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(21) International Application Number: PCT/US99/05902 (22) International Filing Date: 18 March 1999 (18.03.99) (30) Priority Data: 60/078,392 18 March 1998 (18.03.98) US (71) Applicant: E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US). (72) Inventors: MARTENS, Marvin, Michael; 5602 Greenmont Place, Vienna, WV 26105 (US). KOSHIDA, Reiko; 2-14-19-608, Minowa, Kohoku, Yokohama 223 (JP). TOBIN, Wendy; 152 Sherman Street, Kingston, Ontario K7M 4G8 (CA). WILLIS, Joycelyn, M.; 544 Evangeline Avenue, Kingston, Ontario K7M 8R2 (CA). (74) Agent: HAMBY, William, H.; E.I. du Pont de Nemours and Company, Legal Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).		(81) Designated States: CA, JP, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: SYNERGIST FOR FLAME RETARDANT NYLONS (57) Abstract Antimony-free polyamide molding compositions are disclosed having improved flame resistance. These compositions comprise, in with percent, 20-77 % polyamide having a melting point of 215-340 °C; 10-60 % inorganic filler; 10-35 % of a flame retardant having 50-70 % bormine; 3-10 % zinc borate; and, 0-2 % of a heat stabilizer.		

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TITLE

SYNERGIST FOR FLAME RETARDANT NYLONS

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CROSS REFERENCE TO RELATED APPLICATION

This application claims the benefit of U.S. Provisional Application No. 60/078,392 filed March 18, 1998.

BACKGROUND OF THE INVENTION

Field of the Invention

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The invention relates to polyamide molding compositions having improved flame resistance. More particularly, the invention relates to antimony-free polyamide compositions with superior physical properties.

Description of Prior Art

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Typically industry uses a variety of antimony compounds (antimony oxides and sodium antimonate) as a synergist to promote the effectiveness of halogen containing flame-retardants and in nylon molding compositions. Industry has been trying for years to eliminate the toxic heavy metal antimony in flame retardant compositions, but has been unable to develop anything nearly as effective as antimony compounds.

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While looking for improved thermal stability in flame retarded high temperature nylons, it was surprisingly found that zinc borate could be used as a direct (equally effective) replacement for antimony compounds in polyamide compositions.

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Another unexpected side benefit of this discovery is a significant improvement in physical properties (i.e. tensile strength and elongation to break) by using the zinc borate as a synergist.

SUMMARY OF THE INVENTION

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There is disclosed and claimed herein antimony-free polyamide molding compositions with improved flame resistance, comprising, in weight percent,

- (a) 20 - 77% polyamide, having a melting point of 215 - 340°C,
- (b) 10 - 60% inorganic filler,
- (c) 10 - 35% of a flame retardant having 50 - 70% bromine,
- (d) 3 - 10% zinc borate, and
- (e) 0 - 2% of a heat stabilizer

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There is further disclosed and claimed herein processes for improving the temperature stability and flame resistance of polyamide resins, comprising the

steps of providing a molding composition as above and molding this composition to make a molded part. Molded parts made from such processes are also disclosed.

DETAILED DESCRIPTION OF THE INVENTION

5 The polyamide used in this invention may be 20-77% (all percentages are weight percent unless designated otherwise) of any polyamide that has a melting point of from about 215°C to about 340°C. An example of a suitable polyamide is a copolyamide composed of 20-80 mole % of units derived from hexamethylene terephthalamide and 80-20 mole % of units derived from hexamethylene
10 adipamide. This polyamide is referred to hereinafter as 6T/66 copolymer. Other suitable polyamides include polyamides composed of 20-80 mole % of units derived from hexamethylene terephthalamide and 80-20 mole % of units derived from hexamethylene sebacamide, hexamethylene dodecamide, hexamethylene isophthalamide, 2-methylpentamethylene terephthalamide, or mixtures thereof.

