The Effect of Melt Spinning Process Parameters on the Spinnability of Polyetheretherketone

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ABSTRACT: This study has been carried out to investigate the processing parameters affecting polyetheretherketone's (PEEK) spinnability in a melt spinning process. PEEK has excellent mechanical and thermal properties and fibers made from it could be used in extreme environments. Different PEEK grades were characterized thermally and rheologically to see which one is the most suitable for fiber spinning. The spinning tests made with the most suitable grade (Victrex 151G) show that increased processing temperature, increased capillary diameter or shorter spinning path length improves spinnability. The best fibers made in optimal processing conditions (400°C temperature, 30/1 mm capillary, and 5 cm spinning path) were 18 μm in average diameter. Because of the limitations of the system used, variations in fiber thickness were noticeable and worsened the spinning stability. Scanning electron microscope photos confirmed these variations, and they were also visible in an optical microscope. The selected low-viscosity PEEK grade provided good spinnability but gave filaments with only mediocre mechanical properties, the tensile strength being around 280 MPa. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: PEEK; melt spinning; fiber, spinnability, optimization

INTRODUCTION

Polyetheretherketone (PEEK) is a semicrystalline thermoplastic with excellent mechanical and thermal properties and chemical resistance. PEEK has very low water absorption, low flammability, good radiation resistance, and good electrical properties. PEEK's continuous use temperature of 260°C is one of the highest among all plastics. It is approved by food and drug administration. Because of its high price (100 €/kg) PEEK is normally used in high-tech applications such as medical devices, space applications, and as metal replacement.[1,2] The commercial applications of PEEK fibers are currently dry filtration, chemical separation, sport strings, braids, brushes, and cords.[3]

Melt spinning is a process where melt polymeric material is pushed through a small-hole (spinneret), then cooled down by cold air and finally drawn onto a roll by a winding unit.[4] Melt spinning of PEEK is not a new invention. In fact, it has been done since the 1980s.[3] There have been studies concerning melt spinning of ultrafine PEEK filaments[5,6] and even one dissertation about PEEK melt spinning.[7] Ziabicki first stated the six processing parameters affecting spinnability in a melt spinning process: processing temperature, dimensions and the number of spinneret holes, mass throughput, length of the spinning path, take-up velocity and cooling conditions.[4] There are previous data available on how processing parameters affect spinnability generally[4,8–12] and also for PEEK,[6,7] but in these studies, a deeper understanding regarding the relationship between processing parameters and the best spinnable fiber diameter is lacking. Fourne and Golzar have given fairly similar list of suggestions how to spin very thin filaments.[7,9] However, these recommendations do not state what are the actual effects on the spinnability. Golzar has also created a fiber stability map for Victrex PEEK 151G but this map do not show the effects of PEEK grade, processing temperature, capillary dimensions, and the length of the spinning path to the best spinnable fiber diameter.[7] Although the effects of processing parameters are well known in a qualitative level, there is limited amount of literature data available how these processing parameters affect the best spinnable fiber diameter on a quantitative level.

Theoretically, an increase in processing temperature should improve spinnability as Fourne first suggested in 1995.[9] According to Golzar's tests, this is the case with PEEK also.[7] The normal processing temperature of PEEK is 360–400°C, so 400°C should provide the best spinnability. Repkin have shown that increasing the capillary diameter improves process quality and decreases the number of failures.[12]

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In addition to the diameter, the length/diameter (L/D) ratio also affects spinnability. Several publications have shown that the die swell-ratio (the ratio of maximum polymer mass diameter after the capillary and the diameter of the capillary) decreases when L/D-ratio increases.[9,11] It is important to minimize the die swell because it weakens the quality of the fibers by making them more irregular.[4] This means that theoretically capillary should be long and large. Previous studies have shown that short spinning paths should preferably be used when making ultrafine filaments.[7,9] Stress in the spinning line increases along with the spinning path length thus increasing the change of failure.[7,13]

The goal of this project is to manufacture fine PEEK filaments by a lab-scale melt extrusion spinning process and to optimize the most important processing parameters. To get filaments of even quality, and with optimal properties, it is necessary to fine-tune the process parameters (such as piston speed, melt temperature, length of cooling path, etc.). The obtained PEEK filaments where then thoroughly characterized so that any material related problems (e.g., thermal degradation of PEEK, filament breaking) can be minimized during the fiber spinning. The obtained results from the PEEK fiber characterization were then compared with results from previous studies reported in the literature and were also used to evaluate the spinning process.

