

PLASTICS MATERIALS AND ARTICLES CONTAINING ELECTRICALLY
CONDUCTIVE FIBERS

5 The present invention relates to plastics materials
and articles containing electrically conductive fibers.
In particular it relates to plastics materials and
articles having such fibres substantially uniformly
distributed therein at a very low concentration, and
10 also to specific intermediate plastics products useful
in the manufacture of such materials and articles.

 The incorporation of electrically conductive
fibers in plastics is well known, for example for
the purposes of reinforcement and/or for improvement
15 of electrical and/or thermal conductivity.
However, for some time there has been concern about
the environmental hazards of various kinds of electromag-
netic radiation, in particular those with high frequencies
such as radar waves and microwaves and those produced
20 by signals used in electronic circuits, e.g. in digital
devices. The use of radio-frequency and high-frequency
electromagnetic radiation will grow in the future
as a consequence of the widespread application of
microprocessors, digital calculators and weighing
25 scales for cash registers, electronic typewriters,
personal and business computers with associated peripherals,
electronic toys and games, military equipment, and
other electronic devices.

 When such devices are housed in metal boxes,
30 the metal itself affords protection against emission
of radio-frequency and high-frequency radiation since
it reflects the emitted radiation off the interior
surface into the internal space. Interference with
and disturbance of radio, television or other electronic
35 waves are thus avoided.

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There is however a trend to replace metallic boxes by plastics housings. So far, it has been customary to apply electrically conductive coatings to these plastics housings to provide a shield against the emission of electromagnetic radiation but a drawback of such coatings is that they are not very durable. Moreover, in most cases, these coatings require special and expensive processing and application methods.

Attempts at imparting electrical conductivity to the plastics themselves (so that they shield against electromagnetic waves) have been made by the incorporation and dispersion of relatively large quantities of conductive fillers. Such conductive fillers include carbon black, aluminium flakes, cut wire, metal coated glass fibers, wire meshes and carbon fibers. However, drawbacks are associated with these conductive fillers. Some fillers do not permit sufficient dispersion in the plastic matrix and clog together or degrade to very small particles so that their shielding effect is much reduced. This degradation makes it necessary to add a great amount of conductive particles, which renders a uniform dispersion even more difficult while having an adverse effect on the mechanical properties of the material.

Finally, it is known that for effective shielding against electromagnetic radiation the conductive particles in the plastic matrix must possess a large aspect ratio, i.e. length-to-diameter (L/D) ratio; these particles must form a substantially continuous conductive network in the matrix in order to increase the conductivity without, however, greatly changing the physical and mechanical properties of the plastic matrix.

It is an object of the present invention to provide plastics materials and articles which are at least in part electrically conductive and which have relatively low contents of fine, uniformly dispersed electrically conductive fibers.

According to one feature of the present invention, there is provided a plastics material which is at least in part electrically conductive, said part having electrically conductive fibers having a specific conductivity of at least 0.5% of the copper standard randomly and substantially uniformly distributed therein, the fibers having an equivalent diameter (D) and a length (L) such that for a major proportion of the fibers the ratio D/L is from about 0.0005 to about 0.008, and the volume concentration percentage (C%) of the fibers in said part being less than about 0.5%.

Further features of the present invention include the following:

- (i) a plastics article consisting at least in part of a plastics material according to the invention as hereinbefore defined.
- (ii) A plastics grain for use in the production of plastics materials and articles according to the invention as hereinbefore defined having a length between about 0.4cm and about 1.2 cm with electrically conductive fibers distributed therein.
- (iii) a thread for use in the production of plastics grains according to the invention as hereinbefore defined comprising a bundle of conductive fibers embedded in plastic, the fiber content being from 20 volume percent to 70 volume percent and the fiber diameter being not greater than about 0.015 mm.

The present invention thus for example provides plastics article with less than about 0.5% volume of fine electrically conductive fibers, which are randomly and substantially uniformly distributed such that the distributed fibers provide a suitable conductivity in any direction in articles for use, for example, electromagnetic interference (EMI) shielding. The fibers can be uniformly distributed

throughout the body of the article, e.g. a plate or sheet, or alternatively may for example be only in certain predetermined parts of the article, e.g. adjacent to one or both planar surfaces of articles such as plates or sheets. The electrically conductive fibers preferably have an equivalent diameter of from about 0.002 to about 0.015mm.

The present invention can be used to provide articles in the form of or made at least in part from plastics material in sheet or plate form with a shielding effectiveness against electromagnetic radiation of at least 25 dB within a wide frequency range (e.g. between 0.1 and 10 GHz such as for example 1GHz) while maintaining their normal mechanical properties. Such articles are to be understood to include slats, various shaped profile cross-sections, foils, thin films, tubes, housings, bags, covers and other containers made at least in part from plate- or sheet- like materials.

