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VESTAKEEP®

POLYETHER ETHER KETONE

Directory

1 Introduction

The High Performance Polymers Business Unit of Degussa AG markets a line of polyether ether ketone compounds (PEEK[1]) under the trade name VESTAKEEP®*. These products are produced in China by JIDA Degussa High Performance Polymers Changchun Co. Ltd., a joint venture 80 percent owned by Degussa and 20 percent by Jilin University, Changchun. This brochure provides an overview of the properties and applications of PEEK products and instructions for their processing.

VESTAKEEP compounds are particularly characterized by the following material properties:

- very high heat resistance
- high rigidity
- low water absorption and therefore high dimensional stability
- high hardness
- good strength
- excellent sliding friction behavior, minimal abrasion
- good electrical characteristics
- excellent chemical resistance
- excellent hydrolytic stability
- good processability
- low tendency to form stress cracks

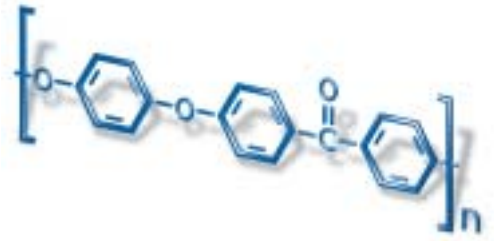
[1] PEEK is the official abbreviation for polyether ether ketone according to ISO 1043. In this brochure it will be used only in this context.

* VESTAKEEP® is the trademark for the polyether ether ketone of Degussa AG, registered in Germany.

Introduction

Manufacture

VESTAKEEP is polycondensed from the building blocks hydrochinone and 4,4´-difluorobenzophenone in a multistage process.



The base polymer has a melt viscosity of 100–3,000 Pas, measured at 400 °C, and a low shear of 1 sec^{-1}, which is right for injection-molding and extrusion applications.

To meet the requirements of different applications, manufacturers can adjust the properties of pure PEEK selectively by adding various additives:

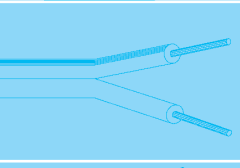
- Processing aids facilitate demolding.
- Fillers and reinforcing materials increase rigidity and dimensional stability upon exposure to heat. Chopped carbon fibers are most effective for this. Minerals and glass microbeads also counteract the tendency to warp.

Applications

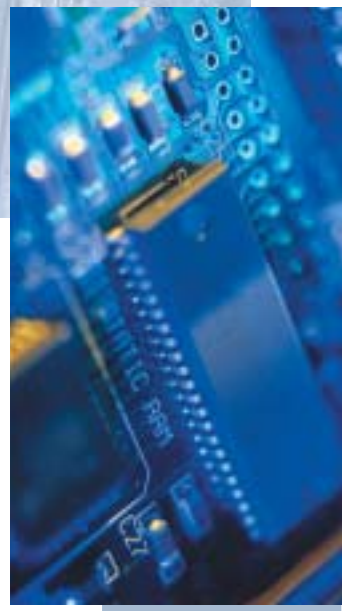
VESTAKEEP compounds can be used for a wide range of applications, such as in electrical, electronic, and communications engineering and in the automotive industry. Table 1 lists the properties that are particularly relevant to various applications.

All high-performance plastics from the High Performance Polymers Business Unit meet the highest quality standards. Our system of quality assurance, which we've applied to our marketed products for many years, is certified according ISO 9001:2000 and ISO/TS 16949:2002. Over the years, numerous customers have tested this quality system and confirmed its superiority. The same care that we devote to our other products is also applied to the manufacture and quality assurance of the new VESTAKEEP product line.

Table 1: Performance profile of polyether ether ketones for particular applications

	**Automotive**
	**Aerospace** **Rail cars**
	**Machinery and apparatus construction**
	**Electrical and cable**
	**Medical technology**
	**Food processing industry**

***"Free of toxic fumes" does not apply to compounds containing PTFE.
See Section 5, "Information about environmental compatibility and safety"



	High temperature resistance
	Chemical resistance
	Hydrolysis resistance
	Physical stability
	Wear resistance
	Fire behavior
	Toxic fumes ***
	Electrical properties
	Degassing
	Ion extraction
	Dimensional stability
	Processability
	Sterilizability

2 Overview of VESTAKEEP compounds and their properties

Commercial products
The PEEK compounds from High Performance Polymers include a variety of different products that have been matched to the requirements of processors and end consumers. Table 2 provides an overview of the characteristics of the most important products and their typical applications. More detailed information about most of these compounds can be found in Table 3. For further information about the other compounds, please contact the persons indicated.

