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(54) IMPROVEMENTS RELATING TO METHODS AND APPARATUS FOR THE PRODUCTION OF CELLULAR THERMOPLASTIC MATERIAL

(71) We, UNION CARBIDE CORPORATION, a corporation organized and existing under the laws of the State of New York, United States of America, of 270 Park Avenue, New York, State of New York 10017, United States of America, (assignee of JOHN CLARK MILLER, ARCHIBALD LOUIS BURNETT and LEONARD SEBASTIAN SCAROLA), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method and apparatus for the production of cellular thermoplastic material.

Heretofore, many systems have been suggested for the production of cellular thermoplastic material. However, it has been found for a number of applications that the degree of uniformity of blowing agent dispersion in the thermoplastic material is highly critical for successful utilization.

In the production of cellular thermoplastic material in an extrusion process, the extended molten product varies in uniformity as a direct result of such non-uniform dispersion. The extrudate uniformity is critical in many electrical applications, such as the production of insulation for CATV cable.

Coaxial cables currently employed to transmit television signals (CATV) cables) predominantly employ cellular low density polyethylene as the insulating material. Processes for the production of such cellular insulation are presently employed, wherein production is effected by melting solid thermoplastic material in an extruder into which a gaseous blowing agent is injected.

CATV cables must have a very uniform composition and diameter or a portion of the signal may be lost. This type of loss is referred to as

structural return loss (SRL). Non-uniformities which cause SRL can be due to variation in the extrusion line, e.g. an eccentric pulley, or in the extrusion process, e.g. extruder surging. Recent runs have shown that gas injection using conventional screws can result in an unstable extrusion. Extruder instability may be measured by fluctuations in head pressure which accurately reflect fluctuations in output or viscosity as shown by the following equation:

$$\text{Pressure} = \text{Viscosity} \times \text{Constant} \times \text{Output}$$

Thus, measurement of head pressure may be employed as a measure of extruder stability.

It is, accordingly, a prime aim of the present invention to provide a method and apparatus for the production of cellular thermoplastic material having high degrees of structural and compositional uniformity.

Other aims and advantages of the present invention will be apparent from the following description and appended claims.

It is, of course, to be understood that the term "thermoplastic material", as employed herein, is well known to those skilled in the extrusion molding art and includes by way of example such resins as polyethylene, polypropylene, polystyrene, polyvinyl chloride and a wide variety of other synthetic organic resinous materials which are accepted as exhibiting thermoplastic properties, and thermosetting and elastomeric resinous materials which also exhibit thermoplastic properties at elevated temperatures below the setting temperature. In addition, the resins may contain various chemical and/or physical property modifiers or additives such as are well known to the art.

Expanding or blowing gas employed in the apparatus of the invention should preferably (especially for wire and cable applications),

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be chemically inert toward, and preferably soluble in the base polymer of the expandable composition and would thus include inert gases, such as nitrogen, argon, helium, neon.

5 Other blowing agents may be used. Thus, such blowing agents would include the fluorocarbon type blowing agents.

10 While the use of nucleating agents is not required in the broadest aspects of the process of the present invention, it has been found preferable to employ nucleating agents.

The nucleating agents which may be used in the compositions of the present invention are materials which provide fine particle sized nucleating sites in the thermoplastic material base polymer during the expansion or blowing thereof, as described below.

15 The particle size of the nucleating agents should be of the order of about 0.01 to 50 microns. Such materials would include polytetrafluoroethylene, azodicarbonamide, p,p'-oxybis(benzene sulfonyl hydrazide), trihydrazino - sym - triazine, and p - toluene sulfonyl semicarbazide.

25 The nucleating agents may be used individually or in combination thereof.

The nucleating agents should be dispersed as uniformly as possible throughout the mass of the base polymer.

30 According to the present invention there is provided an extruder comprising a hollow barrel, a screw mounted in said barrel to advance material therethrough, a mixer head mounted in said barrel and a gaseous blowing agent inlet positioned in said barrel near the upstream end of said mixer head, said mixer head having in the surface thereof a plurality of alternate lands and grooves, each said land having close clearance with the internal walls of said barrel near the upstream end thereof in the region of said blowing agent inlet and being divided to form a land pair in the mixing region downstream of said blowing agent inlet, the lands of the land pairs having alternately spaced and close clearance with said walls of the barrel.

