

PROCESSING GUIDE



Extrusion Molding

Table of Contents

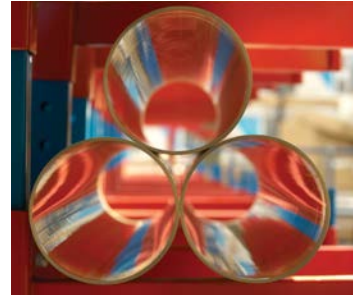
| | |
|------------------------------------|---|
| Introduction | 2 |
| General Process Preparation | 2 |
| Handling | 2 |
| Drying | 2 |
| Processability | 2 |
| Colour | 2 |
| Equipment | 3 |
| General Design | 3 |
| Materials of Construction | 3 |
| Screw / Barrel Design | 4 |
| Barrel Capacity and Residence Time | 4 |
| Barrel Heating | 4 |
| Screens and Breaker Plate | 5 |
| Adapter, Head and Die | 5 |
| Purging and Shutdown | 5 |
| Wire and Cable Coating | 6 |
| Die and Crosshead Design | 6 |
| Downstream Equipment | 7 |
| Crystallinity | 7 |
| Film and Sheet Manufacture | 8 |
| Die Design | 8 |
| Thin Film and Sheet Crystallinity | 8 |
| Monofilament | 9 |

In today's competitive world, you want more from an advanced materials supplier than simply materials. Getting the most from high-performance polymers means considering component design hand-in-hand with material selection and optimised processing. This is the key to maximising an application's value in-use and achieving high manufacturing efficiencies at the lowest possible component cost. With design and material selection decided, processing optimisation becomes critical to drive your product quality and manufacturing yields higher.

With more than 30 years' experience, Victrex Polymer Solutions is uniquely placed to help customers get the very most from polyaryletherketone (PAEK) polymers and products. We offer a wide variety of VICTREX PEEK products that provide exceptional performance over a broad range of temperatures and extreme conditions. Each can be easily processed on standard equipment.

We are able to offer our customers an unrivalled technical capability encompassing design, material selection and processing support for polyaryletherketones. As part of this, we've created this guide to help you optimise your extrusion processing conditions. In addition, our technical teams located around the world can assist you with prototyping, application development, design and simulation as well as support for metal replacement opportunities.

Our increasing number of Technical Centres have processing equipment to support trials for the full range of VICTREX PEEK products, offer hands-on processing training and extensive materials analysis and characterisation capabilities. We can offer bespoke data generation for specific application programs and our resources are backed up by extensive product and application-based datasets which are continually expanding. We are also involved in a number of industry-leading research projects with academic institutions to further extend our knowledge and help us develop more creative solutions with our customers.



VICTREX™ PEEK

VICTREX™ PEEK polymer, along with its higher-temperature variants, VICTREX HT™ polymer and VICTREX ST™ polymer, is widely regarded as one of the highest performing thermoplastics in the world. Products are available as melt filtered granules, milled fine powders, or compounds containing a variety of functional fillers and reinforcements. They are used in the design and manufacture of high performance applications to replace metals and other materials to improve performance, increase design freedom and reduce system costs.

APTIV™ Films

Victrex APTIV™ film provides all of the properties of VICTREX PEEK polymer in a thin, flexible format. The extensive range of properties which includes thermoformability and excellent acoustic performance makes it the highest performing, most versatile thermoplastic film available. APTIV films are a technology enabler to facilitate reduced system costs and improved product performance whilst providing increased design freedom and ease of processing.

VICOTE™ Coatings

VICOTE™ Coatings are a dedicated range of eco-friendly high performance coatings made from VICTREX PEEK polymers. The powder and aqueous dispersions provide high temperature performance, exceptional scratch and wear resistance, high strength and durability. When compared with traditional coatings, these coatings can be considered to improve performance, extend application life, facilitate design freedom and reduce system costs.

VICTREX Pipes™

VICTREX Pipes™ are durable, lightweight pipe and tube extruded from VICTREX PEEK polymer which offer high temperature performance and a unique combination of properties. An excellent alternative to metals and lower performing polymers, VICTREX Pipes provide chemical and corrosion resistance, low permeability, wear abrasion and impact resistance in a polymer-based pipe and tube.

