 to 350. In contrast thereto, using the present invention, it is only necessary to use 5½% to 6% of the aluminum stearate or an equivalent amount of an aluminum soap of any of the higher fatty acids or other acids herein set forth. While the present invention has the advantages set forth, higher amounts than 5½% or 6% may be used if desired, but are not usually necessary.

Further, it is desired to point out that in order to produce an aluminum soap grease of the proper consistency and which was non-bleeding, a certain fraction of the total aluminum soap content comprises the di-salt of 12-hydroxy stearic acid or 12-hydroxy stearate. This is quite an expensive ingredient and may be dispensed with employing the present invention, and yet there will be produced a smooth non-bleeding grease. However, in carrying out the invention, the total soap content may comprise a small amount of the 12-hydroxy stearic acid.

It is also desired to point out that in the preferred form of the invention the mineral oil used in producing the grease has a low to medium viscosity range, as for example 100 to 2000 S. S. U., and when a mineral oil having such a viscosity is used in making greases containing an aluminum soap of the fatty acids of the character herein set forth or of any of the acids as herein set forth, said fatty acids being preferably those containing 14 to 24 carbon atoms in a molecule, it is exceedingly difficult to obtain a non-grainy grease chilling from the fluid state to a solid or semi-solid state in less than one hour.

In accordance with the present invention, an aliphatic amine is incorporated in greases made with these low to medium viscosity range oils, and the greases may be chilled to a non-bleeding smooth coherent gel in much less time than one hour, as for example in from 3 minutes to 40 minutes, and usually 3 minutes to 20 minutes when processed in accordance with the present disclosure.

While the primary object of the present invention is to produce an aluminum soap grease in accordance with the procedure above outlined and to produce grease having the advantages above set forth, some of the most important objects will be set forth.

It is one of the objects of the present invention to provide a method of cooling an aluminum soap grease such as a lubricating grease containing an aluminum soap of the higher fatty acids, and particularly an aluminum stearate soap comprising incorporating in the grease prior to the cooling of the grease and while the grease is in a fluid state, an aliphatic amine, and feeding the grease to a heat conductive surface in an amount to form a thin layer on said conductive surface, and exposing the grease in the absence of shearing stress, that is while the grease is in a quiescent state to the action of a cooling medium which is preferably directed countercurrently to the travel of the grease on the moving hot conductive surface.

The aliphatic amine may be a saturated aliphatic amine, and includes the saturated primary, secondary and tertiary amines and the cyclo-aliphatic amines, all of said amines containing more than 5 carbon atoms and less than 36 carbon atoms per molecule, but preferably containing 10 to 18 carbon atoms per molecule. Instead of using the amines above set forth, their fatty acid soaps may be used, the soaps of said amines containing more than 5 carbon atoms and less than 36 carbon atoms per molecule, and preferably 10 to 18 carbon atoms per molecule, the carbon atoms of the acid radical of the soap, such as the fatty acid radical, being included as part of the carbon atoms of the molecule.

A second object of the present invention is to flow a compounded grease containing an amine crystallization inhibitor of the character set forth in a hot fluid condition on to a relatively thin conductive surface, as for example a thin metal band, so as to form a thin layer of grease varying from approximately 0.1'' to 1'' in thickness on the band, and to thereafter move the metal band and the grease through a cooling zone to form a highly transparent grease.

A third object of the present invention is to provide a method of continuously cooling an aluminum soap lubricating grease in thin layers of the character herein set forth, and while in a quiescent state, the rate of cooling of the grease being varied as the grease cools from its hot fluid state.

A fourth object of the present invention is to quickly cool an aluminum soap grease of the character herein set forth while in thin layers in successive cooling zones, one cooling zone being adjacent the hot end of the cooling belt, and the other cooling zone being adjacent the discharge end of the cooling belt, said zones being for convenience designated respectively hot and cold cooling zones, there passing through the zones a cooling medium, and there being a differential between the velocity of the cooling medium in the two successive cooling zones. The ratio of the velocity of the cooling medium in the cold zone to the velocity of the cooling medium in the heated zone varies over the range of 1:1 to 10:1.