15 "Synthetic polyamide," as used herein, includes a polymer which is made by man, and does not include natural fibers such as wools or silks. By an "aliphatic polyamide" is meant a polymer which has repeat units which include amide groups in the main chain, and in which at least some, preferably at least 50 mole percent, of these amide groups (through the nitrogen atoms and/or carbonyl
20 carbon atoms of the amide groups) are connected to aliphatic carbon atoms. Preferred polyamides include nylon-6,6, nylon-6, nylon 6,12, and copolymers of nylon-6,6 and nylon 6. Nylon-6,6 and nylon-6, and copolymers thereof, are especially preferred and nylon-6,6 is more preferred.

25 There are no particular limitations on the process for the production of the copolyamide used in the composition of the present invention. It may be produced easily by ordinary melt polymerization. One method to produce the copolymer of this invention is an autoclave one-step polymerization process taught in US Patent No. 5,378,800 which is incorporated by reference herein. That process includes feeding to a reactor an aqueous salt solution of an admixture
30 of desired diacids and diamines, heating the solution under pressure, reducing the pressure, maintaining the reaction mixture at a pressure that is not greater than about atmospheric pressure, and discharging the polyamide from the reactor. An alternative process includes preparing a prepolymer and subjecting the prepolymer to solid-phase polymerization or melt-mixing in an extruder to increase the degree
35 of polymerization. The prepolymer is prepared by heating at 150°C-320°C an aqueous solution containing 6T salt (a salt formed from hexamethylenediamine and terephthalic acid) and 66 salt (a salt formed from hexamethylenediamine and

adipic acid). An alternative process consists of subjecting 6T salt and 66 salt directly to solid-phase polymerization at a temperature lower than the melting point.

5 The composition of the present invention contains 10-60% of an inorganic filler or reinforcing agent that includes, for example, fibrous reinforcement such as glass fiber and carbon fiber, glass beads, talc, kaolin, wollastonite and mica. Preferable among them is glass fiber. Glass fibers suitable for use in the present invention are those generally used as a reinforcing agent for thermoplastics resins and thermosetting resins. Preferred glass fiber is in the form of glass rovings,
10 glass chopped strands, and glass yarn made of continuous glass filaments 3-20 micron meters in diameter.

The resin composition of the present invention contains 10-35% of a flame retardant. It is a flame retardant based on brominated polystyrene and/or brominated poly-phenylene ether containing 50-70% by weight bromine. The
15 polystyrene has a weight-average molecular weight higher than 5000, preferably higher than 20,000, and more preferably higher than 28,000. The ether has a molecular weight of at least 6000. A preferred flame retardant is brominated polystyrene or polydibromostyrene.

20 As described above, the present compositions comprise 3-10% zinc borate, but no antimony compounds.

Also present may be up to 2% of a heat stabilizer such as copper iodide.

The copolyamide resin of the present invention may have incorporated in it a variety of additives such as an impact modifier, a viscosity modifier, pigment, dye, antioxidant, and heat resistance improver, in such amounts that they do not
25 harm its characteristic properties.

Moreover, the copolyamide resins of the present invention may include up to 5% of color concentrate (such as carbon black) as well as up to 1% of lubricant. Drip suppressants may also be present in amounts of up to 5%.

EXAMPLES

30 The present invention is illustrated by the following examples and comparative examples.

Samples of various compositions were produced on a commercial scale 58°mm twin screw extruder (Werner and Pfleiderer, Ramsey, New Jersey) at 600 pounds per hour. Product was analyzed on a thermogravimetric analyzer (DuPont
35 Instruments [Series 951], DuPont Company, Wilmington, Delaware) measuring weight retention at 340°C after a 30 minute hold. Also samples were tested for standard physical properties following standard ASTM methods.

The nylon used in Examples E 1- E 22 and Comparative Examples CE 1 - CE 3 was nylon 66/6T (45/55 molar %) with a melting point of about 315°C and in Example E 23 and Comparative Example CE 4 was nylon 66, melting point approximately 265°C. The antimony compound used is 70% antimony trioxide in
5 66 nylon.