High Temperature Performance

Excellent high temperature resistance, with continuous use temperatures of 260°C, which can offer longer life, reliability and increased safety margins in harsh environments.

Mechanical Strength and Dimensional Stability

Excellent strength, stiffness, long term creep and fatigue properties of Victrex materials allow parts to be designed with reduced weight, greater durability or strength.

Wear Resistance

In wet or dry abrasive environments, a low coefficient of friction and excellent wear resistance can help maintain part life and integrity.

Chemical Resistance

Resists corrosion even at elevated temperatures thanks to its ability to withstand a wide range of acids, bases, hydrocarbons and organic solvents.

Hydrolysis Resistance

Victrex materials have been used successfully to increase component reliability because it does not hydrolyse in water, steam or sea water even at elevated temperatures due to its low moisture absorption and low permeability.

Electrical Performance

Excellent electrical properties maintained over a wide frequency and temperature range to meet demanding electrical and electronic engineering needs.

Low Smoke and Toxic Gas Emission

Inherently self extinguishing without the use of additives and has low toxicity of combustion gases.

Purity

Offers exceptionally low outgassing and extractables for cleaner manufacturing.

Environmentally Friendly

Fully recyclable, halogen free, RoHS and REACH compliant.

Quality and Supply Security

All manufacturing is under ISO 9001:2008 registration and EU safety and environmental legislation. Our rigorous attention to detail – we perform over 50 tests on each batch of polymer – assures our customers of product quality and consistency.

As the only vertically-integrated polyketone solutions provider in the world, we have complete control over our key raw material – essential for consistent polymer quality.

Our policy to invest in capacity ahead of demand means that we have an unrivalled capability to assure customers of supply security. Our two independently-operated polymer plants are capable of up to 4,250 tonnes per year. We can also offer fast delivery – typically within 7 days – anywhere in the world through our centralised logistics system and local distribution warehouses.

INTRODUCTION

All general extrusion guidelines applicable to semi-crystalline polymers also apply to extruding Victrex materials. The higher melting points of Victrex materials require special attention in some areas, which are briefly summed up below.

Temperature capability

Equipment must be rated to 450°C. Barrels must be able to operate up to 400°C for processing PEEK, and up to 430°C for processing HT and ST.

Cooling:

Air cooling / hot rollers should be used immediately after exiting the die, and while the temperature is above T_g , to obtain optimally crystalline products.

Water cooling can be used to cool the extrudate after crystallisation has taken place.

Moisture content:

Although not hygroscopic, Victrex materials must be dried prior to extrusion.

Cleanliness:

Contamination must be avoided - dedicated scoops, trays for drying etc. are highly recommended.

Startup Procedures:

For black speck-free processing it is normally necessary to strip down and clean the screw, barrel and die assembly before every use.

Details to this high level summary are addressed in subsequent sections.

GENERAL PROCESS PREPARATION

HANDLING

Victrex materials are supplied in a sealed polyethylene bag inside a heavy-duty cardboard box or a pallet sized box. It is strongly recommended that the materials remain sealed in the original packaging during transportation and storage. When material is required, the boxes should be opened in a clean environment and care taken to avoid contamination. Any remaining material should be re-sealed immediately and kept under standard conditions (15-25°C, in the dry and away from direct sunlight), under which the product be stored in excess of 10 years.

DRYING

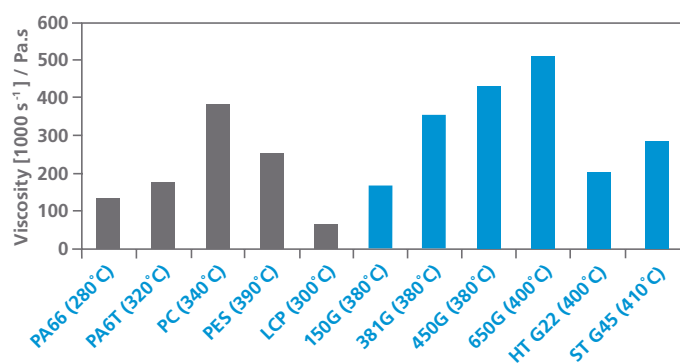
Although Victrex materials are supplied nominally dry, pellets may absorb up to 0.5% atmospheric moisture. It is essential to dry pellets to moisture content below 0.02% prior to processing, since traces of moisture create high vapour pressure at PAEK processing temperatures, resulting in foamy extrudate.