45 According to a further aspect of the present invention there is provided an extruder having a hollow barrel, a screw mounted in said barrel to advance material therethrough, a mixer head longitudinally mounted in said barrel near the terminal end of said screw and a gaseous blowing agent inlet positioned in said barrel near the upstream end of said mixer head, said mixer head having in the surface thereof a plurality of alternate lands and grooves arranged to extend substantially longitudinally defining a plurality of grooves open at the upstream ends and closed at the downstream ends, each said land having close clearance with the internal walls of said barrel near the upstream end thereof in the region of said blowing agent inlet and being divided to form land pairs in the mixing region downstream of said inlet means, the lands of the

land pairs having alternately spaced and close clearance with said walls of the barrel, said mixer head having a hollow internal passage positioned therein over a substantial portion of its terminal length, and a plurality of inlet conduits severally communicating between said plurality of lands and said hollow internal passage, said inlet conduits being positioned between the lands of the land pairs which are, in turn, positioned between said grooves.

70 According to a still further aspect of the present invention there is provided a method of continuously effecting mixing, in an extruder, of a stream of thermoplastic material and a gaseous blowing agent comprising: forming a continuous stream of flowable thermoplastic material; subdividing said stream in to a plurality of component streams; introducing gaseous blowing agent into said plurality of component streams to form mixtures thereof.

80 The present invention will now be further described, by way of example, with reference to the accompanying drawings, in which:—

Fig. 1 is a cross-sectional view of extruder and wire drawing apparatus embodying the invention;

Fig. 2 is an elevational view of mixer head means employed in the embodiment of Fig. 1;

Fig. 3 is a sectional view taken along the line 3—3 of Fig. 2;

Fig. 4 is a sectional view taken along the line 4—4 of Fig. 2;

Fig. 5 is a cross-sectional view of a modified extruder embodying the invention; and

Fig. 6 is a cross-sectional view of another modified extruder embodying the invention.

Referring specifically to the embodiment of Figs. 1 and 2 of the drawings, extruder 10 is provided having outer housing 12, screw 14 and breaker plate 16. Cylindrical mixer head 18 is positioned on the terminal (downstream) end of screw 14 and comprises an internal axial passage 20 and a plurality of external longitudinal grooves 22 open on the upstream ends 26 and closed on the downstream ends 24.

Mixer head 18 is secured to screw 14 as by screw threads 30.

As thermoplastic material is fed to extruder 10 it passes through the screw section where screw 14 is designed to flux and melt the material before passage to the mixer head 18. The molten material is divided into a plurality of parallel flow streams which pass into the plurality of longitudinal grooves 22 formed between longitudinal land pairs 32 and 34.

Gaseous blowing agent is introduced into the extruder 10 through inlet 40 which is positioned to inject the gas into the groove means 22 of the mixer head 18 near the upstream ends thereof. As shown in Fig. 3, the mixer head lands in this region 42 are in close clearance with the inner walls of the extruder barrel. The rotation of the screw 14 including the mixer head 18 causes the lands to constantly sweep the gaseous blowing agent inlet port and intermit-

tently open and close passage of gas under substantially full inlet pressure to the parallel molten material streams passing through the plurality of groove means 22.

5 The lands downstream of the region 42 comprise land pairs 32 and 34 of different heights, as shown in Fig. 4. As there shown, the leading edges 32 of land pairs are in spaced clearance with the walls of the extruder barrel to provide therebetween regions of high shear mixing as the material and gas pass from the plurality of grooves over these edges to radial conduits 36 to the internal axial passage 20. The parallel streams of mixed gas and molten thermoplastic material are there joined in passage 20 and pass as a confluent stream from the mixer head 18 to discharge chamber 44 upstream of breaker plate 16. Conical member 43 is positioned at the upstream end of passage 20 to reduce the degradation of material in that region.

10 The gas and molten material in the plurality of grooves 22 are there mixed by a circular, swirling action caused by rotation and passage along the internal walls of the extruder housing. This action also prevents clogging of material in the mixing grooves.

15 The close clearance of the trailing portion of the land pairs 34 causes a cleaning action along the internal walls of the extruder barrel.

20 The molten material discharging through the breaker plate 16 of extruder 10 passes through chamber 44 to die head 46. An electrical conductor 48, which is to be coated with cellular thermoplastic material is fed to the die head at speeds of from about 20 to 10,000 feet per minute. A uniform coating of molten extrudate is continuously applied to the conductor as it passes through the die head. The initial thickness of the coating is determined by the orifice in die 50 which is located in the side of die head 46, and through which the coated conductor emerges from the die head. The coating thereon begins to expand because of pressure and temperature and is cone shaped for a short distance from the die, but then will stabilize at its maximum expanded thickness to yield the uniformly thick cellular thermoplastic material coating around the electrical conductor.