A fifth object of the present invention is to provide a smooth non-bleeding transparent aluminum base grease of buttery consistency, said grease containing less than 5½% to 6% of aluminum soap of the higher fatty acids, and .05% to .3% of a saturated aliphatic amine or cycloaliphatic amine or amine fatty acid soaps, said amines being of the character hereinbefore set forth.

It is a sixth object of the present invention to produce an aluminum soap lubricating grease having an aliphatic amine of the character set forth by cooling the grease in layers of .1'' to 1'' in thickness while the grease is being subjected to a rapid current of cold air while it is cooling in a single operation from about 350° F. to about 80 to 90° F. within a time period varying from about 3 minutes to 40 minutes while the moving grease is subjected to a countercurrent flow of a cooling medium, said grease being maintained in a quiescent condition and being free from shearing stresses during the cooling period.

The present invention will be described in connection with the accompanying drawing in which:

Figure 1 is a plan view of a grease cooling apparatus in accordance with the present invention;

Fig. 2 is a detail of the end of the belt showing the cooling means and baffling means taken substantially on the line 2—2 of Fig. 1;

Fig. 3 is a section taken on the line 3—3 of Fig. 2, illustrating the grease feeding means;

Fig. 4 is a detail of the supporting means for the upper portion of the belt;

Fig. 5 is a section taken substantially along the line 5—5 of Fig. 1;

Fig. 6 is a side elevation of the grease cooling apparatus of the present invention;

Fig. 7 is a section taken substantially along the line 7—7 of Fig. 6;

Fig. 8 is an enlarged detail of the means for removing the grease from the belt;

Fig. 9 is a side elevation of a modified form of the grease cooling apparatus; and

Fig. 10 is a plan view thereof.

Fig. 11 is an enlarged side elevation of a portion of the casing partly broken away to show the diverting baffles.

Referring to the figures of the drawing, and particularly Fig. 1 thereof, a grease cooling apparatus in which the present invention may be carried out is indicated in general at 10. The apparatus includes a sheet metal casing which is generally rectangular in cross section, as best shown in Fig. 7, including a top 11 and a pair of sides 12 and 13. The casing is supported as by longitudinal I-beams 14, carried on transverse beams 15 which are in turn supported in any conventional fashion on the columns 16. The casing is preferably constructed of sheet metal or may be constructed of any suitable thin material of sufficient structural strength.

Trained over suitable pulleys 17 and 18 is a steel belt 19, provided with a pair of strips preferably of a synthetic rubber, indicated at 20 and 21 respectively. These strips which extend entirely around the belt adjacent the edges thereof, prevent the hot grease from flowing off the edges of the belt. They are preferably bolted to the belt by suitable bolts not shown, although they may be riveted or otherwise suitably fixed to the belt. The right hand pulley 18 is driven by means of a suitable belt 22 from a variable speed transmission and motor indicated in general at 23 (Fig. 1). The belt is kept under tension by a suitable tension means consisting of a tensioning frame 24 urged by means of a weight 25 connected to the tensioning frame as by cables 26 and turned over suitable pulleys 27. Preferably, the weight 25 is sufficient to urge the frame 24 and journals 28 for the pulley 17 with sufficient force so that the steel belt is kept constantly under tension, and tends to assume a horizontal position. The upper course of the belt is preferably supported on a plurality of U-shaped steel rods, best shown in Fig. 4, and indicated by the reference numeral 29. A plurality of steel rods 29 are distributed across the width of the belt in any suitable manner. The lower ends of the steel rods are passed through supporting frame members 30, and are provided with a threaded section carrying nuts 31 and 32. This permits either end of the rods to be raised or lowered, so that the belt may be supported thereon in an absolutely horizontal position. Further, the use of these rods does not present any substantial air resistance to the flow of cooling air which is passing along the lower surface of the steel belt 19. The cooling air is fed into the outlet end of the casing about the belt from a pair of blowers 33 and 34, the blower

33 being positioned to direct air along the upper surface of the belt, and the blower 34 to direct air along the lower surface of the belt, as shown in Fig. 6.