Pellets may be dried in an air circulating oven for 3 hours at 150°C or overnight at 120°C. For larger quantities such as during serial production desiccant dryers with a dew point of -40°C may be preferential.

PROCESSABILITY

A comparative plot of melt viscosities at a shear rate of 1000 s^{-1} for a range of high performance polymers is shown in Figure 1. Although PEEK, HT and ST have some of the highest processing temperatures, their melt viscosities are in the range of polyamide (PI) and polycarbonate (PC) melts.

Figure 1: Shear viscosities at a shear rate of 1000 s^{-1} at typical processing temperatures for a range of thermoplastics



COLOUR

Unfilled and glass fibre reinforced PEEK, HT and ST are available in natural/beige and black. Other compounds are coloured by their constituent materials, i.e. carbon-fibre reinforced materials are black.

Product of other colours may be obtained by either adding masterbatch to natural PEEK, HT or ST or by purchasing pre-coloured product from a Victrex partner.

Please contact your local Victrex representative for further information.

EQUIPMENT

GENERAL DESIGN

Polymers and compounds based on PEEK, HT and ST are readily extruded using conventional processing technology. There are specific requirements which are detailed below.

Extruders should be capable of reaching 400°C for PEEK or 430°C for HT and ST.

Venting is not required. Gear pumps may be used but care must be taken to ensure no dead spots (i.e. gaps around flanges or badly fitting blanking plugs) are introduced to the system as these may cause the formation of gels and black specks. A well-designed screw with stable pressure output should be sufficient for most extrusion processes.

Aim for homogeneous melt with a constant delivery rate at the desired temperature. It is recommended that melt temperature and pressure is monitored to provide adequate control over the process.

The residence time of the polymer in the process equipment influences the quality of the final product. The thermal stability of PEEK is exceptional; whereas due to increasingly high crystalline melting points, HT and ST may be considered as good and fair, respectively. It is possible for degradation to occur during processing for all materials when residence times are extended and / or barrel temperatures are high. Therefore, the capacity and throughput of the extruder should be matched to minimise the chances of formation of gels and black specks in the system.

MATERIALS OF CONSTRUCTION

The key requirement on steels suitable for melt processing is temperature, which needs to be maintained at 400°C for PEEK and 430°C for HT and ST. Abrasive wear of glass or carbon fibre filled Victrex materials is similar to that of other engineering thermoplastics. There are no corrosive decomposition products when processing Victrex materials, except when processing PTFE containing wear grades under excessive temperatures and/or residence times. Equipment suppliers offer extruders off-shelf capable of operating under these conditions.

In case a material needs to be specified, the use of bimetallic barrels (i.e. WEXCO 777 or Xaloy X-800) and powder metallurgical (e.g. CPM-9V or CPM-10V) or chrome alloy screws have proven satisfactory.



Rigorous attention to detail when extrusion processing ensures the highest quality product.

Nitrided materials should only be used with the utmost care to ensure that Victrex materials do not solidify in contact with the nitride surface coatings as these may detach from the steel substrate. Avoid copper and copper alloys in contact with the melt flow as these cause degradation of Victrex materials. For applications such as wire coating in which Victrex materials are in direct contact with copper or copper alloys the contact time between the melt and the metal surface is not sufficient to cause any issue before crystallisation and cooling occurs.

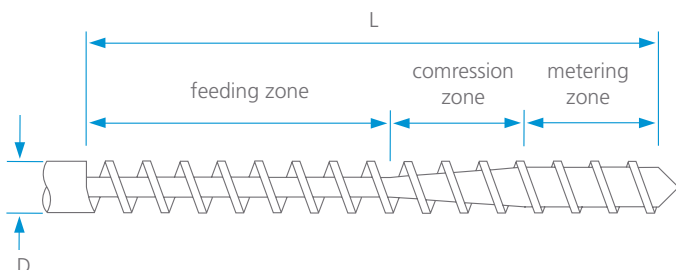
For the adapter, die and head, materials appropriate for the high processing temperatures (e.g. H13, Stavax, Hastalloy or Inconel) should be selected.