EXPERIMENTAL

Materials

Four PEEK grades from Victrex (Lancashire, UK) were used in the study. Three of the grades are in granular form: 151G, 381G, and 450G, and one grade, 704, is a powder. These grades have different molecular weights and rheological properties according to the supplier data.[14] The thermal properties are very similar between the grades, whereas the mechanical properties have more variation. The properties for the granulate grades can be seen in Table I. Powder grade 704 is designed for coatings but was briefly tested in the fiber spinning trials also.

TABLE I
Properties of the PEEK Grades Used[14]

Property\grade	151G	381G	450G
Melting point (°C) ISO11357	343	343	343
Glass transition (°C) ISO11357	143	143	143
Melt viscosity (Pa s) ISO11443	130	300	350
Tensile strength (MPa) ISO527	110	100	100
Tensile modulus (GPa) ISO527	3.9	3.7	3.7
Tensile elongation (%) ISO527	25	40	45
Izod impact strength (kJ m^{-2}) ISO180/A	5.0	6.5	7.5

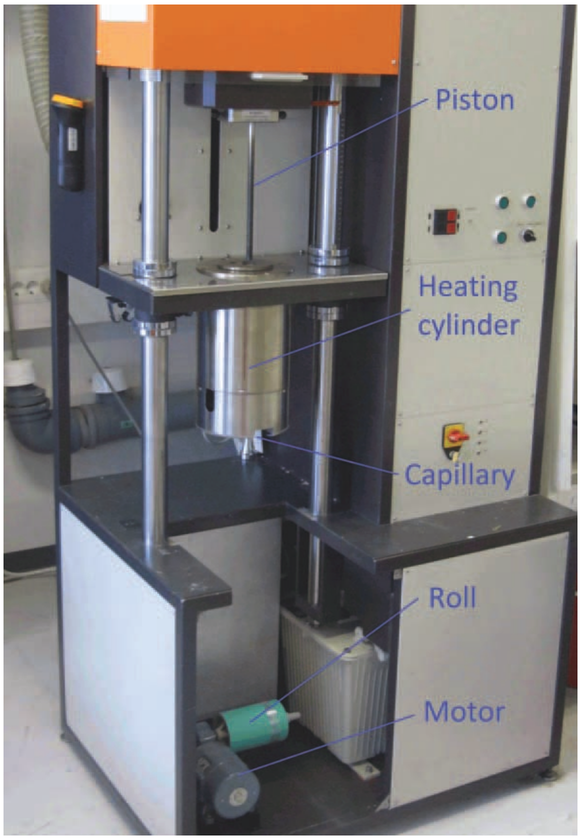

Figure 1 Göttfert capillary rheometer. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Melt spinning

The PEEK melt-spinning system was based on a modified Göttfert Rheograph 6000 capillary rheometer, which can be seen in Figure 1. It is a piston-based system with a maximum temperature of 400°C and volume of about 26 cm^3. The barrel is 12 mm in diameter and 230 mm in length. The maximum length of the capillary used is 30 mm, and its typical diameter is 1 mm. It is possible to manufacture only monofilaments with the system. The filament winding motor unit is located under the capillary and has a maximum speed of about 350 RPM. The roll used in the winder has a diameter of 87 mm, giving a fiber production rate of roughly 100 m/min at full motor speed.

The limit of spinnability was evaluated experimentally. Step by step, the piston speed (fiber diameter) was decreased until the limit was found. If the fiber did not break in 1 min, the process was estimated to be stable. If this was not the case during the first five trials then the limit was found.

Characterization

Differential scanning calorimetry (DSC) tests were carried out in a Netzsch DSC 204 F1 heat-flux DSC.

All the tests were carried out in nitrogen atmosphere. This corresponds to the melt spinning conditions used, as PEEK is not in contact with oxygen in the closed barrel. During the conventional DSC test, PEEK was heated from room temperature to 400°C, then cooled down to room temperature and then heated once more. The heating/cooling rate was 20°C/min. The PEEK sample was also kept at constant 400°C temperature in a DSC analyser for about 18 h to see whether it degrades thermally. The thermogravimetric analysis (TGA) tests were made with a Perkin-Elmer TGA 6. The PEEK samples were heated in a nitrogen atmosphere at a rate of 10°C/min from room temperature to 1000°C.

Capillary rheometer tests were made with the same Göttfert Rheograph 6000 capillary rheometer which was used in the melt spinning. During the rheometer tests a 30/1-mm capillary was used. Test runs were carried out from low to high shear rates and then back. A 140-MPa pressure sensor was used during the tests. The Rabinowitsch correction was made to the capillary rheometer data. The Bagley correction could not be made because only one capillary was used during the capillary rheometer tests.

