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Process for producing moldings of ultra high
molecular weight polyethylene

The invention relates to a process for producing moldings
from pulverulent ultra high molecular weight polyethylene
(UHMWPE).

Ultra high molecular weight polyethylene is taken to mean
linear polyethylenes produced by a low pressure process
and having viscometrically measured average molecular
weights of at least 1×10^6 g/mol, in particular from
 2.5×10^6 g/mol to more than 1×10^7 g/mol. The procedure
for determining the abovementioned molecular weights is
described, for example, in CZ-Chemische Technik 4 (1974),
129 ff.

UHMWPE has a special position among polyethylenes. It has
a number of physical properties which make many
applications possible. Worthy of particular mention are
its high abrasion resistance, its low coefficient of
friction and its exceptional toughness. It is furthermore
markedly resistant to numerous chemicals.

Because of its favorable mechanical, thermal and chemical
properties, UHMWPE has found application in various
fields as a high-value special material. Examples are the
textile industry, mechanical engineering, the chemical
industry and mining.

Yet the scope for using ultra high molecular weight
polyethylene is limited by the difficulties in its
processing. This is because, unlike the low molecular
weight types of polyethylene, the high molecular weight
products can only be processed using presses and screw or
ram extruders, because of their extremely high melt
viscosity. Pressing and extrusion often give only partly
formed pieces from which parts of the desired shape must
be produced by machining off material.

A process for producing solid objects from UHMWPE is



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described in DE-A-24 25 396. It starts from pulverulent material, the particles of which have an average particle size of less than $100\text{ }\mu\text{m}$ with a distribution function (which can be determined from the particle count for a number of particle size intervals) of less than 0.8. The powder is pressed to give a solid preform at a pressure of at least 140 kg/cm^2 at a temperature below the crystalline melting point of the polyethylene. After releasing the pressure, the preform is sintered at a temperature above the crystalline melting point. Since the as-synthesized ultra high molecular weight polyethylene usually contains particles larger than $100\text{ }\mu\text{m}$, this procedure requires comminution of the starting material by appropriate means, for example by milling. An additional disadvantage of the known procedure is that the moldings are not homogeneously plasticated throughout. They can therefore expand during the sintering process as a function of the temperature applied. This phenomenon can be attributed to the preform being sintered outside the pressing mold. As a consequence, the physical properties are not reproducible and vary greatly. Strict dimensional accuracy of the moldings is furthermore not ensured.

Another process for producing moldings from pulverulent polyolefins having molecular weights of at least 1 million is the subject of DE-C-26 34 537. The pulverulent polymers are converted into the rubber-elastic state at from 150 to 250°C , preferably from 190 to 210°C , in a mold loosely closed by a punch. The material is then immediately densified in the mold at pressures of from 2.5 to 25 N/mm^2 in a first stage of at least 2 minutes, then at pressures of from 40 to 100 N/mm^2 in a second stage of at least 1 minute and the molding is then allowed to cool in the mold without pressure being applied. This process has proved successful in practice, but only makes possible the production of moldings having a simple geometry.

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The object was therefore to provide a process which makes possible the manufacture of even complex-shaped moldings from pulverulent ultra high molecular weight polyethylene, without suffering the disadvantages and limitations of the known processes. In particular, it must meet the requirement of giving products which are completely homogeneous and which have uniform physical properties throughout.

The invention achieves the above described object by means of a process for producing moldings from pulverulent ultra high molecular weight polyethylene. It comprises heating the polymer to from 160 to 280°C in a mold loosely closed by a punch in a press, without applying pressure or at a pressure of up to 0.5 MPa, and then cooling it to room temperature at a pressure of from 4 to 20 MPa.

The novel process allows the production from UHMWPE of moldings having varying and even demanding geometries. The moldings have high homogeneity and dimensional accuracy.

The starting material can be any type of pulverulent ultra high molecular weight polyethylene, regardless of how they were produced. It is accordingly possible to use polymers obtained by the Ziegler process by polymerization of ethylene in the presence of transition metals of the 4th to 6th group of the periodic table of the elements together with organometallic compounds of the elements of the 1st to 3rd group of the periodic table of the elements. It is, however, also possible to use ultra high molecular weight polyethylenes which have been produced from anhydrous and oxygen-free ethylene in the gas phase in the presence of supported catalysts containing chromium oxide and metal alkyl. It is particularly remarkable that the polymers can be used with the particle size obtained in the synthesis. The average particle size is, depending on the polymerization

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process, about from 100 to 400 μm , but may also be smaller or larger.