A baffle is provided within the casing, indicated at 35, so that the air flow from the upper blower 33 is in substantial parallelism with the upper surface of the belt 19. A plurality of discharges are provided from the lower blower, one discharge being on each side of the casing, and indicated at 36 and 37 respectively. It will be noted that these discharges also feed in substantially parallel to the lower surface of the belt, and at the outlet end of the casing. The air is exhausted from the casing as by a stack 38 directly communicating with the upper or top of the inlet end of the casing, and provided with a pair of ducts 39 and 39*a* communicating with the casing below the upper course of the belt, as best shown in Figs. 1 and 6. The lower course of the belt is supported in conventional fashion on idler pulleys 40. The lower course of the belt 19*a* may also be supported in any suitable manner, but preferably by steel rods as described above and as shown for support of the upper course of the belt 19 in Fig. 4. This lower course 19*a* of the belt may be encased and provided with means of supplying cooling medium through the casing. In this manner, the lower course 19*a* may be used for the cooling of the grease, whereby the capacity of a given installation may be approximately doubled.

Grease is fed into the apparatus and distributed uniformly over the surface of the belt as by an inlet conduit 41, feeding into a header 42, provided with a plurality of distributing outlets 43. Positioned between the outlets 43 and the exhaust stack 38 is an adjustable baffle 44 which prevents too rapid cooling of the grease immediately after being fed, and permits the grease in hot liquid condition to spread out on the surface of the belt in a uniform layer. A dam in the form of an angle and indicated at 45 is provided to prevent the grease from flowing off the end of the belt. Preferably the dam or angle iron 45 is provided with oil resistant synthetic rubber ends or surfaces where it is in contact with the grease and there is thus provided prior to the exhaust stack of the device a relatively warm zone which is free from the cooling medium, i. e. as otherwise the cold air striking the grease is likely to cause chilling and non-uniformity in layer structure upon being fed.

The outlets 43 in general should be more than six inches and less than eighteen inches apart, preferably from eight to fourteen inches apart, since if they are too close too much grease will be fed, and if they are too far apart non-uniform distribution of grease will be produced. In order to insure uniform flow of the hot fluid grease from all of the outlets, the combined cross sectional area of the outlets 43 should be smaller by about 25 to 50% than the cross sectional area of the header 42.

The belt as shown in Fig. 6 is rotated in a clockwise direction, and the cooled grease is removed from the belt as by a scraper 46, best shown in Fig. 8. A funnel 47 is positioned to catch the grease being removed from the belt and distribute the same into either one of a pair of tanks 48 and 49 respectively, as shown in Fig. 5. The funnel 47 is provided with a pair of ports at each of its sides, closed by sliding covers 50 and 51. When the cover 51 is in the position shown in Fig. 5, grease will be fed into the tank 48. When the cover 51 is slid downwardly to close the opening, and the cover 50 is slid upwardly to open the port, grease will be fed into the tank 49. The tanks 48 and 49 are provided with conical bottoms, and feed through a valve 52 and a positive displacement pump 53 into a suitable grease strainer 54.

In the form of the apparatus shown in Figs. 9 and 10, an intermediate pair of exhaust ducts 55 and 55*a* are provided at a point 56 located approximately one-third of the distance between the hot end of the casing and the cold end of the casing, the point 56 being closer to the hot end of the casing. The point 56 definitely marks the dividing line between the so-called "hot zone" A and the so-called "cold zone" B. Also within the main casing 57 which corresponds to the casing 10 shown in Figs. 1 to 6 inclusive, there are provided adjustable baffles 58 and 58*a* which serve to regulate the relative amount of air flowing through the discharge ducts 55 and 55*a* respectively and the balance of the main casing. The intermediate exhaust ducts may be connected to the exhaust ducts 39 or communicate directly with the stack 38.

