[0005] It is known that variability in the feedstock significantly contributes to poor process stability and lack of quality control for the compounded product. See, *e.g.*, Extrusion: The Definitive Processing Guide and Handbook (2005) by Harold F. Giles Jr. *et al.* Particularly when the feedstock is stored in a solid form, this undesired variability originates from multiple sources (*e.g.*, differences in moisture levels between samples and the age of the feedstock). As a result, drying of the solid feedstock is frequently required prior to extrusion to achieve a controlled and consistent moisture content necessary to prevent significant reduction of relative viscosity (RV) due to hydrolysis. See, *e.g.*, Extrusion: The Definitive Processing Guide and Handbook (2005) by Harold F. Giles Jr. *et al.*

[0006] Poor warehouse management further contributes to the variability observed in the precursor compounding operation when the feedstock is in solid form as the feedstock is also susceptible to air oxidation over time.

[0007] Thus, there remains a recognized need for a compounded polymer, such as a compounded polyamide, such as compounded nylon66, that exhibits superior stability with improved performance with low variability of impurities, and the process for manufacturing such a product at the lowest possible cost. The compounded polymer prepared by the in-line process described herein shows improved molding performance with significantly lower plate out and longer mold life between cleanings compared to conventionally prepared compounded polymers where the feedstock is introduced to the polymer compounding equipment in solid form. The viscosity and quality control ranges for such a product are also less variable than that resulting from conventional pellet fed compounding. This corresponding improvement in quality directly translates into a superior product for the customer in terms of improved performance and lower cost compared to products prepared from a conventional compounding operations using feedstock in solid form.

Summary

[0007a] The invention is defined in the independent claims. Further embodiments of the invention are defined in the dependent claims.

[0008] Various embodiments may provide a process for preparing a compounded polyamide, comprising introducing a molten or substantially molten polyamide feedstock directly to one or more (such as 2, 3, 4, 5, *etc.*) polymer compounding devices, where the polyamide

feedstock is prepared by a polymerization process, wherein the polymerization process is a continuous polymerization process or a batch polymerization process.

[0009] In an aspect of the technique, the polyamide is selected from the group consisting of nylon 6, nylon 4/6, nylon 6/6, nylon 6/10, nylon 6/12, nylon 11, nylon 12, nylon MXD6, copolymer nylon (a copolymer of caprolactam with hexamethylenediamine adipate), nylon block copolymers, and copolymers comprising these nylons as main components. The methods of preparation of polyamides such as nylon 6/6 are well known. See, *e.g.*, U.S. Patent Nos. 6,197,855; 4,981,906; 4,320,213; 4,346,200; 4,713,415; 4,031,164; 2,071,250; 2,071,251; 2,130,523; 2,130,948; 2,241,322; 2,312,966; 2,512,606; and 3,393,210.

[00010] In an aspect of the technique, the nylon is nylon 6/6 (also referred to herein as nylon 66 or nylon66 or polyamide 66 or PA 66 or PA66).

[00011] In an aspect of the technique the compounded polyamide is a homopolymer.

[00012] In an aspect of the technique the compounded polyamide is a copolymer.

[00013] In an aspect of the technique, the molten polyamide feedstock is at a temperature of about 200°C to 400°C entering the polymer compounding device, such as 250°C to 375°C, such as 275°C to 300°C.

[00014] In an aspect of the technique, the polymer compounding device is an extruder.

[00015] In an aspect of the technique, the extruder is independently a twin-screw extruder or a single-screw extruder.

[00016] In an aspect of the technique, the twin-screw extruder is co-rotating or counter-rotating.

[00017] In an aspect of technique, the extruder independently contains at least 2 vent ports.

[00018] In an aspect of the technique, the extruder independently contains at least 3 vent ports, such as 4, 5, 6, 7, 8, 9 or 10 vent ports.

[00019] In an aspect of the technique, the extruder vent ports are at atmospheric pressure (*i.e.*, not subjected to vacuum conditions) or, alternatively, are subjected to vacuum conditions. In the case of multiple vent ports, a portion of the vent ports may be at atmospheric pressure and a portion of the vent ports may be subjected to vacuum conditions.