Other properties of VESTAKEEP compounds and material information on the other products of the High Performance Polymers Business Unit are contained in the plastics data base **Campus®**[2] , which is updated regularly. You'll find Campus on the Web at **www.degussa-hpp.com.**

Development products
Development products are usually designed for a specific application. When we introduce a product onto the market, the findings and feedback we receive allow us to optimize it further. Consequently, a change in the formulation or manufacturing process may lead to some slight changes in the product's properties. We immediately notify our customers of any changes to the material's composition and how these may influence the quality or specifications of the product itself. If you're looking for a product with a special requirements profile, please contact the person indicated.

Powders
In addition we offer VESTAKEEP as powders. Our employees will be happy to provide further information and support.

Delivery
VESTAKEEP compounds are delivered as granules in 25 kg boxes that have moisture-proof polyethylene liners. By mutual agreement, we'll also deliver VESTAKEEP in other containers, such as special octabins holding 500–600 kg. Under normal storage conditions, storage time is practically unlimited provided that the packaging has not been damaged. Avoid storing at temperatures above of 45 °C.

Like other partially crystalline polyaryl ether ketones, unmodified VESTAKEEP appears amber-colored in the melt and grayish in its solid crystalline state (natural colors). VESTAKEEP is translucent in its solid, amorphous state and has a characteristic amber color. We deliver most compounds in their natural colors. Others have a certain color inherent to them because of the additives they contain. They are available in the following viscosity series:

VESTAKEEP	4000 G
VESTAKEEP	3000 G
VESTAKEEP	2000 G
VESTAKEEP	1000 G

[2] Campus® is the registered trademark of CWF GmbH/Frankfurt (Main)

Table 2: Overview of VESTAKEEP compounds and their properties

Product line and applications	**VESTAKEEP**	Properties	Processing
Low to high-viscosity base grades for products such as gear parts, parts used in medical technology and extruded semi-finished products	**1000 G**	unreinforced, low-viscosity, easy-flow	IM
	2000 G	unreinforced, low-viscosity	IM
	3000 G	unreinforced, medium-viscosity	E, (IM)
	4000 G	unreinforced, high-viscosity	E, (IM)
Carbon fiber-filled or glass fiber-reinforced molding compounds with increased or high rigidity, partially low-warpage, e.g., for housing parts	**4000 GF30**	30% chopped strands	IM
	4000 CF30	30% carbon fibers	IM
Special and specialty grades for applications in the electrical engineering, automotive, machinery and apparatus construction industries as molding compound for bearing arrangements and gear parts with a self-lubricating characteristic	**4000 FC30**	10% graphite 10% carbon fibers 10% PTFE	IM
Specialty grade for the cable industry	**3001 G**	unreinforced, melt-filtered	E

IM = Injection molding, E = Extrusion
GF30 = glass-fiber-filled 30%, FC30 = PTFE/graphite/carbon fiber (10:10:10), CF30 = carbon fiber 30%

Properties

Table 3: Physical, thermal and mechanical properties and fire behavior of VESTAKEEP compounds