Utilizing the form of apparatus shown in Figs. 9 and 10, a portion of the cooling area or other cooling gaseous medium may be exhausted, that is let out at an intermediate point of the casing 57, the amount of air which is let out being regulated by means of the baffles 58 and 58*a* to thereby perform the first stage of cooling at a higher temperature level, that is at a lower rate of cooling as is desirable with some greases in order to improve their quality. In other words, the time of cooling is divided up into two periods, a first period and a second period. The rate of cooling when the grease is hot, that is during the first period, is lower than the rate of cooling during the second period when the grease has already cooled somewhat and started to gel. By exhausting the cooling medium intermediate the hot and cold ends of the cooling grease layer, there is maintained a control over the velocity of flow of the cooling medium in the casing while the cooling medium is in contact with the moving layer of the grease. The distance through which the grease spread in a thin layer travels from the hot end of the grease layer to the cold end of the grease layer is divided, as stated, into what may be termed a "hot zone" A and a relative cool zone B, and the grease is then cooled in these zones or stages, namely a first stage and a second stage. In the first stage at and adjacent the hot end of the grease layer the velocity of the cooling medium is preferably lower than the velocity of the cooling medium in the second cooling stage at and adjacent the cool discharge end of the grease layer. The ratio of the velocity of the cooling medium in zone B, that is, the second stage, to the velocity of flow of the cooling medium in the hot zone A, that is the first stage, may vary over the range of 1:1 to 10:1. In other words, the rate of cooling in zone A is lower than the rate of cooling in the second cooling stage in zone B. More specifically, the rate of cooling of the aluminum soap lubricating grease from its hot fluid state which may be as high as 350° F. down to about 175° F. is slower than the rate of cooling in the second cooling stage in zone B where the grease preferably cools to below its transition temperature, that is usually below 100° F. or 90° F.

The cooling medium may be conditioned by adjusting its temperature and/or velocity and by other physical or chemical characteristics to cool the grease at a slower rate of cooling in zone A than in zone B. While in one form of the invention it is desirable to employ two-stage cooling, it is within the province of the present invention to dispense with the two-stage cooling and uniformly cool the grease as it passes from the inlet end of the cooling belt to the outlet end thereof.

Preferably the grease is fed to the belt at such a speed so that a layer of grease of the proper thickness is produced. If the layer on the belt is more than approximately one inch thick, the rate of cooling will be prohibitively low, and in the case of certain greases the grease will tend to flow in an uncontrollable manner along the belt, due to the slow rate of gelation. If the layer is less than 0.1'', the rate of heat transfer through the grease is more rapid than the rate of heat removal from the grease by the air stream within the practicable range of air pressures, and air velocities. Preferably, the grease is cooled by air at room temperature, but the air used for cooling purposes may be passed through a suitable refrigeration plant and supplied to the cooling duct at a temperature as low as 0° F. On the other hand, where warm outside temperatures prevail, the cooling air may be supplied at temperatures as high as 100° F. In other words, in general the cooling air at atmospheric or higher pressures may be supplied at temperatures of 0° to 100° F. Preferably however, the grease should be cooled to approximately 80° to 90° F., and therefore the cool air should be supplied at a temperature lower than 80° to 90° F. and preferably between 40° F. and 90° F. Since the grease moves in a thin layer on the belt, the various particles of grease are not displaced relative to one another, and there is no shearing stress set up.

In general, the belt is preferably approximately 100 feet long, and moves at a speed of 13 feet per minute, so that the total time of cooling for the average grease is approximately 7 to 10 minutes. This is sufficient to cool the grease and set the same at least to a jelled mass. The blowers **33** and **34** may be a standard type of blower capable of blowing approximately 30,000 cu. ft. a minute, and the tanks **48** and **49** are capable of holding one charge of the belt. In the case of a belt approximately 6 ft. wide and 100 ft. long, this would amount to approximately 15,000 pounds. By providing two tanks of this type, the grease may be allowed to remain in a quiescent condition after cooling.

The following is an example of an aluminum soap grease made in accordance with the present invention:

Example I

The grease may be compounded as follows:

Aluminum di-stearate	55 lbs. (5.5%)
Coastal pale oil of 100 vis. at 100° F.	110 lbs.
Coastal pale oil 2000 vis. at 100° F.	830 lbs.
Polyisobutylene solution	3 lb. (.3%)
Stearylamine	.5 lb. (.05%)

The aluminum di-stearate and the coastal pale oil of 100 viscosity at 100 F. are thoroughly mixed at room temperature, and then heated under continued agitation to about 350° F. Then the 2000 viscosity coastal pale oil is added as well as the polyisobutylene solution and the monostearylamine. During the addition of these ingredients, the temperature is preferably held at around 325 to 355° F. After complete homogeneity has been obtained, the hot grease in its fluid condition is pumped on to the moving belt **19** of the cooling apparatus at such a rate that a depth of grease layer of about one-third of an inch is produced. Cooling air is supplied to the tunnel or casing by the blowers **33** and **34** at a velocity of 3500 feet per minute, and at a temperature of approximately 80° F. The belt is moved at a speed of approximately 10 feet per minute so that the grease remains on the belt for approximately 10 minutes, and then is discharged into one of the tanks **48** or **49**. The temperature of the grease upon discharge is about 95° F. The aluminum soap grease is moved as a quiescent mass countercurrent to the air stream.