SCREW / BARREL DESIGN

A barrel length of 24:1 L/D ratio is the minimum that should be used, but processors may have a greater window of operation with a barrel approaching a 30:1 L/D ratio. The barrel should have a smooth bore. Grooved feed sections are not recommended.

As PEEK, HT and ST have higher melt temperatures compared to most conventional polymers, a longer feed section is desirable to allow sufficient residence time in this section of the screw for the pellets to approach the melt point. As depicted in Figure 2, a feeding zone of at least 8D is a good basis. PEEK, HT and ST do not undergo a drastic shift in viscosity around the melting point compared to other polymers such as polyamides or Liquid Crystalline Polymer (LCP). A gradual transition from the feed section to the metering section is preferred, shorter compression sections of 5D may work but compression sections of 8D are better. Compression ratios of 2 to 3 will cover most extrusion process situations. Metering lengths of 8D are typical, though longer lengths can be used. It is possible to use gentle mixing devices in the metering section if it is felt necessary. The screw tips should be rounded or conical shaped to avoid dead zones forming at the end of the screw.

Figure 2: Recommended screw type



BARREL CAPACITY AND RESIDENCE TIME

The size and the output of the extruder should be matched to obtain a residence time of ideally less than 30 minutes. Running an extrusion process at low screw rpm (<10rpm) will lead to longer residence times and increase the chances of process issues occurring due to a longer thermal history. There should be no 'dead spots'. All internal surfaces should be cleaned and polished before extrusion commences.

BARREL HEATING

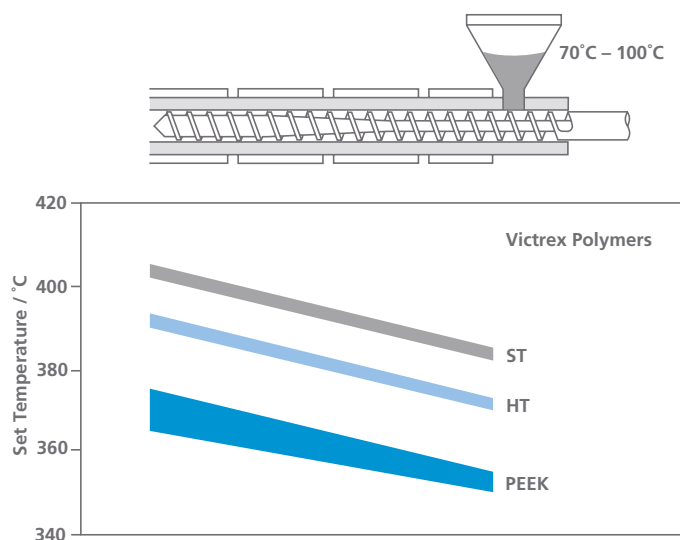
Cylinder heaters must be able to maintain 400°C for extruding PEEK and its compounds, or 430°C for extruding HT and ST and their compounds. Most extrusion machines are capable of reaching these temperatures without the need for modification. If modifications are required these are usually to upgrade heater bands and / or controllers. The preferred ceramic heater bands provide the most consistent control compared to mica heater bands. In addition, barrel blankets may be used to offer processing and cost saving benefits.

Thermal conduction along screw and barrel to the hopper may reduce feed efficiency. To achieve correct hopper feeding, the feed throat should be maintained between 70°C and 100°C. Thermal control may be achieved by water cooling, but care must be taken to maintain the rear zone temperature.

It is recommended that at least four separate heating zones are used, each with its own thermocouple and Proportional Integral Derivative (PID) controller for accurate temperature control. Care must be taken to ensure that sufficient heat is distributed along the entire length of the barrel and to all surfaces of the crosshead, and temperature should be controlled to within $\pm 2^\circ\text{C}$.

Typical barrel temperature settings are illustrated in Figure 3, more specific settings are shown in individual grade datasheets, these are available from your local Victrex representative.

Figure 3: Typical temperature profiles for Victrex materials



SCREENS AND BREAKER PLATE

The breaker plate helps develop back pressure in the screw and stops rotation of the melt; hole size should be in proportion to the extruder size and designed to eliminate dead spots wherever possible. Holes should be chamfered to enhance flow. Usually filter packs are placed in front of the breaker plate to remove any residual impurities in the material or contamination accidentally added and to further help build pressure in the screw. However, the filter pack should not be so fine as to create excessive shear in the melt or excessive pressure drops. Pressure should be monitored and filter packs changed when necessary. Heating and insulation of this area is critical.