25 As shown in the modified embodiment of Fig. 5 of the drawings, an extruder 10 is provided having positioned in the hollow internal passage 20 of mixer head 18 means 45 for the prevention and/or disruption of flow channeling of the thermoplastic material-gaseous blowing agent mixture within passage 20. Such means 45 are secured to breaker plate 16 and extend back toward the upstream end of hollow internal passage 20 to provide a stationary agitator assembly 46 having agitating fins 47 positioned around the periphery thereof. These fins 47 are arranged in a helical pattern, as shown in the embodiment of Fig. 5. The effect of means 45 and 46 with appended fins 47

is to cause the thermoplastic material-gaseous blowing agent mixture passing through hollow internal passage means 20 to be constantly subjected to a mixing action which prevents and/or disrupts the flow channeling of this mixture within the hollow passage 20. The stream is then passed through an outwardly flaring passage 48 to an annular manifolding region 44 from which it is passed through the passages of breaker plate 16 which are disposed in a circular pattern around the outer periphery of the breaker plate.

70 Another modification of apparatus suitable for employment in the practice of the invention is shown in Fig. 6 of the drawings wherein a modified means 45' for the prevention and/or disruption of flow channeling is employed comprising a series of ribbon-shaped spiral sections 47' which are axially mounted at right angles to each other along the hollow internal passage 20 of the mixer head 18 and are secured to the mixer head by rod member 49 at the upstream end thereof. In this modified embodiment, agitating action of the ribbon shaped means 45 acts to prevent and/or disrupt the flow channeling of the mixture of thermoplastic material and gaseous blowing agent passing through the hollow internal passage 20 and, in turn, through manifold passage 44 and breaker plate 16.

80 Employing a 2-1/2", 24/1-L/D ratio extruder, various screw designs were evaluated with gas injection. Extruder stability was measured by head pressure fluctuations, which were recorded on a strip chart. With some designs, the screw was pushed out after the polymer was solidified, revealing the flow and dispersion of the gas. The results are summarized in the following table.

85 In all runs the basic screw used was a conventional polyethylene extruder screw having the following design:

| | | | |
|--------------------|----------|--------------|-----|
| Feed Section | 3 turns | 0.450" depth | |
| Transition Section | 13 turns | | |
| Metering Section | 4 turns | 0.095" depth | 110 |
| Pitch = Diameter | | (2.5") | |

The downstream end of the screw was threaded so that various designs could be added making a total L/D ratio of about 23/1. The extruder barrel was drilled to permit gas injection at many locations.

115 In Run 1, the added end design was a mixing section preceded by an annular space or unflighted section. The mixing section had a standard LeRoy type (U.S.P. No. 3,486,192) fluted (grooved) design with three inflow flutes and three outflow flutes. This section provided mixing and shearing of the gas-polymer mixture. The annular space provided an unflighted section into which the gas could be injected. It was thought that the absence of a

flight would prevent the gas flow from being cut off momentarily, and provide a more stable extrusion. The resulting short term pressure fluctuations were good, but a portion of the gas tended to collect in the annulus section until saturation level was reached, then it would rapidly discharge from the section and exit the extruder, in some cases completely undispersed. This was continuously repeated on a 5 to 10 minute cycle, and the foam density varied greatly.

In Run 2, the screw end was a section with 12 longitudinal flutes. For purposes of differentiation, this is called a fluted non-mixing section. In this Run, it served only to convey the resin from the end of the screw to the extruder head. The gas was injected four turns upstream of the flutes and represented injection into a flighted section of a conventional screw. The buildup-discharge cycle of Run 1 was not observed, apparently due to the flight breaking up the gas once per revolution. However, the stability was not good, and observation of a screw pushout revealed that some of the gas tended to collect and flow along the low pressure side (trailing edge) of the screw flight. This resulted in poor mixing and accounted for the head pressure variation.

Run 3 employed the same screw as Run 2, but the gas was injected at the upstream end of the fluted-non-mixing section. This resulted in better stability than Run 2 even though there was much less extruder residence time for the gas/polymer mixture. This showed the im-

portance of breaking the gas into small discrete volumes.

In Run 4, the fluted non-mixing section was replaced by the fluted mixing section described in Run 1. This provided better stability than the fluted non-mixing section despite the fewer number of flutes. This is attributed to the mixing and shearing provided by this LeRoy type fluted section.