The resulting grease is of smooth texture, transparent, non-bleeding and slightly tacky. The ASTM worked penetration after 60 strokes is 325 decimillimeters, and after 300 strokes 348 decimillimeters.

It is to be noted that the grease cooled in accordance with the above required about 10 minutes to cool, and did not require any interruption for the transformation of the rubbery stringy grease mass into a smooth transparent coherent gel at the 120° F. temperature level as is required by the prior art.

Referring to the above example, it is noted that the amount of aluminum stearate grease used is 5½%. Proceeding in accordance with the prior art, it would have required about 6½% of aluminum distearate plus 0.6% of aluminum di-12-hydroxystearate in order to obtain a grease of like consistency, and the grease would have to be processed in an entirely different manner. As pointed out, the aluminum di-12-hydroxy stearate is a rather expensive ingredient, and its use is eliminated by the present invention.

Example II

An aluminum stearate grease may be compounded as follows:

Aluminum stearate	11 lbs. (5.5%)
Paraffin oil, 100 vis. at 100° F.	50 lbs.
Red oil, 2000 vis. at 100° F.	139 lbs.
Polyisobutylene solution (7% con.)	½ lb. (.25%)
Dicyclohexylamine	2 oz. (0.06%)

The procedure set forth in the first example was employed in the present example, except that the grease was maintained at a temperature of about 320 F. and cooled as previously set forth in a thin film varying in thickness from ⅛'' to ¼'', the time of cooling grease on the 100' belt being 8 minutes. The grease is cooled in a continuous single operation within 8 minutes to a buttery consistency, non-bleeding and free from grains; its ASTM worked penetration at 77° F. after 60 strokes is 336 and after 300 strokes 357 decimillimeters.

Example III

Instead of using dicyclohexylamine, as set forth in Example II, there may be substituted therefor hexadecylamine in amounts varying from 0.05% to 0.25%.

In the above examples, there may be added between 0.05% and 0.25%, and preferably between 0.10% and 0.15% of gum rosin or abietic acid to increase the hardness of the grease. The aluminum stearate added to the grease may be the mono-, di-, or tri-stearate, or a mixture of any of these salts.

In carrying out the present invention, saponifiable organic constituents of the grease-making batch may be any of the saponifiable organic constituents generally used in the production of a lubricating grease, said organic constituents being combined with aluminum to produce an aluminum soap. The fatty acids usually used in grease making are in general the saturated fatty acids containing up to 32 carbon atoms, and usually from 14 to 24 carbon atoms; and the unsaturated acids containing up to 32 carbon atoms and usually from 18 to 22 carbon atoms. The fatty acid which is used to make the aluminum salt may be a saturated fatty acid or an unsaturated fatty acid, and includes stearic acid to produce aluminum stearate, 12-hydroxy stearic acid, 9, 10-hydroxy stearic acid (that is the 9 and 10 carbon atoms carry the two hydroxy groups), 4-hydroxy palmitic acid, iso-stearic acid, iso-palmitic acid, 12-hydroxy, 9-oleic acid (ricinoleic acid), oleic acid, linoleic acid, hydrogenated fish oil fatty acids, palm oil fatty acids, cotton seed oil fatty acids.

The following table shows the results obtained using primary, secondary, tertiary and cyclo-aliphatic amines and fatty soaps thereof:

primary or secondary amines or the soaps thereof, there may be used as the crystallization inhibitor, a heterocyclic nuclear amine of between 5 and 24 carbon atoms per molecule, but preferably having between 10 and 18 carbon atoms per molecule. Instead of using the heterocyclic nuclear amine of the character above set forth, there can be used in lieu thereof a fatty acid soap of the heterocyclic nuclear amine, said amine soap having between 10 and 28 carbon atoms per molecule.