The flammability rating was conducted using UL Test No. UL-94 dated April 19, 1995.

Table

Example (E) / Compar Example (CE)		Weight Percent of Compounds								Properties			
	Nylon	Glass	Base or Buffer ¹	Flame Retardant	Antimony Com-pounds	Heat Stabi-lizer	Surlyn ®	Lubri-cant	Color Con-centrate	TGA Retention (%)	Tensile Strength (psi)	Elonga-tion (%)	Flame Rating (1/32")
CE 1	40.0	30.0	3A, .15B	24.0 BR	5.3	-	-	0.2	-	82.4	25.7	2.1	V0
E 1	40.7	30.0	4.0A, .15B	24.0 BR	0.0	-	1.0	0.2	-	96.0	27.0	2.4	V0
E-2	40.5	30.0	4.0A, .15B	24.0 BR	0.0	0.2	1.0	0.2	-	95.8	27.6	2.5	V0
E-3	40.3	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.2	-	96.1	28.0	2.8	V0
E 4	39.5	30.0	4.0A, .15B	24.0 BR	0.0	0.2	1.0	0.2	1.0	95.9	25.9	2.1	V0
E 5	38.5	30.0	5.0A, .15B	24.0 BR	0.0	0.2	1.0	0.2	1.0	96.0	26.2	2.2	V0
E 6	38.5	30.0	4.0A, .15B, 1.0C	24.0 BR	0.0	0.2	1.0	0.2	1.0	96.1	26.0	2.1	V0
CE 2	39.1	30.0	.3B, .15B	24.0 BR	5.3	0.0	1.0	0.2	1.0	82.1	25.5	2.0	V0
CE 3	40.2	30.0	.3A	24.0 BR	5.3	0.0	0.0	0.2	-	88.8	24.6	2.0	V0
E 7	40.4	30.0	4.0A	24.0 BR	0.0	0.4	1.0	0.2	-	96.4	27.0	2.4	V0
E 8	40.2	30.0	4.0A	24.0 BR	0.0	0.4	1.0	0.4	-	96.7	26.9	2.5	V0
E 9	40.1	30.0	4.0A	24.0 BR	0.0	0.4	1.0	0.5	-	96.3	25.7	2.3	V0
E 10	40.3	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.2	-	96.4	27.3	2.4	V0
E 11	40.1	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.4	-	96.1	27.7	2.4	V0
E 12	40.0	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.5	-	96.1	27.4	2.4	V0
E 13	36.9	35.0	4.0A	22.5 BR	0.0	0.4	1.0	0.2	-	96.8	27.6	2.1	V0
E 14	51.2	15.0	5.0A	27.0 BR	0.0	0.4	1.3	0.2	-	96.4	20.5	2.8	V0
E 15	39.4	30.0	4.0A	24.0 BR	0.0	0.4	1.0	0.2	1.0	96.8	26.3	2.2	V0
E 16	39.2	30.0	4.0A	24.0 BR	0.0	0.4	1.0	0.4	1.0	97.0	25.4	2.2	V0
E 17	39.1	30.0	4.0A	24.0 BR	0.0	4.0	1.0	0.5	1.0	96.6	25.9	2.2	V0
E 18	39.3	30.0	4.0A, 15B	24.0 BR	0.0	0.4	1.0	0.2	1.0	96.8	27.2	2.3	V0
E 19	39.1	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.4	1.0	96.4	26.6	2.2	V0
E 20	39.0	30.0	4.0A, .15B	24.0 BR	0.0	0.4	1.0	0.5	1.0	96.7	27.3	2.3	V0
E 21	35.9	35.0	4.0A	22.5 BR	0.0	0.4	1.0	0.2	1.0	96.3	26.8	2.0	V0
E 22	49.2	15.0	5.0A	27.0 BR	0.0	0.4	1.3	0.2	1.0	97.5	20.7	2.7	V0
CE 4	43.0	25.0	-	23.5 BR	7.0	-	1.5	-	-	63.0	27.0	2.8	V0
E 23	45.0	25.0	5.0A	23.5 BR	-	-	1.5	-	-	95.0	26.7	2.8	V0