[00020] In an aspect of the technique, the twin-screw extruder has a length to diameter ratio of the screw shaft in the range of 24:1 to 56:1.

[00021] In an aspect of the technique, the twin-screw extruder has a length to diameter ratio of the screw shaft in the range of 36:1 to 48:1.

[00022] In an aspect of the technique, one or more additives, fillers, reinforcing agents or modifiers are introduced into the compounding extruder.

[00023] In an aspect of the technique, fiberglass is compounded with nylon66 in a range of about 5% to about 60% by weight such as about 10% to 55%, such as 15% to 50%, such as 20% to 50%, such as 25% to 50%, such as 35% to 45%.

[00024] In an aspect of the technique, the vent ports are used for removal of moisture or impurities from the compounded polyamide.

[00025] In an aspect of the technique, only a portion (such as, for example, greater than 0% up to about 99%, such as between about 5% up to about 95%, such as between about 15% up to about 85%) of the molten or substantially molten polyamide feedstock is sent directly to the one or more polymer compounding devices.

[00026] In an aspect of the technique, the polymerization process is located less than 500 feet from the polymer compounding device.

[00027] In an aspect of the technique, the compounded polyamide has a relative viscosity (RV) range between about 35 and about 120, such as between about 40 and about 100, such as between about 40 and about 80, such as between about 45 and about 60.

[00028] An aspect of the technique is a polyamide prepared by a process as described herein.

[00029] An aspect of the technique is a compounded polyamide having a plate out value of less than about 3 mg/1,000 shots, such as less than about 2.5 mg/1,000 shots, such as less than about 1.5 mg/1,000 shots, such as less than about 1.0 mg/1,000 shots, such as less than about 0.5 mg/1,000 shots, such as less than about 0.3 mg/1,000 shots, with the lowest value being 0 or a value greater than zero such as, for example, 0.01 mg/1,000 shots or 0.05 mg/1,000 shots or 0.1 mg/1,000 shots.

[00030] An aspect of the technique is a compounded polyamide exhibiting greater than about 200 shots before detection of burn material in the injection mold, such as greater than about 300 shots, such as greater than about 400 shots, such as greater than about 500 shots, such as greater than about 600 shots, such as greater than about 700 shots, such as greater than about 800 shots, and including ranges such as between about 400 to about 800 shots before detection of burn material, such as between about 500 to about 800 shots such as between about 600 to about 800 shots.

[00031] In an aspect of the technique, the compounded polyamide has a relative viscosity (RV) range between samples of less than about 3 units, such as less than about 2 units, such as less than about 1 unit, such as less than about 0.8 units, such as less than about 0.5 units.

Brief Description of the Drawings

[00032] The figures represent exemplary embodiments and are not intended to limit the scope of the present invention as otherwise described herein.

[00033] **Figure 1** represents a schematic of a molten stream fed compounding operation suitable for use in the present technique. Raw materials, comprising hexamethylene diamine (HMD), adipic acid, water, catalyst, modifiers, stabilizers and additives, are fed to a

conventional continuous polymerization process where a nylon polymer comprising PA66 is produced at RV levels between about 35 to about 200. This molten polymer is not allowed to freeze for the purpose of pellet formation or for any other solid method of production – *i.e.*, no significant solidification of the molten polymer is allowed to occur between the continuous polymerization and the subsequent compounding processes. Instead, the molten stream is fed directly to compounding equipment in proximity where any of many types of additives, minerals, reinforcers, modifiers, fillers, *etc*., are mixed into the polymer to form a new compounded product. This new compounded product is processed through a pelletization system, stored, conveyed and packaged in any combination of conventionally employed containers in the industry.

[00034] **Figure 2** represents a schematic of a molten stream fed compounding operation using a twin screw extruder as the compounding device. Molten polymer from the continuous polymerization process flows directly in to the compounding twin screw extruder. Multiple vent ports are provided for progressing the incremental decrease of absolute pressure in the venting zones. Combi-barrels are used to introduce solid type additives, modifiers, fillers, reinforcers, *etc*., and injection ports are used to introduce those same components most appropriately introduced in liquid form. Following the compounding unit, the molten product stream is typically fed to a stranding and pelletizing unit with drying and moisture control capabilities known to those skilled in the art. Following the pelletization, the compounded product is conveyed, stored, and packaged in any standard packaging container available for compounded plastics products.