It is desired to point out that while the present invention is applicable to both lubricating greases containing aluminum di-salts of fatty acid soaps, as for example, aluminum distearate and those containing aluminum mono-salts of fatty acids, as for example aluminum monostearate. The invention is particularly applicable to those greases which contain the aluminum mono-salts of fatty acids. As far as known, the prior art aluminum base greases are the aluminum di-salts of fatty acid greases, and not the aluminum mono-salts of fatty acid greases. For example, in the trade, when aluminum stearate is referred to it is well understood that what is meant is the aluminum distearate, and not the aluminum monostearate.

	Additive, percent	Aluminum Stearate, percent	Bleeding	Cracking	Texture	Penetrations	
						60 St.	300 St.
Aliphatic Amines:							
C_{18}—NH_2	0.5	8	None	None	V. S	302	337
Do	0.25	8	do	do	V. S	295	328
Do	0.10	6.5	do	do	V. S	280	305
Do	0.05	6.3	V. Sl	V. Sl	S	295	318
Do	0.10	6.6	V. Sl	None	V. S	300	335
Do	0.10	5.5	None	do	V. S	312	339
C_{16}—NH_2	0.10	5.5	do	do	V. S	332	355
C_{14}—NH_2	0.10	5.5	V. Sl	do	V. S	334	352
C_{12}—NH_2	0.10	5.5	V. Sl	do	V. S	327	359
C_8—NH_2	0.05	5.5	Sl	V. Sl	S	340	350
$(C_5)_2NH$	0.10	5.5	Sl	Sl	S	325	348
$(C_5)_3N$	0.10	5.5	pos	pos	s. g	314	336
$(C_4)_3N$	0.10	5.5	do	Sl	S. G	313	330
C_8NH_2—Oleate	0.10	5.5	Sl	Sl	S. G	345	360
C_8—NH_2	0.10	5.5	V. Sl	None	V. S	344	365
Cyclo-Aliphatic Amines:							
Di-cyclohexylamine	0.10	5.5	V. Sl	do	V. S	336	357
Do	0.06	5.5	Sl	V. Sl	S		

The abbreviations used in the above table are as follows:
V. S.—Very smooth, does not require milling
S.—Smooth after light milling
S. G.—Slightly grainy after milling
G.—Grainy after milling
V. Sl.—Very slight bleeding or cracking
Sl.—Slight bleeding or cracking

In one form of the invention a suspension of aluminum stearate in a lubricating oil of 100 to 500 S. S. U. at 100° F. may be made and the suspension of aluminum stearate in the oil is heated to 350° F. There is then added to the mixture additional lubricating oil of 100 to 4000, but preferably 100 to 2000 S. S. U. at 100° F., together with 0.05 to 0.30% of an aliphatic primary or secondary amine containing 5 to 36 carbon atoms per molecule, and preferably 10 to 18 carbon atoms per molecule, the temperature of the resulting mixture being maintained between 310° F. and 350° F. The mixture is then flowed on to the cooling belt 19 and cooled as a thin quiescent layer to below the transition temperature of the grease in a single operation to produce a smooth homogeneous grease gel. Instead of using an aliphatic, primary or secondary amine, as set forth in the preceding paragraph, the fatty acid soaps of the primary or secondary amine may be used, said aliphatic amine soaps containing between 10 and 36 atoms per molecule and preferably, 10 to 23 atoms per molecule, the carbon atoms of the fatty acid being counted as part of the molecule. Instead of using the aliphatic,

The aluminum monostearate greases, when quickly cooled, exhibit a substantially greater tendency to grain, that is, crystallize and bleed after gelling than do the aluminum distearate greases. It is only using the present invention employing aliphatic amines that it is possible to quickly cool aluminum base greases containing the aluminum mono soaps, as for example, aluminum mono salts of fatty acid soaps containing 14 to 24 carbon atoms in a molecule of which aluminum monostearate is a specific example.