Properties		Test method	Unit	VESTAKEEP 4000 G	VESTAKEEP 4000 GF30
Physical and thermal properties and fire behavior					
Density	23 °C	ISO 1183	g/cm³	1.30	1.50
Melting point	DSC		°C	340	340
Volume flow rate (MVR)	379 °C/5.00 kg	ISO 1133	cm³/10 min	10	2.0
Temperature of deflection under load		ISO 75-1/2			
Method A	1.8 MPa		°C	153	>240
Method B	0.45 MPa		°C	176	>240
Linear thermal expansion					
	23–55 °C	ISO 11359			
	longitudinal		10^{-4} K^{-1}	0.58	0.26
Oxygen index		ISO 4589	%	35	45
Flammability acc. UL94	0.8 mm	IEC 60695		V-0	V-1
	0.6 mm			V-0	V-0
Glow wire test	2 mm	IEC 60695-2-1/0-3	°C	960	960
Mold shrinkage		ISO 294-4			
in flow direction			%	1.11	0.38
in transvers direction			%	1.82	0.73
Mechanical properties					
Tensile test	50 mm/min	ISO 527-1/-2			
Stress at yield			MPa	95	
Strain at yield			%	5.3	
Nominal strain at break			%	>25	
Tensile test	5 mm/min	ISO 527-1/-2			
Tensile strength			MPa		165
Strain at break			%		2.5
Tensile modulus		ISO 527-1/-2	MPa	3500	11000
CHARPY impact strength	23 °C	ISO 179/1eU	kJ/m²	N	70 C
	-30 °C		kJ/m²	N	75 C
CHARPY notched impact strength	23 °C	ISO 179/1eA	kJ/m²	6.5 C	10 C
	-30 °C		kJ/m²	6.5 C	9 C

	VESTAKEEP 4000 CF30	VESTAKEEP 4000 FC30
	1.40	1.45
	340	340
	2.5	2.5
	>240 >240	>240 >240
	0.11	0.17
	46	44
	V-0 V-0	V-0 V-0
	960	960
	0.05 0.59	0.30 0.54
	210 2.0	140 2.5
	20000	11500
	55 C 55 C	45 C 45 C
	9 C 8 C	8 C 7 C

3 Processing

Extrusion
We recommend a linear temperature profile for extrusion:
Hopper: 100 °C–120 °C
Temperature zone 1 - T-nozzle: 350 °C–380 °C
For further information please contact the persons indicated.

Injection molding

General Information	
■ For injection molding, VESTAKEEP polymers and compounds are primarily processed in granular form. Most standard screw injection molding machines are suitable for this. The plasticating unit must be temperable to approximately 450 °C. It may also be necessary to modify the controller, band heaters, and temperature sensors. In addition, we recommend that the instructions listed below be observed when using PEEK for injection molding. ■ Comprehensive application advice is part of our technical service. For example, we employ various Computer Aided Engineering (CAE) programs to solve many problems arising from the mold and part design. Please contact the indicated contact persons for further information.	
Plasticating unit	
Screw and barrel	■ Standard screw (three-zone screw) with a length between 18 and 24 D - Zone breakdown: feed 12 D, compression 2 D, metering 6 D ■ "Polyamide screw" - Zone breakdown: feed 12 D, compression 4–6 D, metering 4–6 D - Screw length 18–24 D ■ Flight depth ratio 2–3:1 ■ Screw and cylinder diameter should be of such a magnitude that a metering stroke between 1 D and 3 D is possible.
Back flow valve	■ Back flow valve is required ■ Clearance between back flow valve and cylinder will be ≤ 0.02 mm
Nozzle	■ In general, free-flow nozzles are recommended. Work should be performed when the melt outlet is in a state of **decompression**. ■ Shut-off nozzles are less suitable, because there is a hazard of injection pressure losses from thermal "dead spots." ■ The nozzles must be adequately heated to keep the melt from transitioning and prevent the formation of cold "plugs." ■ Bore of nozzle should be app. 0.5 to 1 mm smaller than the gate.
Tool steel	■ For the production of PEEK-compatible processing components, we recommend corrosion-resistant steels and bimetals for screws and barrels. ■ Metallic areas that come into direct contact with the melt should be highly polished to prevent local adhesion of the melt to the metal. Such adhesion could increase the dwell time and disturb the flow of the melt. ■ In conventional nitriding, you have to make sure that the PEEK melt does not cool on the surface and solidify on the nitride layer. The adhesion can be so strong that the nitride layer can peel off from the steel core.