In Run 5, an internal fluted mixing section having six flutes of the embodiment shown in Figs. 1 and 2 of the drawings was used. Gas was injected at the upstream end of the flutes. The gas/polymer mixture flowed down the flutes, over a high shear barrier, through a slot into the center of the section, and exited from the center. This design provided excellent stability. It performed better than the mixing section of Run 4 because it had more in-flow flutes and thus could break the gas into smaller volumes, and additionally because it provided added residence time while the resin slowly flowed through the center of the section.

Run 6 employed the same screw and mixer head section as Run 5, but gas was not injected. This is a control to illustrate that gas injection does reduce extrusion stability.

In these runs, the blowing agent employed was nitrogen gas, the extruder had a screw speed of 80 RPM and an output rate of 80 Lbs/Hr. The resin was polyethylene having a density of 0.920 g/cc and a melt index of 0.1 dg/min.

TABLE

| Run | Location of Injection Port | Foam Density g/cc | Extrusion Stability (Head Pressure Fluctuations) psi |
|-----|---|-------------------|--|
| 1 | Annulus (unflighted section) | varied | ± 1.0 |
| 2 | Four turns upstream of fluted section (injection into flighted section) | 0.45 | ± 3.0 |
| 3 | Upstream end of fluted section | 0.45 | ± 2.0 |
| 4 | Upstream end of fluted mixing section | 0.50 | ± 1.5 |
| 5 | Upstream end of fluted mixing section | 0.42 | ± 0.5 |
| 6 | No gas injected | 0.92 | ± 0.25 |

WHAT WE CLAIM IS:—

1. An extruder comprising a hollow barrel, a screw mounted in said barrel to advance material therethrough, a mixer head mounted in said barrel and a gaseous blowing agent inlet positioned in said barrel near the upstream end of said mixer head, said mixer head having in the surface thereof a plurality of alternate lands and grooves, each said land having close clearance with the internal walls of said barrel near the upstream end thereof in the region of said blowing agent inlet and

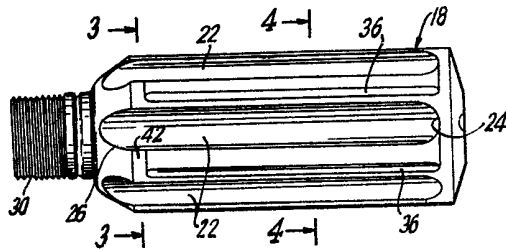
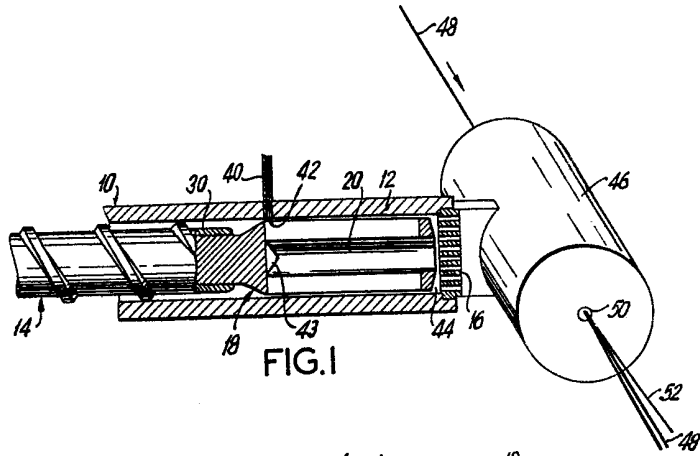
being divided to form a land pair in the mixing region downstream of said blowing agent inlet, the lands of the land pairs having alternately spaced and close clearance with said walls of the barrel.

2. An extruder as claimed in claim 1 in which said mixer head has a hollow internal passage positioned therein over a substantial portion of its terminal length, and a plurality of inlet conduits severally communicating between said plurality of lands and said hollow internal passage means.