[00035] **Figure 3** represents a schematic of a multi-barrel extruder suitable for use in the present technique as the compounding process. While any of several barrel and screw designs are possible for producing the various compounded products capable of being produced by the described system, the depicted combination has been observed to be highly effective for producing superior plate-out results with 50% glass-filled nylon PA66 with a target RV of 48 and a moisture content of 0.15% by weight.

Detailed Description

Definitions

[00044] As described herein, “weight stability” is a measure of the mass consistency from shot-to-shot in a molding process.

[00045] As defined herein, “warpage” is a measure of the dimensional deviation of a molded part to that of the mold.

[00046] As defined herein, “scrap rate” is a measure of the amount of out-of-spec parts that are produced on a molding process.

[00047] Various embodiments provide a process for producing an improved compounded polymer such that the compounded polymer exhibits less variability in viscosity, and when used in molding or extrusion operations, results in less injection molding deposits (“plate-out”), thus producing better quality molded parts and extending the operational life of the mold (*i.e.*, the machinery) between overhauls required to remove the deposits. In conventional compounding operations, the polymer (*e.g.*, a polyamide) feedstock is supplied to an extruder in a solid form. In contrast, various embodiments introduce a polyamide feedstock in molten form (*e.g.*, from a continuous or batch polymerization process) directly into polymer compounding equipment in a process referred to herein as inline compounding (“ILC”). In an exemplary embodiment, the continuous or batch polymerization process is in proximity to the polymer compounding equipment (*e.g.*, an extruder), such as within 1,000 feet, such as within the same building, such as within 500 feet, such as within 300 feet, such as within 200 feet, such as within 100 feet, such as within 50 feet, such as within 20 feet. By introducing a polyamide feed to polymer compounding equipment where the polyamide feed is already in molten form, additional extruder barrels, normally used for melting the resin, can alternatively be used for increasing vacuum exposure. This increased vacuum exposure in turn results in increased impurity removal and a cleaner product. An additional benefit resulting from applying vacuum in more zones of the polymer compounding equipment (*e.g.*, an extruder) is that the final vacuum zone can operate at a lower pressure (*i.e.,* at a decreased equipment cost) using common vacuum equipment, due to the significant removal of impurities that has already occurred in prior zones.

[00048] In addition, the continuous polymerization or batch polymerization molten feed stream to the polymer compounding equipment results in a compounded polymer with improved

viscosity and/or moisture stability. In an exemplary embodiment, the polyamide feed to extrusion requires less than one hour (such as between 1 and 50 minutes, such as between 1 and 40 minutes, such as between 1 and 30 minutes, such as between 1 and 20 minutes, such as between 1 and 10 minutes) in melt transit to the extruder via a transfer pipe. This molten stream is low in melt history and heat cycles as it is not solidified (in contrast to when the polymer feed stock is present in a solid form) prior to use in the compounding operation. Having at least one less product handling system in place between the polymerization and compounding operations reduces the chance of contamination or foreign body inclusion. Yield improvement and labor cost reduction are also realized with the present technique.

[00049] Continuous polymerization processes such as those employed by the present techniqueare well known. See, *e.g.*, U.S. Patent Nos. 3,113,843; 3,947,424; 4,851,466; 5,674,974; and Handbook of Fiber Chemistry (2007) 3rd ed., CRC Press, pp. 35-77.