ADAPTER, HEAD AND DIE

Dead spots / low flow can lead to local degradation leading to discoloured material or black specks in the melt. Therefore, flow through the adapter, head and die should be streamlined to prevent dead spots / low flow, cross section changes should be gradual and joints must be aligned and streamlined. Modelling of the system can be done to ensure there are no areas of low flow: sections should be reduced if there are concerns. Separately controlled heater zones (of sufficiently large size) are recommended for each section (adapter, head and die), with insulation to be applied wherever possible.

PURGING AND SHUTDOWN

Normal shutdown procedure involves emptying the extruder first followed by a complete step by step strip down and cleaning of all tooling, including removal of the screw. Some components will need to be placed into a furnace for full cleaning. For daily production it may be necessary to have two sets of dies and screws for each extruder.



Critical tolerances can be achieved with the correct process parameters and die design.



Extruded VICTREX Pipes.

The DDR is expressed as a ratio of the cross sectional area of the annulus to that of the final coating. The recommended draw-down ratio for natural PEEK, HT and ST is between 3:1 and 10:1, although DDRs close to 50:1 have been reported for very thin wall coatings. Close attention should be paid to the DBR, which should be maintained as close to 1:1 as possible.

The crosshead design for tube-on systems is not critical to the process. However, the preferred design is a single flow splitter which redirects the melt through 90° while maintaining good stream-lined flow. Although more complex flow splitters have proved satisfactory in service, these systems are more difficult to clean.

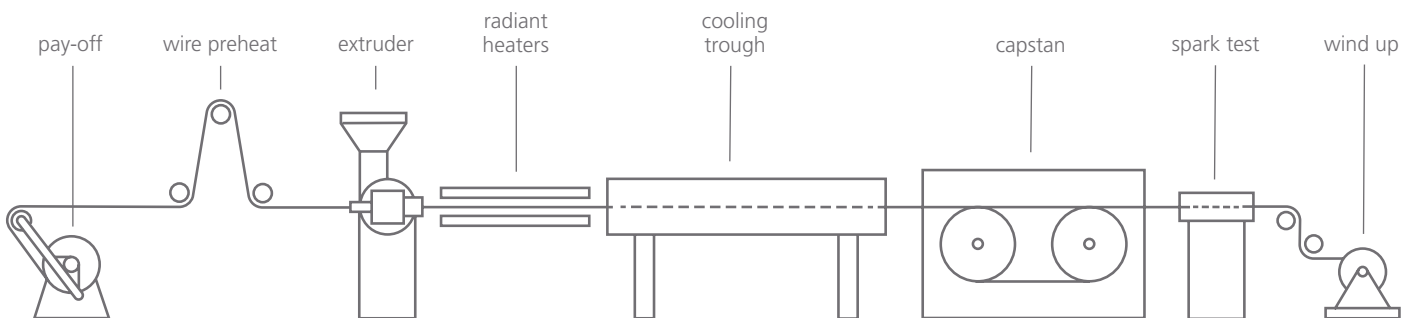
DOWNSTREAM EQUIPMENT

A schematic diagram of a basic wire coating setup is shown in Figure 6. However requirements vary depending on the wire coating application so every line has differences.



Appropriate downstream equipment allows the process to be tailored to achieve the required crystallinity.

Figure 6: Wire Coating Schematic Diagram



CRYSTALLINITY

Many of the outstanding physical properties of PEEK, HT and ST result from their semi-crystalline morphology.

In wire and cable coating, the melt is drawn from the die crosshead and allowed to cool in air for approximately 1 meter (depending on line speed). While cooling, the colour of natural PEEK, HT and ST changes from transparent dark brown to an opaque grey/beige. This change in colour is due to cooling and crystallisation of the surface of the insulation.

Once this transition has taken place, additional water-cooling may be used since crystallisation within the bulk of the molten polymer will not be affected significantly. Should an amorphous wire coating be required the water bath may be moved closer to the die.