3. An extruder as claimed in claim 2, in which means for the prevention and/or disruption of flow channeling are positioned in said hollow internal passage of said mixer head. 5
4. An extruder as claimed in claim 3, in which said means for the prevention and/or disruption of flow channeling is mounted so as to be stationary with respect to said extruder barrel. 10
5. An extruder as claimed in claim 4, in which said means for the prevention and/or disruption of flow channeling is mounted so as to corotate with said rotating mixer head means. 15
6. An extruder having a hollow barrel, a screw mounted in said barrel to advance material therethrough, a mixer head longitudinally mounted in said barrel near the terminal, end of said screw and a gaseous blowing agent inlet positioned in said barrel near the upstream end of said mixer head, said mixer head having in the surface thereof a plurality of alternate lands and grooves arranged to extend substantially longitudinally defining a plurality of grooves open at the upstream ends and closed at the downstream ends, each said land having close clearance with the internal walls of said barrel near the upstream end thereof in the region of said blowing agent inlet and being divided to form land pairs in the mixing region downstream of said inlet means, the lands of the land pairs having alternately spaced and close clearance with said walls of the barrel, said mixer head having a hollow internal passage positioned therein over a substantial portion of its terminal length, and a plurality of inlet conduits severally communicating between said plurality of lands and said hollow internal passage, said inlet conduits being positioned between the lands of the land pairs which are, in turn, positioned between said grooves. 20 25 30 35 40
7. An extruder as claimed in claim 6, in which means for the prevention and/or disruption of flow channeling are positioned in said hollow internal passage of said mixer head means. 45
8. An extruder as claimed in claim 6, in which said means for the prevention and/or disruption of flow channeling is mounted so as to be stationary with respect to said extruder barrel. 50
9. An extruder as claimed in claim 6, in which said means for the prevention and/or disruption of flow channeling is mounted so as to corotate with said rotating mixer head means. 55
10. A method of continuously effecting mixing, in an extruder, of a stream of thermoplastic material and a gaseous blowing agent comprising: forming a continuous stream of flowable thermoplastic material; subdividing said stream into a plurality of component streams; introducing gaseous blowing agent into said plurality of component streams to form mixtures thereof. 60 65
11. The method claimed in claim 10, further comprising the steps of intimately mixing each said component stream; rejoining said plurality of component streams to form a single stream; and mixing said single stream while preventing and/or disrupting channeling of said stream in its further passage through said extruder. 70
12. A method of continuously effecting mixing, in an extruder, of a stream of thermoplastic material and a gaseous blowing agent, substantially as hereinbefore described with reference to the accompanying drawings. 75
13. An extruder constructed and arranged substantially as hereinbefore described with reference to and as illustrated in Figs. 1 to 4, or Fig. 5, or Fig. 6 of the accompanying drawings. 80

W. P. THOMPSON & CO.,
Coopers Buildings,
Church Street,
Liverpool, L1 3AB,
Chartered Patent Agents.

Reference has been directed in pursuance of section 9, subsection (1) of the Patents Act 1949, to patent No. 1,359,103.



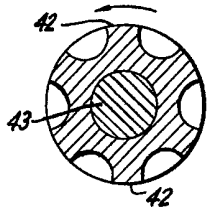


FIG. 3

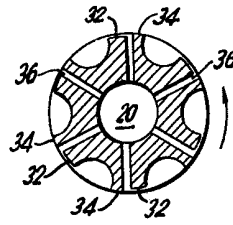


FIG. 4

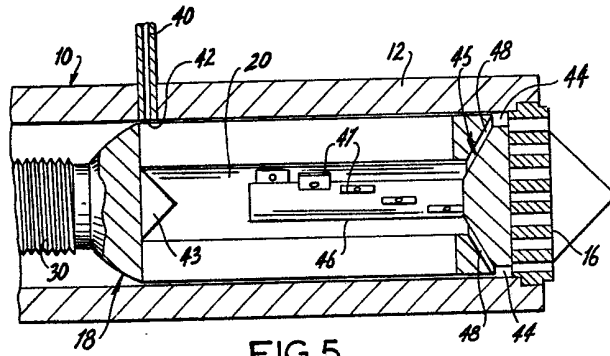


FIG. 5

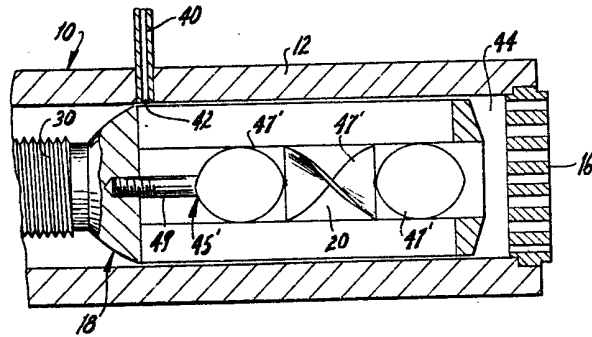


FIG. 6