In accordance with the present invention electrically conductive fibers are dispersed in the plastics material or article, such fibers having an equivalent diameter to average length ratio (D/L) which varies from about 0.0005 to about 0.008 for a major part of the fibers. These fibers may, for example, be metal fibers with an average length (L) between 0.5 mm and 5 mm. The term "equivalent diameter" (D) means the square root of the quotient of the surface area of the fiber cross-section divided by π . The average length (L) means the total sum of the lengths of the incorporated fibers divided by the number of fibers. At an average length of $L = 0.5$ mm, there will certainly be fibers with a length shorter than 0.5 mm. However, a major proportion of the fibers will have a length approximating to the average length.

According to the invention the specified fiber dimension limits have been found to meet the above-mentioned shielding requirements at an exceptionally low volume concentration percentage C (%) of conductive fillers, namely between about 0.03 volume percent and about 0.5 volume percent. The preferred volume concentration percentage (C%) is preferably from about 0.05 volume percent to about 0.5 volume percent. For articles in sheet or plate form wherein the thickness of the plate or sheet is less than 3 mm, it has been found that the above-mentioned shielding requirements are met when the concentration C lies within the limits defined by line 1 and the ordinate axis of Figure 4 of the accompanying drawings; for plate thicknesses between 3 mm and 6 mm the above-mentioned shielding requirements are met when the concentration C lies within the limits defined by line 3 and the ordinate axis of Figure 4 of the accompanying drawings. These low concentrations exert almost no influence on the appearance of the plastic articles.

It has been further discovered that antistatic plastics materials and articles can be produced by dispersing electrically conductive fibers in the plastic at still lower concentrations (less than about 0.3% volume), wherein the concentration C with respect to the fiber dimensions in the said antistatic plastic may even lie within the limits defined by line 3 and the abscissa of Figure 4 of the accompanying drawings, when the fibers are present at least next to the outer surface of the articles.

It is thus possible according to the invention to make plastics composite articles with low conductive fiber content by randomly and uniformly distributing electrically conductive fibers in the plastic such that the article has a predetermined level of conductivity. The conductive, fiber concentration can in general be varied between about 0.03% volume and about 0.5% volume. Optimum D/L limits can be reached by adding the fibers during the production of plastic articles in accordance with the process of the invention as hereinbefore defined. The D/L limits preferably satisfy the relationship whereby the concentration C lies within the limits defined by line 2 and the ordinate axis of Figure 4 of the accompanying drawings.

Since, in the plastics matrix, the contact between the fibers must be as good as possible to provide good electrical conductivity, it is important that they should possess a relatively smooth surface. This means that roughnesses on the fiber surface should preferably project above or extend under the average level of the fiber surface by less than about 1 μm . In this way it is statistically most probable that there will be the optimum number of contact surfaces between neighbouring fibers.

Stainless steel fibers, manufactured by a method of bundle drawing as described e.g. in the U.S. Patent No. 2,050,298 or No. 3,379,000, show particularly suitable intrinsic conductive properties for use in the present invention. Probably this is attributable to the fact that they are less prone to form an insulating oxide layer on their surfaces in contrast with, for example, aluminium or copper fibers. This means that the contact resistance in the fiber contact points remains low. Usually, they are also more inert than Al or Cu towards most plastics. Other fibers such as Hastelloy-X, Inconel, Ti or Ni may also be used. The specific conductivity of the fibers must be at least 0.5% of the copper standard.

In principle, the invention is generally applicable to thermoplastic and thermosetting materials and is preferably used in connection with plastics materials which can be formed by the usual processing techniques such as for example casting, extrusion, injection molding, press molding and foaming. Accordingly, the articles may have a flexible, rigid or elastomeric nature. It is readily applicable to thermoplastic resins and to use in conjunction with conventional shaping techniques such as extrusion and injection molding with the use of plastics pellets as a starting material. The conductive fibers may conveniently be added to the plastics pellets or be incorporated into these pellets so that their compatibility with the plastics is not threatened and an optimally uniform dispersion of the conductive fibers in the plastics is reached during conventional shaping processes. According to an important aspect of the invention, a uniform dispersion is obtained by using plastics grains as an intermediate product for the manufacture of the article whereby the grains are preferably at least about 0.4 cm long and loaded with conductive fibers. The average length of the fibers in the grains will slightly exceed that of the fibers in the final article since during the molding process a number of fibers are inevitably broken. Measures which can be used for counteracting this fiber breakage are described below.

The volume concentration of conductive fibers in the grains will generally be greater than the required final concentration in the molded article. If, for example, it is desired to manufacture an article comprising 100 percent of the above described grains and with a final concentration of 0.3 percent by volume of metal fibers in the article, then the average volume concentration of metal fibers in the grains should be at least 0.33 percent. If, however, it is desired to make an article with the same final concentration of metal fibers (0.3 volume percent) on the basis of a mixture of 67 percent by volume of pure plastics

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pellets and 33 percent by volume of plastics grains loaded with metal fibers, then the average volume concentration of metal fibers in these grains should preferably be at least 0.99 percent.

5 A process for the production of a plastics material which is at least in part electrically conductive is described and claimed in divisional application 8401468.

In general, processes for making plastics articles having predetermined conductive portions therein
10 conveniently include the following steps.