Rotational rheometer characterizations were carried out with Anton Paar Physica MCR 301 equipment. Tests were carried out with plate–plate geometry in a nitrogen atmosphere. The used tests were constant shear rate tests from low to high shear rates.

The mechanical testing of the filaments was made according to the standard ISO 5079:1995 "Textiles fibres—Determination of breaking force and elongation of individual fibres" by using Lenzing Vibroskop and Vibrodyn. Instead of the recommended 50 measurements, only 20 were done because PEEK is a synthetic fiber and causes problems with the Vibroskop. First, the Vibroskop was used to determine the Tex number and then the same fiber was stretched until breaking. The advantage of measuring the Tex number first is that more accurate data are obtained because fiber cross-sectional density is known.

A conventional optical microscope was used to measure the fiber thickness at ×1000 magnification. A bunch of fibers was spread onto the microglass plate, and by using the microscopes rotating measuring scale the filament diameter could be read. The diameters of total of 60 individual fibers were measured, to ensure reproducibility. A Philips XL30 scanning electron microscope (SEM) was used to investigate the morphology of the produced PEEK filaments.

Theoretical calculations

The piston speed in the Göttfert capillary rheometer can be controlled by a computer in a fairly accurate manner. The control program calculates the shear rate for different capillaries and piston speeds.

Mass throughput ($m_{\text{throughput}}$) can be calculated from the piston speed (v) if the material density is known, using Eq. (1)—PEEK's melt density (ρ) varies as a function of temperature and pressure, which makes the calculations for exact mass throughput difficult. Therefore, the density's literature value for solid PEEK (1.32 g/cm^3) has been used, which may cause some inaccuracy. Barrel radius (r) is 0.6 cm. The unit for v_{piston} is (cm/s), barrel radius is (cm), and for $m_{\text{throughput}}$ (g/min).

$$m_{\text{throughput}} = v_{\text{piston}} * \pi * r_{\text{barrel}}^2 * \rho * 60\,\text{s/min} \quad (1)$$

The theoretical fiber diameter can be expressed as a function of the piston speed. The very simplified formula for fiber diameter in the system used is:

$$\varnothing = 0.30\,(\text{mm/s})^{0.5} * (v_{\text{piston}})^{0.5} \quad (2)$$

where the fiber diameter Ø is in mm, and v_{piston} in mm/s. A more detailed formula can be found in Reference 15.

The Tex number is commonly used to measure sizes in linear and continuous products such as cables and fibers. It is a measure of linear mass density. One tex is the mass in gram for a 1000-m-long filament, and it is commonly regarded as the fiber fineness. Decitex is also used, it is the mass in grams for a 10,000-m-long fiber. The decitex-number can be expressed as a function of the fiber diameter if the density of the material is known. In the calculations, PEEK's density of 1.32 g/cm^3 has been used.

$$\text{dtex} = 250,000 * \varnothing^2 * \pi * \rho \quad (3)$$

The unit of dtex is (g/10,000 m), Ø (cm), and ρ (g/cm^3).

RESULTS AND DISCUSSION

Thermal characterization

Before starting the fiber spinning tests, the PEEK grades were characterized thoroughly. Theoretically PEEK should have excellent thermal properties, and the thermal characterization techniques DSC and TGA were used to verify this. These thermal tests were done on the grades 151G and 381G.

A conventional DSC test, where the temperature changes at a constant rate, shows that both grades have good and almost similar thermal properties (Fig. 2). This is in line with the literature data provided by Victrex.[14] The peak melting temperature is around 340°C. The curves for the first and second heating are almost similar, so there is no evident

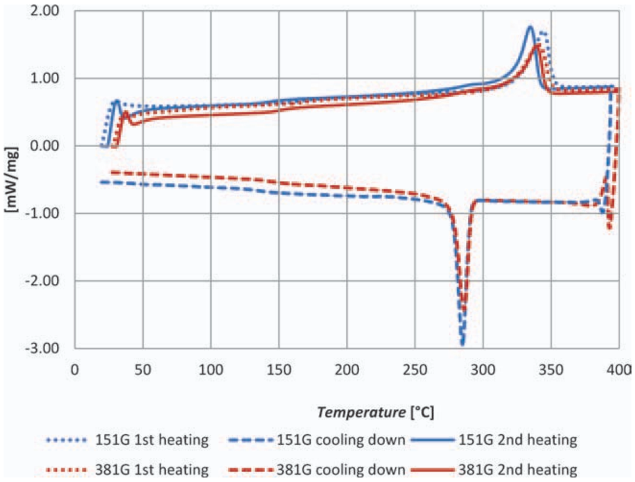

Figure 2 DSC scans for grades 151G and 381G. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

thermal history, and no thermal degradation occurs during the measurements. Victrex recommends the minimum processing temperature to be 360°C, and according to the DSC tests, PEEK is completely melted at this temperature.