The following is a specific example illustrating the manufacture of grease employing aluminum monostearate together with mono-myristyl amine:

Example IV

Aluminum monostearate	45 grams (4½%)
Coastal pale oil of 100° Vis. at 100° F	155 grams
Preheated coastal red oil of 2000 Vis. at 100° F	5 grams (.5%)
Polyisobutylene	800 grams
Mono-myristyl amine	.8 gram (.05%)
Rosin	1 gram

The aluminum monostearate is suspended in 155 grams of the coastal pale oil at room temperature, and the smooth mixture is then heated to about 370° F. until a homogeneous melt is obtained. Thereafter the preheated coastal red oil, the polyisobutylene solution, the mono-myristyl amine, and the rosin are added while the entire mass is kept at about 340° F. The mass is stirred until a homogeneous structure is obtained and the resulting mixture is then poured in a thin layer of about 1/4'' to 3/8'' in thickness and exposed to a blast of cool air having a temperature of 80° F., the grease being cooled in accordance with the method herein and in the apparatus herein set forth, the hot grease in its fluid condition being pumped on to the belt 19 of the cooling apparatus. The belt is moved at a speed of approximately 15 feet per minute so that the grease remains on the belt for approximately 15 minutes. The aluminum soap grease is moved as a quiescent mass countercurrent to the air stream.

The resulting grease is a non-bleeding smooth buttery gel. The consistency or ASTM worked penetration of the grease after 60 strokes is 346 decimillimeters and after 300 strokes, 367 decimillimeters.

A further example using aluminum monostearate is as follows:

Example V

Aluminum monostearate	60 grams 6%
Coastal pale oil 100 Vis. at 100° F	140 grams
Coastal pale oil 100 Vis. at 100° F	800 grams
Polyisobutylene	5 grams .5%
Dodecylethylenediamine	0.8 grams .08%

The above grease was cooked and cooled in accordance with the procedure set forth in Example IV except that the time of cooling was 20 minutes. The grease is cooled in a continuous single operation in 20 minutes to a non-bleeding smooth buttery gel having an ASTM worked penetration at 77° F. after 60 strokes of 320 decimillimeters and after 300 strokes, 336 decimillimeters.

The di-N,N' dodecylethylenediamine is:

$$C_{12}H_{25}-\underset{H}{N}-C_2H_4-\underset{H}{N}-C_{12}H_{35}$$

In the above examples the aluminum monostearate content is 15.61% alumina. It is desired to point out that the aluminum trioxide content, that is the Al_2O_3 content of the aluminum monostearate is usually 15%±1%. The aluminum trioxide content, that is the Al_2O_3 content of aluminum distearate, is usually 8.1%+1%.

While in the above specific examples the aluminum monostearate is set forth, it is, of course, obvious that other aluminum salts of other fatty acids may be used, said fatty acids containing up to 32 carbon atoms, but preferably 14 to 24 carbon atoms for the saturated fatty acids. The mono aluminum soaps of the saturated fatty acids may be used containing up to 32 carbon atoms and usually from 18 to 22 carbon atoms, as for example, aluminum mono palmitate, and the like. These compounds may also be designated the mono fatty acid salts of aluminum. The generic formula for mono fatty acid salts of aluminum then becomes

$$R-\overset{O}{\overset{\|}{C}}-O-Al(OH)_2$$

The formula for the di-salt is

$$R-\overset{O}{\overset{\|}{C}}-O-\underset{OH}{\underset{|}{Al}}-O-\overset{O}{\overset{\|}{C}}-R$$

than when the di-aluminum soaps of the fatty acids are used.

In carrying out the present invention, for the dicyclohexylamine there may be substituted monocyclohexylamine. However, the latter is somewhat difficult to incorporate in the grease because of its low boiling point.

While the aluminum soap greases can be cooled in successive cooling zones and using different velocities for the cooling medium in the successive cooling zones, it is within the province of the present invention to cool in one zone and use only a single given velocity for the cooling medium.

The amount of polyisobutylene used may vary from 0.3 to 0.5% based on the weight of the grease. When the aluminum soap grease is cooled in successive hot and cold cooling stages the grease in the hot zone may be cooled to a temperature varying between about 200° F. and about 250° F., and the grease in the second cooling zone is then cooled to below the transition temperature of the grease.

All of the above amine crystallization inhibitors may be added in amounts varying between 0.05% to 0.3% based on the total weight of the grease; all of the percentages herein set forth are based on the total weight of the grease. These greases are preferably cooled in thin quiescent layers of 0.10 to 1.0'' thickness, but preferably in layers having a thickness varying between 0.20 and 0.40.