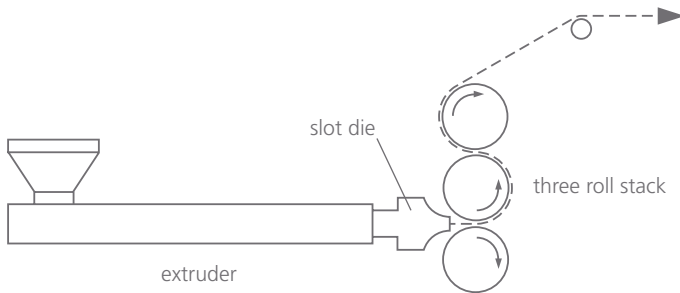
The temperature of the conductor may retard crystallisation in wire and cable coating operations.

Therefore, when high crystallinity is required, it is advisable to heat the conductor prior to entry into the crosshead. The preheat temperature will depend on the nature and geometry of the conductor, but excellent results have been achieved with temperatures in the 120°C to 200°C range. If the desired level of crystallinity cannot be achieved on-line, it is possible to post-crystallise the insulation by subsequent thermal treatment.

FILM AND SHEET MANUFACTURE

Natural PEEK, HT and ST may be used to form film/sheets. The processing is carried out using a conventional extruder with suitable die and haul-off equipment, as depicted in Figure 7.

Figure 7: Sheet Producing Equipment



DIE DESIGN

Slot dies are usually used for processing natural PEEK, HT and ST into sheets. These systems should have a streamlined melt flow with polished interiors to prevent hold-up or die stick-slip. Control of the temperature of the die lip is crucial for good surface finish and dimensional control.

THIN FILM AND SHEET CRYSTALLINITY

Thin sheet (<500 μm) may be produced in either semi-crystalline or amorphous form by controlling the temperature of the casting drums. A drum temperature below T_g will produce an amorphous transparent film, while a temperature above 170°C will give opaque semi-crystalline film. Thicker films crystallise under their own retained heat.



Measuring the thickness of extruded APTIV Film.



Equipment cleanliness and tight process control is essential for achieving high quality film.

MONOFILAMENT

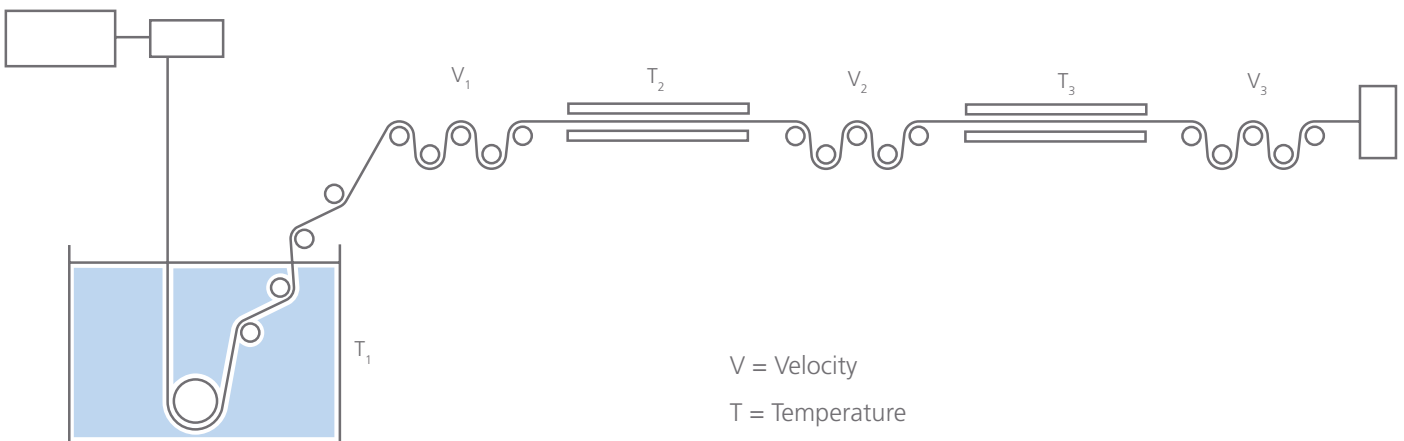
PEEK, HT and ST may be processed into monofilament using an extruder with downstream haul-off and draw facilities. Extruders used for the production of monofilament are generally fitted with gear pumps, which ensure that an accurately metered supply of melt is fed to the die at constant pressure. Other non-metered systems have proved satisfactory in service. A typical monofilament production line is depicted in Figure 8.