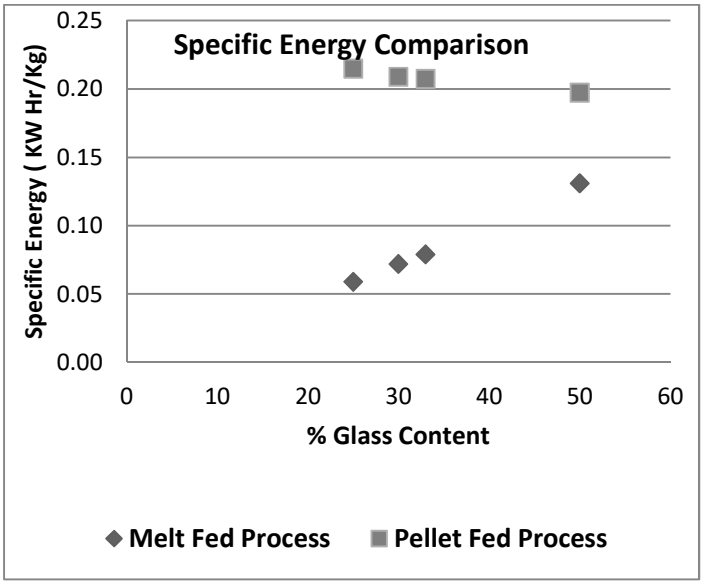
[00050] Batch (also referred to as "clave" or "autoclave") polymerization processes are also suitable for use in the present technique, *e.g.*, as a molten feed directly introduced to a polymerization compounding device. Batch polymerization processes are well known. See, *e.g.*, U.S. Patent Nos. 4,851,466; 5,245,005; and 4,032,517.

[00051] Known injection molding, extrusion molding, blow molding, press molding, compression molding and gas assist molding techniques are generally suitable for molding of the compounded polyamides. See, *e.g.*, U.S. Patent No. 8,658,757; 4,707,513; 7,858,172; and 8,192,664.

[00052] The compounded polymer prepared by the ILC process shows improved molding performance with significantly lower plate out and longer mold life between cleanings compared to conventionally prepared compounded polymers where the feedstock is introduced to the polymer compounding equipment in solid form. The viscosity and quality control ranges for such a product are also less variable than that resulting from conventional pellet fed compounding. This corresponding improvement in quality directly translates into a superior product for the customer in terms of improved performance and lower cost compared to products prepared from a conventional compounding operations using feedstock in solid form.

[00053] Also, because the feedstock is conveyed in a molten state to the polymer compounding equipment, the polymer compounding equipment motor size can be reduced since the initial energy required to melt the feedstock is avoided. This advantage translates to lower investment costs and lower energy consumption per pound of product during preparation of the compounded product. In the exemplary embodiment where polyamide PA66 is compounded to provide a product comprising 35% fiberglass, the extruder motor size reduction is approximately 62%. In an exemplary embodiment, the molten polyamide feed stream is introduced directly from a continuous or batch polymerization process to a first portion of a twin-screw extruder with no solidification processing steps prior to introduction to the extruder. By introducing a molten feed to the extruder, the energy consumption of the extruder is reduced relative to an extruder receiving and compounding a solid feedstock. In a particular embodiment, it has been determined that for production of 33% fiberglass-filled nylon, the extruder required a specific energy of 0.071 kw-hr/kg with a molten feedstock, compared to the significantly higher 0.21 kw-hr/kg with conventional pelletized feedstock. This significant difference in energy can be largely attributed to the energy required to initially melt the incoming solid nylon feedstock. Additional savings occur with molten feedstock directly introduced to the extruder since the cumulative costs to pelletize, package, store and handle the feedstock are not incurred. Generally, between about 5 and about 10 cents per pound are saved by implementation of the present technique that consists of a reduced number of steps compared to conventional technologies. Lower feedstock working volumes and lower labor requirements are also included in these savings. In the exemplary embodiment where polyamide PA66 is compounded to provide a product comprising 50% fiberglass, the extruder motor size reduction is approximately 35%. Table 1 shows an exemplary energy comparison.

Table 1



[00054] Since the step of melting the solid feedstock provided to the polymer compounding equipment is no longer necessary in view of the feedstock arriving from the continuous or batch polymerization process in a molten state, the performance of the polymer compounding equipment is enhanced for mixing additives, fillers, reinforcing agents and modifiers, resulting in more flexibility in, for example, the screw of an extruder. For the same cost investment of a typical pellet fed compounding extruder, the molten fed extruder of the present technique is able to achieve greater flexibility in producing higher volumes of different products due to the increased number of barrels available for additive feed and mixing. As a result, compounded products can be prepared in greater amounts and diversity using the molten fed compounding equipment of the present technique.