A fiber/plastics composite is provided having a conductive fiber content ranging from about 20% to about 70% vol. with a substantially parallel fiber arrangement therein. This composite is admixed with
15 a predetermined amount of substantially pure plastics material and the blend is introduced into the hopper of, for example, an extrusion mixer. In such apparatus the plastics material is heated to soften it and worked (kneaded) to evenly disperse the fibers therein.
20 Low shear forces are applied to avoid excessive breakage of the fibers; the shear forces must however be sufficiently high to uniformly distribute the fibers within the plastic. To form the article, the so worked viscous mass can then be further forwarded by an extruder
25 screw through suitable orifices, channels or slots to a mold or it can be directly and continuously extruded to rods, tubes, sheets, films or plates or be injection molded.

When using a mixture of pure plastics pellets
30 and composite grains which include fibers as described above, then cylindrical composite grains with a diameter at least equal to the average thickness of the pure pellets are preferably used. This measure usually reduces the proneness of the embedded conductive fibers
35 to break during the hot mixing and kneading of the grain pellet mix prior to the actual molding. The length of the composite grain will, preferably, be between about 0.4 cm and about 1.2 cm.

For practical considerations, it is useful to
40 provide plastics grains with standard dimensions and

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standard concentration and which can easily be mixed and processed with conventional plastics pellets in the desired proportion for obtaining a predetermined volume concentration of conductive fibers in the end product. Obviously, the plastics material of these grains will preferably be the same resin as that of the article to be formed. The cross-sectional surface of the composite grains will, moreover, preferably be at least equal to that of the pure resin pellets.

10 For example, a metal fiber volume percentage in the composite grains of 1 percent has proved to be suitable. The metal fiber content in the grains can conveniently vary between about 0.5% vol. and about 2% vol.

If desired, the composite grains may also contain plastics material different from that of the article to be made. The softening and melting point of the resin in the composite grains must, however, be not higher than that of the plastic from which the article will be made to enable the composite grains, at the processing and molding temperatures of the article, to spread easily and mix with the main plastics material used for the article, thereby allowing the conductive fibers to disperse therein under minimal shear forces.

The main plastics material of the article to be produced must also be compatible with the resin of the composite grains. For example, the latter resin must not disintegrate or react with the main plastics material of the article when the latter is heated to its processing and molding temperature.

30 The conductive fibers to be incorporated are conveniently in the form of a filament bundle, although other fiber bundles such as fiber slivers and staple fiber yarns can also if desired be used. The fiber lengths should be sufficiently long and the fiber

35 slivers should possess a sufficient yarn number or tex (titre) to form a properly coherent bundle with sufficient tensile strength for handling and processing. Average fiber lengths of 7 cm with approximately 2,000

fibers per sliver cross-section are suitable. Generally, the fiber bundles are embedded in a plastic matrix so that the fiber content therein is between 20 volume percent and 70 volume percent. The impregnated fiber bundle is allowed to stiffen (e.g. by cooling) in order to produce a so-called thread having a cross-sectional area preferably not smaller than and advantageously about equal to the cross-sectional size of the plastic pellets of the main raw material.

10 The thread may be round or have a variety of other cross-section shapes, e.g. oval, flattened or rectangular, to facilitate winding up and chopping into particles. The thread may comprise 35,000 adjacent filaments (or fibres) in its cross section, but a lower number (at least about 1,000 filaments) is advantageously used.

It is often preferably to envelop the impregnated bundle with a sheath made of either the same plastic as the main plastics material of the article to be produced or the same or another plastic as that with which the bundle has been impregnated. This assists with the gradual disintegration of the cut bundle and the uniform dispersion of the fibers in the plastic matrix while mixing at high temperatures. The thread is chopped into predetermined lengths, referred to hereinafter as granules, with such lengths ranging from 0.4cm to 1.5cm.

It is evident that the plastics material with which the fiber bundle has been impregnated and sheathed must be compatible with the main plastics material of the article to be formed. For example, where the latter plastics material is a thermoplastic material the impregnating resin is preferably a relatively low molecular weight thermoplastic polymer such as a polyethylene, polypropylene, polyester, polyacrylate, polymethacrylate, polystyrene or polyvinyl chloride homopolymer or copolymer.

The thermoplastic grains with the conductive fibers dispersed therein are conveniently prepared by making a dry mix of pure plastics pellets (the main plastics material) and a number of granules in which an appropriate quantity of parallel fibers are embedded, the fibers possessing approximately or predominantly the same lengths as the granules. This mix is subsequently kneaded in an extrusion mixer at elevated temperature and with the application of low shear forces in order to disperse the conductive fibers in the plastics material. Thereafter, the soft mass is extruded into one or more threads with suitable cross-sections and cooled. Finally, the threads are transversally chopped into grains with lengths of at least about 0.4cm.

To manufacture articles of thermoplastics foam material in molds, a predetermined mix of pure plastics pellets containing an appropriate amount of blowing agent may be used. It is also possible to mix the blowing agent in powder form with pure plastics pellets, together with a suitable amount of composite grains.

For example, the pellets can be moistened so that the powder sticking to them can spread sufficiently evenly over them. Afterwards, the mixture can be fed to the injection molding machine in the usual manner.