Cleaning	
General	■ Remove other polymers from the machine completely before processing VESTAKEEP molding compounds. This can be accomplished either by cleaning the cylinder and screw mechanically or by using suitable cleaning materials. These are materials that are thermally stable up to approximately 380 °C. One suitable material is a high-viscosity PC containing glass fibers (e.g., Makrolon® 8345). Other suitable materials include PES, PEI and, with limitations, high-viscosity PP. Since PP decomposes at these temperatures, effective ventilation is important.
Cleaning while **starting** the injection molding machine	1. Set the temperature to the temperature normally used when processing the material to be removed. 2. Introduce the cleaning material and continue rinsing until no traces of the material to be removed can be detected. 3. Run the screw idle. 4. Set the cylinder temperatures to the values required for PEEK processing. 5. When the temperatures have been attained, feed the material into the cylinder and extrude until a clean melt is present.
Cleaning while **shutting down** the injection molding machine	Completely remove the PEEK melt from the cylinder before processing another material. There exists the danger that the melt could solidify with the nitride layer of the cylinder and screw while cooling. Because of the high adhesive forces, this layer could peel and damage the screw (see "Tool steel"). This means that the cylinder may be allowed to cool only after cleaning and careful rinsing. Cleaning process: 1. Remove material form the injection molding machine (hopper). 2. Introduce the cleaning material and continue extruding until there are no longer any visible traces of the PEEK material. 3. Reduce cylinder temperatures to a lower temperature (350 °C) that is still acceptable for PEEK. 4. Continue rinsing with the cleaning material until the actual cylinder temperature drops below 300 °C. An even lower temperature (< 250 °C) may be required, depending on cleaning material.
Clamping unit	
Clamping force	■ An adequate clamping force must be ensured since the injection pressures of 100 to 150 MPa are very high in comparison with other polymers. ■ The production of precision parts involves pressures in excess of 200 MPa.
Tool	
Tool steel	■ For the cavity, use steel grades that still have a hardness of about 54 HRC at the high processing temperatures, for example 1.2343 (X38CrMoV51), 1.2379 (X155CrVMo121) - core hardened, 1.2083 (X42Cr13) - core hardened, corrosion-resistant
Wall thickness	■ Minimum wall thickness: - 1 mm for unfilled PEEK molding compounds - 1.5 mm for filled PEEK molding compounds

Processing

Tool	
Flow-distance/ wall-thickness ratio	■ Maximum attainable flow distance/wall thickness ratios for unfilled materials and 2 mm wall thickness up to 180 : 1 (conditions: melt temperature 380 °C, mold temperature 180 °C, injection pressure 140 MPa)
Sprue	■ Minimum diameter: 4 mm, for direct gating 1 to 1.5 times the thickness of the molded article ■ Demolding draft angle: at least 2° ■ Pull-out plug: special for direct gating ■ Manifold: round or trapezoidal (cross section as large as possible)
Gate	■ Dependent on melt volume, number of cavities, component geometry; nearly all common systems are suitable; avoid tunnel gates, however; thin flow areas should be avoided; - Minimum gate diameter: 1.0 mm for unfilled materials 2.0 mm for reinforced materials
Hot runner system	■ Special hot-runner systems matched to the PEEK processing temperatures are possible. It must be possible to set the system to 450 °C indefinitely.
Venting	■ Venting slots in mold parting surface: 5 to 10 µm deep, 4 to 5 mm wide ■ Further support of ventilation by means of appropriately fashioned ejector pins is possible
Pressure gauge	■ We recommend the use of an internal pressure gauge to set the switching point precisely.
Temperature control	■ Usually with heat transfer oil - Because of potential heat losses, it must be possible for temperatures to reach 250 °C. - Tightly screw down die feeders using temperature-resistant seals. ■ Electrically using cartridge heaters - Exact heat control more difficult because heat cannot be dissipated. ■ We recommend that the mold be insulated externally to achieve better temperature constancy.
Processing conditions	
Cylinder temperatures	■ The optimum melt temperature depends on various factors. Choose the starting temperature based on the viscosity and the filler of the material. - Set the cylinder temperature (feed – nozzle) to increase slightly (15–20 °C) - Feed temperature: > 350 °C - Temperature in the hopper area: 70–100 °C **Melt temperatures for VESTAKEEP:** unfilled materials / reinforced materials 1000 G: 360 °C 2000 G: 360 °C — 4000 GF30: 380 °C 3000 G: 365 °C — 4000 FC30: 385 °C 4000 G: 370 °C — 4000 CF30: 400 °C