The post-extruder processing depicted in Figure 8 can be considered in two distinct parts: melt orientation and relaxation. During melt orientation the extrudate is air and water cooled. The filament is drawn at a higher velocity ($V_2 > V_1$) through an oven which is set above the glass transition of the material. The increased velocity serves to draw the polymer, reducing the diameter and orienting the filament.



Extruded VICTREX PEEK fibres.

Figure 8: A schematic representation of monofilament production equipment



During melt relaxation, the polymer is 'heat set' by passing through a second oven which is close to the melt temperature of the material.

TECHNICAL SUPPORT

Victrex Polymer Solutions is uniquely committed to polyaryletherketone products and is well-placed to meet your full range of quality, technical and supply security requirements. In today's competitive environment, working with a leading supplier with advanced technologies and most in-depth and responsive technical services available can be critical for success.

If you would like more information or assistance, please contact your local Victrex Polymer Solutions representative or visit us at www.victrex.com.



Based in the UK, Victrex is an innovative and world leading global provider of high-performance polymer solutions for the aerospace, automotive, electronics, energy and medical industries. Every day, millions of people rely on products and applications containing our polymers – from smart phones, aircraft and cars all the way to medical devices and oil and gas installations. With over 35 years' experience, we provide cutting-edge technological solutions that shape future performance for our customers and markets and drive value for our shareholders.

www.victrex.com

World Headquarters

Victrex plc
Hillhouse International
Thornton Cleveleys
Lancashire
FY5 4QD
United Kingdom

TEL +44 (0)1253 897700
FAX +44 (0)1253 897701
MAIL victrexplc@victrex.com

Americas

Victrex USA Inc
300 Conshohocken State Road
Suite 120
West Conshohocken
PA 19428
USA

TEL +1 800-VICTREX
TEL +1 484-342-6001
FAX +1 484-342-6002
MAIL americas@victrex.com

Europe

Victrex Europa GmbH
Langgasse 16
65719 Hofheim/Ts.
Germany

TEL +49 (0)6192 96490
FAX +49 (0)6192 964948
MAIL customerservice@victrex.com

Japan

Victrex Japan Inc
Mita Kokusai Building Annex
4-28, Mita 1-chome
Minato-ku
Tokyo 108-0073
Japan

TEL +81 (0)3 5427 4650
FAX +81 (0)3 5427 4651
MAIL japansales@victrex.com

Asia Pacific

Victrex High Performance
Materials (Shanghai) Co Ltd
Part B Building G
No. 1688 Zhuanxing Road
Xinzhuan Industry Park
Shanghai 201108
China

TEL +86 (0)21-6113 6900
FAX +86 (0)21-6113 6901
MAIL scsales@victrex.com

©Victrex plc September 2016

Victrex plc believes that the information contained in this document is an accurate description of the typical characteristics and/or uses of the product or products, but it is the customer's responsibility to thoroughly test the product in each specific application to determine its performance, efficacy, and safety for each end-use product, device or other application. Suggestions of uses should not be taken as inducements to infringe any particular patent. The information and data contained here in are based on information we believe reliable. Mention of a product in this document is not a guarantee of availability. Victrex plc reserves the right to modify products, specifications and/or packaging as part of a continuous program of product development. Victrex plc makes no warranties, express or implied, including, without limitation, a warranty of fitness for a particular purpose or of intellectual property non-fringement, including, but not limited to patent non-infringement, which are expressly disclaimed, whether express or implied, in factor by law. Further, Victrex plc makes no warranty to your customers or agents, and has not authorized anyone to make any representation or warranty other than as provided above. Victrex plc shall in no event be liable for any general, indirect, special, consequential, punitive, incidental or similar damages, including without limitation, damages for harm to business, lost profits or lost savings, even if Victrex has been advised of the possibility of such damages regardless of the form of action.

VICTREX™, APTIV™, VICOTE™, VICTREX PIPES™, VICTREX HT™, VICTREX ST™, VICTREX WG™, PEEK-ESD™ and the Triangle (Device), are trademarks of Victrex plc or its group companies.