For the preparation of thermoplastics elastomer articles (e.g. comprising an elastomeric polyester such as Hytrel), elastomer pellets can be used mixed with a suitable proportion of composite grains prepared on the basis of the same elastomer. However, the shear forces must be particularly low during the kneading and molding processes.

For sheet molding pre-impregnated fiber sheets (prepregs), it is possible as a preliminary step to disperse the conductive fibers in the liquid resin in an appropriate concentration. For bulk molding viscous mixtures

of resin and fibers, the conductive fibers can be dispersed in the mass in a similar way.

In particular, it is possible to mix the conductive fibers first with other fibers, e.g. reinforcing fibers such as glass fibers, carbon fibers or polyaramid fibers, and to disperse this fiber mix in some way in the resin. For processing into thermoplastic resins, it is possible to replace the aforescribed thread of conductive fibers embedded in plastics by a thread comprising a mixture of glass fibers and conductive fibers in the desired proportion. It is also possible to impregnate glass fiber bundles in a side-by-side disposition with bundles of conductive fibers to form the thread. Finally, it may be preferred to mix threads comprising reinforcement fibers and cut into granules with granules containing conductive fibers in an appropriate weight proportion and to feed them to the molding machine, while adding, if so desired, a suitable quantity of pure plastics pellets (main plastics material).

An advantageous method of distributing in the plastic a very low percentage of conductive fibers such as metal fibers, consists of starting with a blended sliver comprising thermoplastic textile fibers with a relatively low melting point intermingled with a desired percentage of such metal fibers. The blended sliver is then impregnated, or impregnated and coated with e.g. a relatively low molecular weight polymer to obtain a thread which, after solidification, is further chopped to granules. When adding the granules to the plastics pellets and hot working the mixture, the thermoplastic textile fibers in the granules are softened and disappear in the plastic matrix. The step of preblending the metal fibers among said textile fibers enables a better separation of the metal fibers in the plastic and eliminates any occurrence of metal fiber clusters during the hot kneading process prior to molding.

Certain additives may if desired be incorporated into the plastic for example to improve the electrical conductivity of the plastic or to facilitate the dispersion of the conductive fibers during processing or both.

- 5 Some flame retarding agents added during compounding of the raw plastic material have, in combination with the incorporation of stainless steel fibers in plastics as described above, improved the properties of the materials according to the invention as shields against
10 radio frequency and high frequency waves.

- Stainless steel fibers are used in the examples below. Other electrically conductive fibers are, in principle, also applicable, e.g. glass fibers with a metal coating provided that the dispersion process in the plastic
15 matrix can take place under sufficiently low shear forces in order to counteract the proneness or tendency of the fibers to break. When injection molding is used, it may be necessary to adapt the conditions e.g. by modifying the rheology of the plastics during
20 injection molding and the injection speed. The diameter of the extrusion orifice should preferably be at least twice the thickness of the plate to be molded.

- In addition to the polymers described in the examples below, numerous other resins can be used
25 in producing the finished product which incorporates conductive fibers. These include but are not limited to polycarbonates, polyacetates, polyarylates, polyvinyl-chloride, fluoro polymers such as polyvinylidene-fluoride, polyolefins, polyacetals and polystyrene.
30 The invention will now be further described by means of the following non-limiting examples with reference to the accompanying drawings, in which:

- Figure 1 is a partial perspective view of the formative and finished stages of a thread formed from
35 an impregnated and sheathed bundle of conductive fibers and a granule cut from this thread;

Figure 1A is a partial perspective view of a thread as in Figure 1 but having a flattened cross-section;

Figure 2 is a drawing of a plastic grain containing
5 dispersed conductive fibers;

Figure 3 is a graphic representation of the relationship between the wave frequency (f) of electromagnetic radiation and the shielding effectiveness (SE) of a 3mm-thick plastic plate containing conductive
10 fillers; and

Figure 4 is a graphic representation of the optimal field of operation for the invention in terms of fiber concentrations and D/L ratios.

Example 1

15 With reference first to Figure 1, a substantially round, not twisted, bundle 1 of 20,400 stainless steel filaments, AISI 316L of the type BEKINOX[®] (trademark of applicant) with an equivalent filament diameter of 0.008 mm, was passed through a bath, containing
20 a solution of 20% by weight of a relatively low molecular weight linear polyester (M.W. circa 14,000) of the type Dynapol L850 (Dynamit Nobel) in trichloroethylene. After leaving the bath, the bundle was pulled through a round stripping orifice, with a diameter of 1.8mm,
25 then dried. The dried bundle thus comprised 6.2 percent by weight of resin (which equals 70 percent by volume of metal fibers). The impregnated bundle was enveloped in a wire sheath extruder (type Maillefer with fixed centering) with the same polyester Dynapol L850.
30 The round extrusion nozzle had a diameter of 2mm. After the thus extruded thread 2 had cooled down, it was chopped into cylindrical granules 3 with a length of 1 cm. The granules comprised approximately 13 percent by weight of resin which equals approximately