[00055] While a particular embodiment of this invention is demonstrated using nylon66, other nylons, other polyamides (such as poly(aminoundecanoamide), poly(aminododecanoamide), polyhexamethylene sebacamide, poly(p-xylylene-azeleamide), poly(m-xylylene adipamide), and polyamides from bis(p-aminocyclohexyl)methane and azelaic, sebacic and homologous aliphatic dicarboxylic acids) and the like are also envisioned to benefit from the process of the present technique. Copolymers of polyamides are also suitable for the process of the present technique. See, *e.g.*, U.S. Patent Nos. 3,236,914; 3,472,916; 3,373,223; 3,984,497; and 3,546,319.

performance is achieved with the present technique compared to conventional methods. Melt vacuum exposure time was determined according to the calculations below:

Variables:

N=number of screws

ID = inner diameter of screws (mm)

OD = outer diameter of screws (mm)

F = % fill in the vacuum section

L = length under vacuum (mm)

R = flow rate through extruder (kg/hr)

D = density of compound (g/cm^3)

Vf = volumetric flow rate = (R*1000)/(D*3600)

C = total cross sectional area = N* PI*(OD/2)^2-PI*(ID/2)^2

Cp = total cross sectional area filled with polymer given = C*F

Vv = total volume in vacuum section = Cp*L/1000

Vacuum Exposure Time = Vv/Vf (seconds)

The following constants were used for these calculations:

# screws:	2
ID:	70 mm
OD:	96 mm
Density:	1.6 g/cm^3
% Fill (F):	25%
Length under vacuum (L):	768 mm

Additional information relating to the devolatilization performance of a twin screw extruder can be found, for example, in Polymer Extrusion, (2001) 4th Edition, by Chris Rauwendaal, pp. 618-622. The high surface renewal and thin film generation in the extruder enhances devolatilization effectively with low overall residence time.

Table 2

Operating Conditions			Molding Performance	
Conventional Process				
Compounding Rate (lbs/hr)	Vacuum (in Hg)	Vacuum Exposure Time (s)	Shots Until Burning	Plate Out (mg/100 shots)
2500	25	1.52	>600 shots	1.8
3000	25	1.36	341	2.8
3500	25	1.21	318	2.7
3700 (high plateout conditions)	20	1.14	88	5.8
Improved ILC Process				
6000	28	2.75	>600 shots	0.77
6000	28	2.75	>600 shots	0.62

[00069] Reviewing the results of Table 2, it is apparent that the present technique results in significantly improved molding performance compared to the conditions in which the nylon feedstock is introduced in a pelletized (i.e., solid) form (conventional process). For example, a solid feedstock exposed to the least amount of vacuum time exhibited the lowest number of shots until burning (88) and the highest plate-out value (5.8). In contrast, the molding performance of the molten nylon feedstock (ILC process) showed no burning in the mold and, more significantly, the least amount of plate-out (0.7 or less) compared to all tests using solid feedstock. For the customer, these improvements translate into extended up-time on equipment since there are fewer overhauls of the molds, allowing the equipment to produce more parts with the same capital cost and lower operating costs. Product quality also is improved with less plate-out as the deposits can contaminate the product and impart surface defects on the produced molded parts. In customer molding applications exhibiting complicated or intricate mold venting manifolds, this benefit has even more impact in improving product quality and process output or yield. Cost savings and earnings for these plants could be increased as much as 50% or more given the potential for lower plate-out. In a particular

embodiment, a customer typically operates Monday through Friday producing injection molded parts by conventional means with a compounded product feedstock. Then they schedule a necessary cleaning of the molding equipment over the two-day weekend. Employing the low plate-out compounded product of the present technique as the feedstock, this molding customer can operate continuously for at least two weeks before needing to shut down to clean the their equipment, thus allowing for the significant increase in productivity of 2 days out of 14 days, an increase of about 15%.