A DSC test where the PEEK grades 151G and 381G were kept under nitrogen atmosphere at 400°C for 18 h did not show any signs of thermal degradation, because the curve is a flat line. This is important because during the processing in the equipment PEEK may stay for hours at high temperatures in the barrel. Thermal degradation would worsen the mechanical properties and make fiber spinning more difficult or even impossible. This thermal stability is in line with the expected thermal stability for PEEK.

TGA test data in Figure 3 also shows the excellent thermal properties of PEEK. PEEK's mass remains the same up to a temperature of 550°C for both grades. For some reason, grade 381G loses mass faster than grade 151G at temperatures higher than 700°C, but this difference is not significant in the applications for PEEK fibers. At 1000°C, 40% of the mass of PEEK's grade 151G is still left, which must be considered remarkable for an organic plastic material.

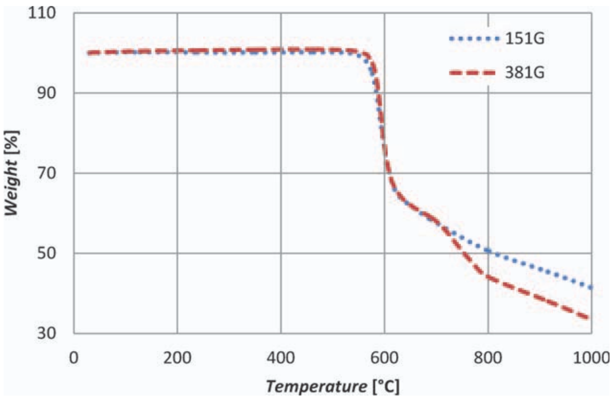

Figure 3 TGA test for grades 151G and 381G. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

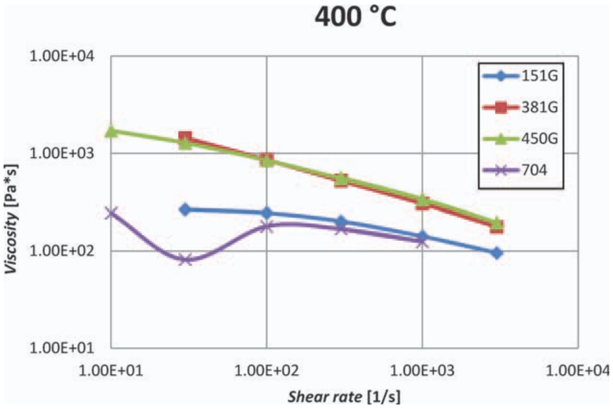

Figure 4 Capillary rheometer test at 400°C for grades 151G, 381G, 450G, and 704. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Rheological characterization

Viscosity tests at 400°C were made on all four PEEK grades and at the same time, the rheological behavior in the melt spinning was also evaluated. Figure 4 shows that the viscosities of grades 381G and 450G are almost similar. This is not surprising considering that the grade number indicates viscosity at 400°C temperature and 1000 1/s shear rate according to the Victrex own test TM-VX-12.[14] The viscosity of grade 151G is therefore considerably lower. The grade number does not correlate with the viscosity of the powder grades, in fact the viscosity of grade 704 is the lowest of the used grades. Because of its powder form, it was very difficult to load grade 704 into the rheometer barrel. The powder got stuck into the barrel walls, and during the measurement, the piston had difficulties to move. Therefore, the test results with grade 704 are inconsistent at least with small shear rates, and the test was not repeated.

Capillary rheometer tests were carried out from low to high shear rates and then back. The obtained results give very similar data regardless of the direction of the measurement. The high viscosity grades 381G and 450G have more shear thinning effects than the low viscosity grade 151G. Fiber spinning tests are done at very small shear rates, so the differences in viscosities are in fact greater than the differences in the manufacturer's theoretical values.