Processing conditions	
Nozzle temperature	■ Front cylinder temperature +5 °C
Mold temperature	■ Surface temperature: 160–210 °C Select high temperatures to achieve a high degree of crystallization.
Speed of screw	**Circumferential speed** **Rotational speed, e.g., #30 screw** ■ Unfilled materials: 5–10 m/min 50–100 rpm ■ Reinforced materials: max. 6 m/min 60 rpm Higher speeds are not recommended because of the possibility of thermal overload of the melt caused by frictional heating from large local shear effects.
Back pressure	■ 2–8 MPa Corresponds to a hydraulic pressure of approx. 0.2–0.7 MPa
Decompression	■ We recommend a decompression of ≥ 3 mm for melt ejection from the nozzle.
Injection speed	■ The injection speed should be as high as allowed by the prevailing mold conditions (gate dimensioning, flashing, ventilation, etc.).
Injection pressure	■ 70–150 MPa, depending on the melt and mold temperatures and the flow-distance/wall-thickness ratio of the component.
Holding pressure	■ Up to about 120 MPa; set the values of the holding pressure and holding pressure time in such a manner that the component will not have any sink marks, if possible, assuming that adequate pressure permeability exists (dimensioning of the sprue system)
Holding pressure time	■ Since PEEK materials have a high solidification point, the holding pressure times are relatively short. About 10 seconds is sufficient for a component with a wall thickness of 4 mm.
Production stops	■ For relatively short production stops (up to 1 hour), the material can be kept at 360 °C without any significant decomposition. ■ For downtimes longer than 1–3 hours, the temperature must be dropped to 340 °C. The material possesses adequate melt stability at this temperature. ■ When restarting, rinse the cylinder adequately and reject the first molded parts. ■ For interruptions lasting more than 3 hours, a cleaning is recommended. See "Cleaning."
Trouble shooting	■ The initial instructions are found in Table 4. ■ To trouble shoot problems in injection molding machines, see the guidelines in the common literature (e.g., "Guide to surface defects on thermoplastic injection molded parts," published by Kunststoff-Institut für die mittelständische Wirtschaft, Karolinenstraße 8, 58507 Lüdenscheid, Germany). ■ For further information, please contact the persons indicated.

Table 4: Measures to eliminate defects in PEEK injection molded parts

Defect in the molding		Measure									
	Possible cause	Melt temperature	Mold temperature	Nozzle temperature	Nozzle contact time	Rotational speed of screw	Injection speed	Shot volume	Injection pressure	Holding pressure	
Brittleness	Overheating	↓				↓					
	Stresses	↑	↑						↓		
	Flow line	↑	↑				↑				
Incompletely filled	Too little injected							↑			
	Insufficient flux	↑	↑				↑		↑		
	Mold design										
Transparent edges/dark regions	Mold temperature too low		↑								
Cold plugs	Melt transitions within the nozzle			↑	↓						
Sink marks/voids	Inadequate time and pressure conditions	↓							↑	↑	
	Mold design										
Burn marks	Air trapped in cavity						↓		↓		
Flashing	Clamping force too small/fitting accuracy of the mold halves	↓	↓				↓		↓	P	
Streaking	Overheated molding compound	↓		↓		↓	↓				
	Humid material										
Dull surfaces (Reinforced grades)	Insufficient injection speed	↑	↑				↑				
	Shear on the melt too strong					↓					

↑ = increase ↓ = decrease • = do **P** = profile

													→	←		Cycle time
					•	•				→			→			Gate cross section
					•	•				•		•				Move the gate position
					•					•						Improve venting of cavity
				→												Clamping force
		•														Dry the material

4 Physiological and toxicological evaluation of VESTAKEEP compounds

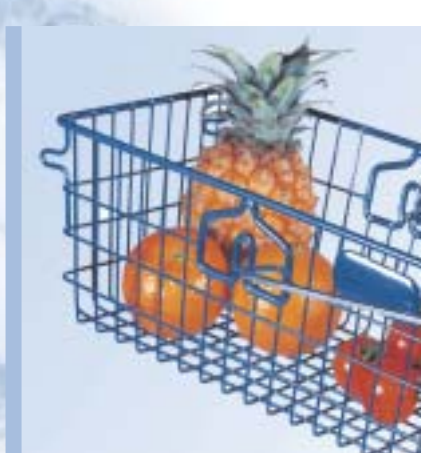
The Environment, Health, Safety & Quality Department, which is responsible for the High Performance Polymers Business Unit, provides general information on the toxicological properties of VESTAKEEP compounds and relevant analysis pertaining to their contact with foodstuffs. The department is also responsible for providing information about product safety and producing the EC Safety Data Sheets for VESTAKEEP. Please direct all questions on the subject to the indicated contact persons.