52 percent by volume of metal fibers. When cutting the bundle almost no metal fiber ends were pulled out of it and hook formation and flattening of the fiber ends was avoided. This was important to assure a reliable dosage and fluent dispersion. Then the granules were dry mixed by tumble blending techniques with the usual thermoplastic pellets of various kinds of resins in the proportion of 9.75 percent by weight of granules to 90.25 percent pure plastic pellets and extruded into a substantially round thread with a diameter of 4mm and a metal fiber content of approximately 8 weight percent. After cooling, this extruded thread was cut again into grains 4 (Figure 2) with a length of 1cm. In these grains, the metal fibers appeared to be evenly dispersed with a volume content of about 1.1 percent. The shear forces encountered during extrusion were held sufficiently low so that excessive fiber breakage was avoided. One of the measures applied to keep the shear forces down to a minimal level involved the removal of the filter plates at the inlet of the nozzle. The temperature at the nozzle of the single-screw extruder was 260 degrees centigrade when NORYL-SE90 (a modified polyphenyleneoxide of General Electric) was used. When using Cycolac AM1000AS (an ABS resin of Borg Warner) the extrusion temperature at the nozzle was 220 degrees centigrade. When using Lexan L13848-141R-111 (a polycarbonate of General Electric) it was 225 degrees centigrade. The extruder was of the type Samafor 45 with a length-to-diameter ratio of the screw equal to 25. The feeding channel in the head next to the extrusion orifice was a ring like space between a tapering outer surface of a mandrel and the concentrically arranged conical inner surface of the nozzle head. The channel was thereby confining towards the extrusion orifice and shear was thereby somewhat increased and this resulted in a better fiber dispersion whereby

the fibers were more or less oriented in the extrusion direction.

The thus obtained composite grains were dry mixed with an equal weight quantity of pure plastics pellets and fed to an injection molding machine of the Ankerwerk V24/20 type with a screw to which a mold was connected for molding plaques with a thickness of 2.3mm, a length of 30cm, and a width of 25 cm. The temperatures in the screw chamber were respectively 250 degrees centigrade, 210 degrees centigrade and 290 degrees centigrade, for the Noryl, Cycolac, and Lexan resins, and the temperature of the molds was set at respectively 80 degrees centigrade, 50 degrees centigrade and 90 degrees centigrade. The screw rotated at 44 revolutions per minute. The nozzle opening had a diameter of approximately 1cm. The Noryl-, Cycolac- and Lexan-plates had smooth surfaces and the fiber dispersion or distribution throughout the plates was even. The concentration of metal fibers amounted to 4 weight percent or 0.5 volume percent. The Bekinox[®] stainless steel fibers had a specific conductivity of about 2% of the copper standard.

Example 2

Under similar conditions as in Example 1, injection molded plates were made of the thermoplastic resins mentioned above. However, a flat bundle of 20,400 adjacent Bekinox[®] filaments with a diameter of 0.008 mm was used as is shown in Figure 1A. As in Example 1, the flat bundle was again impregnated with a Dynapol L850 solution and stripped through a rectangular 5mm x 0.5mm orifice. The dried bundle comprised 6.4 percent by weight of resin and was enveloped with the same polyester resin in a slot extruder at 160 degrees centigrade. The dimensions of the rectangular extrusion nozzle were 5mm x 0.6 mm and the obtained cooled strand comprised 23 percent by weight of resin which equals approximately 39 percent by volume of metal fiber.

The flat thread was chopped in 1cm lengths whereby hook formation and flattening of the fiber ends were absolutely avoided. Clamping of the fibers in a flat bundle in the resin matrix for the sake of accurately cutting the granules proved to be very effective. The obtained flat granules were then, without any difficulty, dry mixed with pure plastics pellets in a ratio between 10.66 and 89.33 weight percent and extruded into a substantially round thread with a diameter of 4 mm (see Example 1). The metal fiber content amounted to approximately 8 weight percent which corresponds to approximately 1.1 volume percent. Composite grains with a length of 1 cm were cut from this thread. After dry mixing these composite grains with an equal weight of pure plastic pellets and injection molding of the mixture as described above, an even dispersion was observed. The average fiber length was estimated at approximately 1.5 mm and the final concentration again amounted to 0.5 volume percent. See area A in Figure 4.

The shielding behavior against electromagnetic radiation of the injection molded plates was tested. As known, the shielding behavior of a plastic material loaded with conductive filler can be determined in proportion to the plate thickness by comparing the reflection R (%) measured at one radiation frequency (e.g. 10 GHz) with the reflection (100%) on a reference material such as a metal plate. If the electrical properties of the material are sufficiently homogeneous and the conductive filler in the plastic forms a network with a sufficiently small mesh size (e.g. of an order of magnitude smaller than the wave length of the radiation to be shielded), then the shielding behavior can be extrapolated for the full frequency range. Moreover, it is known that for a great number of applications of electrically conductive plastics, the shielding requirements are met when a shielding effectiveness (SE) of 25 dB is

obtained at a frequency of 1 GHz. It was also found that the SE value for electric fields and for materials with a specific resistance between 0.01 Ω cm and 100 Ω cm always is minimal in the vicinity of 0.4 to 5 GHz for plate thicknesses between 1 and 6 mm and with a distance of approximately 1 cm to 10 cm between the wave source and the plastic plate. A relationship between the shielding effectiveness SE and the wave frequency f is shown in Figure 3 for a plate thickness of 3 mm and a distance between the source and the plate of 1 cm. Curve 1 refers to the relationship for reflection values R = 99%, measured at 10 GHz, whereas curve 2 shows the relationship for R = 70%, again at 10 GHz. If, for example, for a conductive plastic plate with a thickness of 3 mm, a reflection R is measured of 80% at 10 GHz (source-to-plate distance is 1 cm), then it can be derived from Figure 3 that the SE value will be at least 35 dB at any frequency. When R= 70% and 1 GHz, then SE =38 dB.