I claim:

1. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2000 SSU at 100° F., up to 6% of aluminum monostearate, and 0.05% to 0.3% of a saturated aliphatic amine having 5 to 36 carbon atoms per molecule, feeding the grease in hot fluid condition to a heat conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a gaseous heat absorbing medium until there is produced a smooth substantially homogeneous grease gel.

2. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2000 SSU at 100° F., aluminum monostearate, and 0.05% to 0.3% of a saturated aliphatic amine having 5 to 36 carbon atoms per molecule, feeding the grease in hot fluid condition to a heat conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a gaseous heat absorbing medium until there is produced a smooth substantially homogeneous grease gel.

3. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2000 SSU at 100° F., up to 6% aluminum monostearate, 0.05% to 0.3% of a saturated aliphatic amine having 5 to 36 carbon atoms per molecule, a small amount of rosin sufficient to aid in thickening and hardening of the grease, feeding the grease in a hot fluid condition to a heat conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in

a quiescent state by subjecting the grease to the action of a gaseous heat absorbing medium until there is produced a smooth substantially homogeneous grease gel.

4. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2000 SSU at 100° F., up to 6% aluminum monostearate, and 0.05% to 0.3% of a saturated aliphatic amine having 5 to 36 carbon atoms per molecule, a small amount of rosin and about .3% to .5% of polyisobutylene, feeding the grease in a hot fluid condition to a heat conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a gaseous heat absorbing medium until there is produced a smooth substantially homogeneous grease gel.

5. The method of producing an aluminum soap grease containing up to 6% aluminum soap comprising incorporating in the grease 0.05% to 0.3% of a saturated aliphatic amine having more than 5 carbon atoms in the molecule and a small amount sufficient to impart tackiness to said grease of polyisobutylene, feeding the grease in a hot fluid condition to a heat-conductive moving surface, forming a thin layer of grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a heat absorbing air until there is produced a smooth and homogeneous grease gel.

6. The method comprising forming an aluminum soap grease containing up to 6% of an aluminum soap, from about 0.25% to about 0.5% of polyisobutylene, and from 0.05% to 0.3% of a saturated aliphatic amine having more than 5 carbon atoms in a molecule, feeding the grease in a hot fluid condition to a heat-conductive moving surface, forming a thin layer of grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a heat absorbing cooling air until there is produced a smooth substantially homogeneous grease gel.

7. The method of producing an aluminum soap grease containing from about 5.5% to about 8% of aluminum soap comprising incorporating in the grease 0.05% to 0.3% of a saturated aliphatic amine having at least 5 carbon atoms in a molecule, feeding the grease in a hot fluid condition to a heat conductive surface, forming a thin layer of grease on said surface and cooling the grease while in a quiescent state in successive hot and cold cooling stages in the presence of a heat absorbing cooling air, the grease in the hot zone being cooled to a temperature varying between 200° to 250° F. and the grease in the second cooling stage being cooled to below the transition temperature of the grease, the ratio of the velocity of flow of the cooling air in the cold stage adjacent the discharge end of the heat conducting surface to the velocity of the cooling air in the hot stage adjacent the intake end of the heat conductive surface varying over the range of 1:1 to 10:1.

8. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2,000 SSV at 100° F., up to 6% of aluminum distearate, from 0.05% to 0.3% stearylamine and from about 0.3% to 0.5% of polyisobutylene, feeding the grease in hot fluid condition to a heat-conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a gaseous heat-absorbing medium until there is produced a smooth, substantially homogeneous grease gel.

9. The method comprising forming an aluminum soap grease from a mixture of a mineral oil base having a viscosity of less than 2,000 SSV at 100% F., up to 6% of aluminum monostearate, from 0.05% to 0.3% dodecylethylene diamine and from about 0.3% to 0.5% of polyisobutylene, feeding the grease in hot fluid condition to a heat-conductive moving surface, forming a thin layer of the grease thereon, and cooling the grease while in a quiescent state by subjecting the grease to the action of a gaseous heat-absorbing medium until there is produced a smooth, substantially homogeneous grease gel.

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