[00070] In Table 3, comparisons are made with conventionally prepared products (standard pellet fed configuration) and a product prepared from the ILC process of the technique. Clearly, the plate-out results indicate that an inline molten feed produces a material substantially superior to that obtained from standard pellet fed operations. Unlike the products prepared from the pellet fed operations, the ILC product has substantially lower plate-out performance which would be realized as increased uptime and productivity for customers using this material.

Table 3

Source	Glass %FG	Plate-out mg/1000 Shots
Pellet-fed – High Plate-out Conditions	33	10.50
Pellet-fed – Low Plate-out Conditions	33	3.50
Competitor A nylon product	33	3.50
Competitor B nylon product	33	5.80
Continuous Polymerization (ILC) Molten Fed Commercial Line	33	2.00
Pellet-fed Normal Conditions	50	2.80
Competitor A nylon product	50	3.80
Continuous Polymerization (ILC) side stream Molten fed Pilot Line	50	0.90
Continuous Polymerization (ILC) Molten Fed Commercial Line	50	0.90

[00071] In Table 3, “ILC” refers to inline compounding. Competitor A and B products refer to commercial nylon products and are tested to illustrate the superior performance achieved using the ILC process of the technique with regards to plate-out.

What is Claimed Is:

1. A process for preparing a compounded polyamide from a molten polyamide feedstock, the process comprising:

preparing by a batch polymerization process or a continuous polymerization process a polyamide feedstock in a molten state;

introducing the molten polyamide feedstock directly to one or more extruders; and

mixing and/or blending the molten polyamide feedstock with one or more materials to provide the compounded polyamide,

wherein a range of relative viscosity (RV) between samples of the compounded polyamide prepared by the polymerization process is less than about 3 units.

2. The process according to claim 1, wherein the molten polyamide feedstock is a molten nylon66 feedstock.

3. The process according to claim 1, wherein the extruder is a twin-screw extruder.

4. The process according to claim 3, wherein the twin-screw extruder contains at least 2 vent ports.

5. The process according to claim 3, wherein the twin-screw extruder contains at least 3 vent ports.

6. The process according to claim 4, wherein the vent ports are at atmospheric pressure.

7. The process according to claim 4, wherein the vent ports are subjected to vacuum conditions.

8. The process according to claim 3, wherein the twin-screw extruder has a length to diameter ratio of the screw shaft in the range of 24:1 to 56:1.

9. The process according to claim 3, wherein the twin-screw extruder has a length to diameter ratio of the screw shaft in the range of 36:1 to 48:1.

10. The process according to claim 1, wherein only a portion of the molten feedstock is sent directly to the one or more polymer compounding devices.

11. The process according to claim 1, wherein the materials comprise one or more fibers, waxes, minerals, chain terminators, viscosity modifiers, plasticizers, heat stabilizers, UV stabilizers, colorants, catalysts, flame retardants, delusterants, fillers, reinforcing agents, antimicrobial agents, antistatic agents, optical brighteners, extenders and processing aids.

12. The process according to claim 1, wherein the polymer polymerization process occurs less than 50 feet from the one or more polymer compounding devices.

13. The process according to claim 1, wherein the range of relative viscosity (RV) between samples of compounded polyamide prepared by the polymerization process is less than about 2 units.

14. The process according to claim 1, wherein the range of relative viscosity (RV) between samples of compounded polyamide prepared by the polymerization process is less than about 1 unit.

15. A compounded polyamide prepared by the process of claim 1.

16. The compounded polyamide according to claim 15, having a plate-out value of less than about 2.0 mg/1,000 shots.

17. The compounded polyamide according to claim 16, having a plate-out value of less than about 1.0 mg/1,000 shots.

18. The compounded polyamide according to claim 15, having a relative viscosity (RV) range between samples of less than about 2 units.

19. The compounded polyamide according to claim 15 exhibiting greater than about 500 shots before detection of burn material in a mold for mold injection.

20. The compounded polyamide according to claim 16 exhibiting greater than about 500 shots before detection of burn material in a mold for mold injection.

21. The compounded polyamide according to any one of claims 15 to 20, wherein the polyamide is nylon66.