The process of the invention can be carried out in a simple manner. The pulverulent polymer is introduced
5 into a mold of any desired shape, which is loosely closed by a fitting but not airtight punch lying on top. In this process stage the punch applies only a slight pressure (contact pressure) of up to 0.5 MPa to the powder. The powder in the mold is subsequently heated to a tempera-
10 ture of from 160 to 280°C, preferably from 220 to 250°C. This procedure can be carried out directly in the press, heat being conducted from the upper and lower platens. A heatable mold can, however, also be used. Finally it is possible to carry out the heating in a special facility,
15 for example in an electric oven. Owing to the poor thermal conduction, a temperature gradient is established in the powder bed with the result that the temperature decreases from the outside layers into the middle of the mold. To avoid inhomogeneities in the final pressed
20 product, care must be taken that temperatures in all parts of the powder bed are within the abovementioned ranges. Overheating, even in limited regions, must be avoided so that the material is not thermally damaged. Oxidative damage to the heated UHMWPE can be combated by
25 maintenance of an inert gas atmosphere, for example nitrogen. The duration of heating is dependent on the powder volume and on the geometry of the molding. During heating the ultra high molecular weight polyethylene, which possesses not an actual melting point but a
30 crystallite melting range (about 130 to 135°C), becomes viscoelastic. The weight of the loosely overlying punch leads to a minor densification of the powder, and to substantial degassing of the material.

As soon as the processing temperature of from 160 to
35 280°C, preferably from 220 to 250°C, has been reached, the polymer in the mold is placed under a pressure of from 4 to 20 MPa, preferably from 8 to 14 MPa. The

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pressure to be used is dependent in particular on the amount of polymer to be processed and on the geometry of the molding. Higher pressures should be used for large amounts of polymer and for shapes which are composed of regions of widely differing wall thicknesses. The processing temperature is maintained until the punch reaches its final position in the mold at constant pressure. This stage is generally attained within from 1 to 5 min. The molding is then cooled to room temperature while maintaining the pressure. Cooling may occur by heat exchange with the surrounding atmosphere; it can be accelerated by use of coolants in the upper and lower platens or in the mold. The cooling time depends on the size of the molding and on the type of heat exchange.

In each phase of the process prior to the cooling under pressure it is possible to add more pulverulent UHMWPE to the mold, if the amount of material needed for production of the desired molding has to be supplemented during the process. Account must here be taken of the fact that the bulk volume of the polyethylene powder is from about 2 to 2.5 times as great as the volume of the molding manufactured from it. The newly added powder must, of course, be subjected to the same thermal and pressure treatment as that originally used. It is, however, more advantageous to provide the mold with an upstream chamber from which further polymer powder can be continuously fed in such quantity as is necessary to fill the mold, and to calculate the stroke so that the powder can be densified in one operation. The cooled body is completely plasticated and free of voids; it can easily be removed from the mold.

The process of the invention makes possible the production of dimensionally accurate moldings from ultra high molecular weight polyethylene by pressing. No wasteful machining to remove material is necessary. The pressurization of the plasticated bodies takes only a short time, so that press capacity can be effectively

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used. The pressurization can furthermore be carried out at room temperature after the heating stage; in that way it consumes little energy. The novel procedure ensures a high production rate by use of multiple-die presses or of automatic processing methods. It is suitable for manufacturing moldings of any desired shape. Complex-shaped machine parts such as pump housings, pump rotors and valve components, for example for chemical apparatus, can be produced in high quality. It is, however, also possible to manufacture sheets of UHMWPE having a thickness of about 10 mm and less, which could previously only be obtained by splitting polyolefin blocks.

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What is claimed is:

1. A process for producing moldings from pulverulent ultra high molecular weight polyethylene, which comprises heating the polymer to from 160 to 280°C in a mold loosely closed by a punch in a press, without applying pressure or at a pressure of up to 0.5 MPa, and then cooling it to room temperature at a pressure of from 4 to 20 MPa.
2. The process as claimed in claim 1, wherein the ultra high molecular weight polyethylene is heated to from 220 to 250°C.
3. The process as claimed in claim 1 or 2, wherein the heating is carried out in an inert gas atmosphere.
4. The process as claimed in one or more of claims 1 to 3, wherein the polymer is cooled in the press at a pressure of from 8 to 14 MPa.

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