Analogously, the following values hold for other plate thicknesses, measured at a distance of 1 cm between source and plate:

Thickness (mm)	10GHz		1 GHz	
	R (%)	SE (dB)	R (%)	SE (dB)
4	70	35	70	41
2	85	35	70	34
1	95	35	70	27

From the shielding theory (Schultz) it can further be derived that the specific resistance ρ (Ω cm), for homogeneously conductive plastic plates and independently of the plate thickness, shows the following values as R(%) varies

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	R (%)	ρ (Ω cm)
	99	0.11
	95	0.53
	90	1.1
5	80	2.2
	70	3.3

Hence, it can be derived from the data that a thicker plate may possess a lower specific conductivity ($1/\rho$) and a lower reflection value to reach the same shielding effectiveness (SE) at a given frequency (e.g. at 1 GHz). Thus, the D/L value of the fibers may at a same fiber concentration be higher in a thicker plate than in a thinner plate, or, in other words, the fiber concentration in a thicker plate may be smaller than in a thinner plate when D/L is equal in both plates.

Transmission, reflection and resistivity measurements were conducted on the injection-molded plates. The transmission and reflection measurements were made at 10 GHz. For these measurements the plates were placed between a wave emitter (an oscillator) to which, via a circulator, a first horn antenna was connected, and a second horn antenna, which is connected to a second detector. The energy generated by the oscillator is sent to the plate via the first antenna and the transmitted energy is, via the second antenna, registered by the second detector connected thereto. The reflected energy is returned to the first antenna and registered by a first detector connected thereto. This amount of

reflected energy is expressed in percent (R-value) of the amount of energy (100%) which is reflected by a metal plate in the same circumstances. When the amount of transmitted energy is equal to zero, then, for the purpose of reflection measurement and registration, the plate is reciprocated at constant speed between and from near the first antenna to the second antenna over a distance of 22 cm. This movement starts at least 14.5 cm away from the circulator. This dynamic method enables the avoidance of measuring errors which might occur in static measurements when the position of the various plates relative to the circulator is not exactly the same during the successive measurements. Indeed, the measured reflection signal is always the result of successive reflections and rereflections between the plate specimen and the metal (circulator, antenna). This produces a standing wave pattern as a function of the distance between specimen and emitter. In the dynamic method, the average value of the registered standing wave pattern is determined by a microprocessor.

For the measurement of the specific resistance (resistivity), the plates or sheets are connected near their opposite edges between clamps in an electric circuit. To obtain good conductive contact between these clamps and the conductive fibers in the clamped plate edges, the latter are scoured and coated with silver paint.

The measuring results were as follows.
(average values):

	Reflection (%)	Transmission (%)	Specific resistance (Ω cm)
Noryl	65	0	2
Lexan	71	0	3
Cycolac	65.5	0	4

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This shows that the injection-molded plates with a thickness of 2.3 mm were on the limit between insufficient and sufficient shielding effectiveness (35 dB) for certain applications. See area A in Figure 4.

Example 3

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A similar resin-impregnated flat filament bundle (thread) as in Example 2 was chopped into granules of 1 cm length and as in Example 2 mixed with pure resin pellets (Cycolac) in the desired proportion. These resin pellets had the usual dimensions (approximately 0.5 cm long, 0.5 cm wide, and 0.2 cm thick). The mixture was extruded into a round thread and cut to form composite grains containing approximately 1.1 percent by volume of metal fibers (see Example 2). The composite grains were then dry mixed with pure plastic pellets in a 50/50 proportion and were fed to an injection molding machine of the Maurer type with a nozzle orifice having a diameter of 0.95 cm. The same temperatures as in Example 2 were applied. If the shielding characteristics must be sufficient in the immediate vicinity of the nozzle, the injection will preferably take place at a slow pace and/or an after-pressure which is as low as possible will be applied at the

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end of the injection process. The injection molded plates were 5mm thick. The average fiber length L was determined by cutting very thin slices from these plates, and subsequently dissolving the resin from these slices and analysing the remaining fiber netting under a microscope. Area B in Figure 4 corresponds with the thus determined fiber length distribution. The shielding and conductivity measurements were conducted as described above. The results are summarized in the table below:

	Reflection (%)	Transmission (%)	Specific resistance (Ω cm)
Cyoolac	63	0	4

Example 4

Flat granules comprising 20,400 parallel stainless steel fibers with a diameter of $8 \mu\text{m}$ and a length of 3 mm embedded in 8 percent by weight of acrylate resin K70 (from the company Kontakt Chemie) were, under careful stirring, directly added to a 45% solution of a thermo-hardening polyester resin Derakene 411 in styrene. The fibers from the granules were evenly and randomly dispersed in the resin and the usual accelerators were added, as well as a catalyzer. The relatively liquid mass was molded into 30 cm x 30 cm x 3 mm plates and de-aerated. The mold was closed and rotated during the cold hardening process to prevent the metal fibers from settling to the bottom of the mold. The hardened plate comprised 0.5 percent by volume of metal fibers. In Figure 4 this mix

composition corresponds with point G. The measured reflection amounted to 92% at a specific resistance of 0.43 Ω cm and at a transmission of 0%.

5 Similar plates (same dimensions) were made with compositions as mentioned hereunder. Reflection, transmission and specific resistance were measured.

	D (mm)	L (mm)	C (%)	R (%)	specif. resist. (Ω cm)	transmission (%)	point in Figure 4
10	0.008	3	0.25	70	1.44	0	C
	0.004	3	0.25	87	1.68	0	D
	0.004	3	0.50	84	3.11	0	E
	0.004	3	0.12	70	15.1	0	F

15 From the examples and results limits were derived for the volume concentration of the fibers (C%) as a function of the D/L ratio of the fibers. According to the invention, the area between the two straight lines 1 and 2 in Figure 4 determines the optimal conditions for C, D and L to provide sufficient shielding effectiveness for plates with a thickness smaller than 20 3 mm. For plate-or sheet-like articles with a thickness between 3 mm and 6 mm, the straight line 3 in Figure 4 will be the lower limit for providing sufficient shielding.

Example 5

A substantially round, non-twisted bundle of about 10,000 Bekinox stainless steel filaments AISI 316L with an equivalent filament diameter of 0.004 mm was impregnated and sheathed, for example, with a Dynapol L850 solution as explained in Example 1 to form a strand. Granules of 0.5 cm in length were cut from this strand and dry blended in the appropriate proportion with CYCOLAC-KJB-pellets to make grains. The grains were again made by extrusion on the Samafor 45 extruder (Example 1) and comprised about 0.5 % volume of the fibers. Their length was chosen at 1 cm. After dry blending these grains with an equal amount in weight of Cycolac KJB pellets, the mixture was fed to the injection molding machine used in Example 1 to mold a plate of 2.3 mm thick. An even dispersion of about 0.23% volume of fibers was realized in the plate and the average fiber length was estimated at about 0.7 mm. This result is indicated by line H in Figure 4. The antistatic performance of this plate was estimated by rubbing the plate with a textile pad so as to generate an electrical charge on its surface. The plate was then brought in the vicinity of a certain quantity of fine cigarette ash dust laying on a table. There was no significant tendency for the ash dust to lift from the table and to deposit itself on the underside of the plate. However, when repeating the same antistatic dust test with a pure CYCOLAC-KJB-resin plate, devoid of metal fibers, the ash dust was immediately attracted to the plate.

Example 6

A bundle of about 10,000 BEKINOX stainless steel fibers in sliver form with an equivalent fiber diameter of 0.0074mm was impregnated and sheathed with a Dynapol L850 resin as described in Example 1. The strand had a metal fiber content of about 25 % by vol. In two tests, granules 0.6 cm and 0.3cm respectively in length were cut from this strand and dry tumble blended with plastics pellets of Cycolac KJB (grey) to obtain a composition of 0.5 % by vol. metal fibers in the balance resin. The blend was directly fed into the hopper of an injection molding machine of the Type Stubbe S150/235 (operating pressure 130 kg/cm², injection pressure 30 kg/cm², after pressure 30 kg/cm²). The temperature at the injection orifice was 205°C and the injection time 4 sec. for a molded plate measuring 30cm by 30cm and with a thickness of 3mm. The metal fibers were substantially uniformly distributed in the plastic. The electrical properties are given in the table below (average values).

FIBER LENGTH IN GRANULE (mm)	REFLECTION (%)	TRANSMISSION (%)	SPECIFIC RESISTANCE (Ω cm)
3	70	0	7
6	67	0	11

The reflection value at a metal fiber content of 0.5 % in the plastic still results in a shielding effectiveness of more than 25 dB.

According to our experience we can expect a sufficient shielding performance (25 dB) with less stainless steel fibers having a diameter of about 0.0065mm and with a direct feed at the injection molding machine of a mixture containing granules of about

3 to 5mm in length and a metal fiber content in the granules of about 65 % by vol. e.g. at about 10,000 fibers per granule.

5 This experiment thus proves that good shielding results are achievable with a direct introduction of granules at the injection molding stage, thus deleting the intermediate step of making grains.