VESTAKEEP compounds are water-insoluble, solid polymers that are largely inert physiologically. No toxicity is expected from single contact or even multiple contacts, because VESTAKEEP products are not absorbed either through the skin or through the gastrointestinal tract. As in the case of other inert dusts, exposure to VESTAKEEP dusts could possibly result in mechanical irritation in the upper respiratory passages and the mucous membranes of the eye. Irritation or sensitization of the skin is not expected. Based on our best current understanding, VESTAKEEP does not have any adverse effects on man, animals, plants, or microorganisms. Please direct any further questions regarding product safety to the indicated contact persons.

Food Contact – EU-Status

Uniform regulations for plastics that come into contact with foodstuffs exist at the European level. The consolidated EU Directive 2002/72/EC and its amendments apply. VESTAKEEP compounds are approved for direct food contact in the European Union because they are based on monomers that are listed favorably in this directive. Restrictive migration limits must be observed on the finished article.

Plastic additives permitted for food-contact are listed favorably in the "incomplete list" of the EU Plastics Directive. This means that the additives in the EU list and the additives approved by national regulations (in Germany, these are the recommendations of the Federal Institute for Risk Assessment, BfR) may be used. The "incomplete list" of approved substances is scheduled to become a "complete list" by the end of 2006 so that, starting in 2007, only additives appearing in the EU list may be used. Because of these continuous revisions, we're currently unable to make any general binding statements about the status of our VESTAKEEP compounds.



Medical applications

For medical applications, the European approval procedure is laid down in Directive 93/42/EEC. The national implementation of this directive into German law is the Medizinproduktegesetz (Medical Products Act) of August 1994. The detailed procedure to be followed is described in the pertinent international and national standards (e.g., ISO 10993, DIN EN 30993-1). The DAB monographs (German Pharmacopoeia, current edition) or those of the European Pharmacopoeia (current edition 2005) can be used as supplementary regulatory works to make the decision in special cases.

In cases of doubt, the moldings or semi-finished products must be investigated by the manufacturer or user, taking the relevant conditions of use into consideration. Our staff can provide you with information about their experiences with various approval processes.

5 Information about environmental compatibility and safety

VESTAKEEP compounds are non-hazardous substances that are not governed by any particular safety regulations. They can be disposed of in accordance with local ordinances. Further information can be found in the EC safety data sheet for VESTAKEEP. Recycling is, however, preferred and advisable for economic reasons.

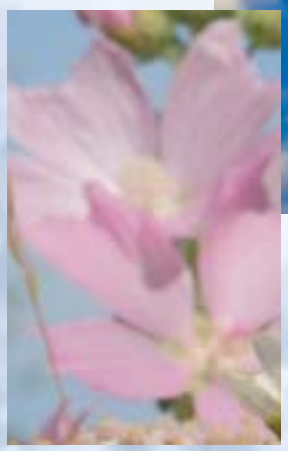
No dangerous byproducts are formed if VESTAKEEP is processed properly. Care should be taken, however, to ventilate the working area properly. Detailed directions about handling VESTAKEEP products can be found in the "Processing" section of this brochure.

Degradation of the material during processing is shown by a discoloration of the melt. Degraded material should be quickly removed from the machine and cooled under water in order to minimize any troublesome smells or fumes.

No pigments or additives containing cadmium are used.

VESTAKEEP compounds are noncombustible. Flammable gases can be released at melt temperatures above 450 °C. Since the spectrum of crack and combustion products greatly depends on the combustion conditions, it is not possible to make any general statements here.

VESTAKEEP compounds, which are filled with PTFE (FC grades), can release toxic gases at temperatures exceeding 380 °C. Without adequate ventilation and special protective measures, direct exposure to these gases may result in poisoning symptoms and can even lead to death in extreme cases. In addition to our instructions, please also comply with the safety data sheet for the compound in question.



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® = registered trademark

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