Components: A Zinc Phosphate, B Hydroxy-Terminated Polybutadiene, C Polyethylene Glycol, D Polyethylene Glycol, E Polyethylene Glycol, F Polyethylene Glycol, G Polyethylene Glycol, H Polyethylene Glycol, I Polyethylene Glycol, J Polyethylene Glycol, K Polyethylene Glycol, L Polyethylene Glycol, M Polyethylene Glycol, N Polyethylene Glycol, O Polyethylene Glycol, P Polyethylene Glycol, Q Polyethylene Glycol, R Polyethylene Glycol, S Polyethylene Glycol, T Polyethylene Glycol, U Polyethylene Glycol, V Polyethylene Glycol, W Polyethylene Glycol, X Polyethylene Glycol, Y Polyethylene Glycol, Z Polyethylene Glycol, AA Polyethylene Glycol, AB Polyethylene Glycol, AC Polyethylene Glycol, AD Polyethylene Glycol, AE Polyethylene Glycol, AF Polyethylene Glycol, AG Polyethylene Glycol, AH Polyethylene Glycol, AI Polyethylene Glycol, AJ Polyethylene Glycol, AK Polyethylene Glycol, AL Polyethylene Glycol, AM Polyethylene Glycol, AN Polyethylene Glycol, AO Polyethylene Glycol, AP Polyethylene 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¹Components: A - Zinc Borate; B - Hydrotalcite; C - Calcium Oxide.

Heat Stabilizers - Variety of Organic and inorganic common heat stabilizers

Surlyn® is ethylene copolymer ionomer by E. I. DuPont, Wilmington, Delaware

CLAIMS

What is claimed is:

1. An antimony-free polyamide molding composition with improved
5 flame resistance comprising, in weight percent,
 - (a) 20-77% polyamide, having a melting point of 215-340°C,
 - (b) 10-60% inorganic filler,
 - (c) 10-35% of a flame retardant having 50-70% bromine,
 - (d) 3-10% zinc borate, and
 - 10 (e) 0-2% of a heat stabilizer.
2. The composition of Claim 1 wherein the polyamide (a) is a
copolyamide composed of 20 - 80 mole % of units derived from hexamethylene
terephthalamide and 80 - 20 mole % of units derived from hexamethylene
15 adipamide.
3. The composition of Claim 1 wherein the inorganic filler (b) is selected
from the group consisting of glass fiber, carbon fiber, glass beads, talc, kaolin,
wollastonite and mica.
20
4. The composition of Claim 1 wherein the flame retardant (c) is either
brominated polystyrene or polydibromostyrene.
5. The composition of Claim 1 wherein the heat stabilizer (e) is copper
25 iodide.
6. A process for improving the flame retardance of a polyamide resin,
comprising the steps of:
providing a molding composition of Claim 1, and
30 molding the molding composition to make a molded part.
7. A molded part made from the process of Claim 6.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 99/05902

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C08K3/00 C08K13/02 C08L77/00 //(C08K3/00,3:00,3:38),
(C08K13/02,3:00,3:38,5:00)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08K C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 122 699 A (ICI PLC) 24 October 1984 see table 1 see claims 1,4 ---	1-7
X	EP 0 410 301 A (BASF AG) 30 January 1991 see page 6, line 17 - line 18 see claim 1 ---	1-7
A	EP 0 055 893 A (DU PONT) 14 July 1982 see claim 1 -----	1-7

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

30 June 1999

Date of mailing of the international search report

09/07/1999

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 99/05902

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