10 The invention has been particularly described with reference to plastic materials for shielding against radio frequency and high frequency waves. In case of a relatively high L/D ratio of the thin conductive fibers in the plastic matrix, electromagnetic waves in the radar frequency range can be greatly absorbed. The volume concentration of fibers may
15 in this case be very low since good conductivity is no requirement for camouflage against radar waves. Here, the surface resistivity of plastic sheets containing dispersed conductive fibers will preferably be higher than 100 Ω /sq. A reflection value of 10% is sufficient,
20 but generally it will be approximately 40-50%. The relationship between fiber concentration and D/L will in most cases correspond to a point in the area to the left of the straight line 2 in Figure 4 at concentrations lower than 0.25 volume percent.

25 The words HASTELLOY, INCONEL, HYTREL, DYNAPOL, NORYL, CYCOLAC, LEXAN and DERAKEENE are registered Trade marks.



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CLAIMS

1. A plastics material which is at least in part electrically conductive, said part having electrically conductive fibers having a specific conductivity of at least 0.5% of the copper standard randomly and substantially
5 uniformly distributed therein, the fibers having an equivalent diameter (D) and a length (L) such that for a major proportion of the fibers the ratio D/L is from 0.0005 to 0.008, and the volume concentration percentage (C%) of the fibers in said part being less
10 than about 0.5%.
2. A material according to claim 1 wherein the volume concentration percentage (C%) of the fibers in said part is 0.03 to 0.5%.
3. A material according to claim 2 wherein the
15 said volume concentration percentage (C%) is from 0.05 to 0.5%.
4. A material according to any of the preceding claims wherein the equivalent diameter (D) is from 0.002 mm to 0.015 mm.
- 20 5. A material according to any of the preceding claims wherein the fibers are metal fibers having an average length of from 0.5 to 5mm.
6. A material according to any of the preceding
25 claims wherein the conductive fibers have relatively smooth surfaces.



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7. A material according to any of the preceding claims wherein the conductive fibers comprise stainless steel fibers.
8. A material according to any of the preceding claims wherein the plastic comprises a thermosetting resin.
9. A material according to any of claims 1 to 7 wherein the plastic comprises a thermoplastic resin.
10. A material according to either of claims 8 and 9 wherein the resin is an elastomeric resin.
11. A material according to any of claims 3 to 10 wherein the resin is a foamed resin.
12. A material according to any of the preceding, claims which contains fibers in addition to the electrically conductive fibers.
13. A material according to claim 12 wherein at least part of the additional fibers is provided by reinforcing fibers.
14. A material according to any of the preceding claims which contains a flame retarding agent.
15. A plastics article consisting at least in part of a plastics material as claimed in any of the preceding claims.
16. An article according to claim 15 in the form of or made at least in part from a plastics material in sheet or plate form.



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17. An article according to claim 16 wherein the thickness of the plate or sheet is less than 3 mm and the volume concentration (C) of conductive fibers in the plate or sheet is within the limits defined by line 1 and the ordinate axis
5 of Figure 4 of the accompanying drawings.
18. An article according to claim 16 wherein the thickness of the plate or sheet is between 3 mm and 6 mm and the volume concentration (C) of conductive fibers in the plate or sheet is within the limits defined by line 3 and the ordinate
10 axis of Figure 4 of the accompanying drawings.
19. An article according to any of claims 16 to 18 wherein the volume concentration (C) of conductive fibers in the plate or sheet is within the limits defined by line 2 and the ordinate axis of Figure 4 of the accompanying drawings.
- 15 20. An article according to any of claims 15 to 19 made by injection molding.
21. An article as claimed in any of claims 15 to 20 having a shielding effectiveness against electromagnetic radiation of at least about 25 dB within a frequency
20 range of 0.1 to 10GHz.
22. A plastics grain for use in the production of plastics materials and articles as claimed in any of claims 1 to 21, the plastics grain having a length between 0.4cm and 1.2cm with electrically conductive
25 fibers distributed therein.
23. A grain according to claim 22 wherein the average length of the electrically conductive fibers is greater than that of a predetermined average length for the electrically conductive fibers in a plastics material
30 or article to be produced therefrom.



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24. A grain according to claim 22 or claim 23 wherein the fiber content is greater than a predetermined fibre content for the plastics material or article to be produced therefrom.
- 5 25. A grain according to any of claims 22 to 24 having a volume concentration of the fibers of from 0.5% to 2%.
26. A grain according to any of claims 22 to 25 which contains fibers in addition to the electrically
10 conductive fibers.
27. A thread for use in the production of plastics grains as claimed in any of claims 22 to 26 comprising a bundle of conductive fibers embedded in plastic, the fiber content being from 20 volume percent to
15 70 volume percent and the fiber diameter being not greater than about 0.015mm.
28. A thread according to claim 27 having a flattened cross-sectional surface.
29. A thread according to either of claims 27 and
20 28 wherein the fiber bundle contains in its cross section from 1,000 to 35,000 fibers.
30. A thread according to any of claims 27 to 29 wherein the plastic in which the bundle is embedded comprises a thermoplastic polymer of relatively low
25 molecular weight.
31. A plastics material or article as claimed in any of claims 1 to 21 which is at least in part electrically conductive substantially as herein described.



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32. A plastics material or article as claimed in any
of claims 1 to 21 which is at least in part
electrically conductive substantially as herein
described with reference to any of the accompanying
5 drawings.