Additional viscosity tests were made on grades 151G and 381G (Fig. 5) which seem to be the best candidates for fiber spinning. Grade 704 was not tested because of its powder form and grade 451G

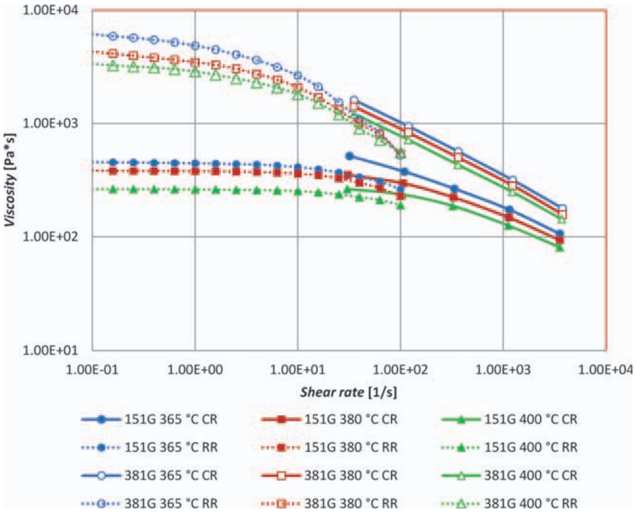

Figure 5 Capillary (CR) and rotational rheometer (RR) tests at different temperatures for grades 151G and 381G. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was left out of the testing because it is very similar to grade 381G. Grades 151G and 381G were tested at three different temperatures with a capillary and also with a rotational rheometer. Very small shear rates are difficult to test with a capillary rheometer, and therefore, a rotational rheometer was used.

The results show that the used PEEK grade affects viscosity more than temperature. PEEK grade 381G has a higher viscosity at 400°C than 151G has at 365°C. Shear thinning of grade 381G begins at significantly low shear rates compared with grade 151G. Values measured with the capillary rheometer are a little higher than those measured with the rotational rheometer at the same shear rates. One reason for this is that the capillary rheometer tests were made with only one capillary, and therefore, the Bagley correction could not be made. The second possible reason is the melt fracture effect that happens at high shear rates with the rotational rheometer. This is evident at least with grade 381G at shear rates higher than 20 1/s.

Fiber spinning

Four PEEK grades were initially used in the experimental study, and according to the preliminary tests, one of these grades, 151G, was selected for the large-scale melt spinning experiments. The decision was based not only on fiber properties but also on the ease to load and clean the material. The spinning properties of the grades can be found in Table II. To find the optimal melt spinning parameters, a rather extensive experimental program is necessary to do, and therefore the chosen PEEK grade should be not only easy to spin but also easy to load into the barrel and clean afterwards, to minimize the time for the trials.

The differences between fiber properties (minimum fiber diameter) were so remarkable that very precise tests for all grades were not needed. The powder grade 704 was almost impossible to load into the barrel, which is unfortunate because it seemed to give fibers with very good properties. Preliminary tests indicate that good fiber properties are related to low viscosity because lower viscosity grades seemed to have better spinnability. Lower viscosity grades were also easier to clean afterward because they did not stick tightly to the piston and barrel walls.

Testing of all possible process parameter combinations would not have been possible for practical reasons. Therefore, it was decided to first evaluate the more important parameters and then use the obtained results in subsequent experimental work. The importance of a parameter was estimated by the preliminary tests and data obtained from the literature review. The order of the parameters tested was the material grade, processing temperature, capillary dimensions, and the length of the spinning path. The results can be found in Table III.

As the preliminary tests showed, low-viscosity grades have much better spinnability compared to high-viscosity grades. The differences in average filament diameter are significant. The best fibers achieved with grade 381G were 39 μm in diameter and for grade 151G 19 μm in diameter.

The first actual processing parameter tested was the processing temperature. As mentioned in the introduction, an increase in processing temperature should improve spinnability and according to our tests, this is really the case. The best processing temperature was 400°C. However, the improvement is rather small compared to processing at 385°C. At 370°C, it was difficult to get the process stable at all because the fiber diameter varied all the time. At higher temperatures, this problem was not visible to the naked eye.

The second tested parameter was the capillary dimensions. There are a lot of combinations for capillary diameter and length. Unfortunately, there were several problems especially with short capillaries. The only suitable capillary length turned out to be 30 mm, which is the longest possible in the system used. Spinning with shorter capillaries was

TABLE II
Grade Selection Table

Grade	151G	381G	450G	704
Fiber properties	Good	Moderate	Moderate	Very good
Loading	Easy	Easy	Easy	Very difficult
Cleaning	Normal	Difficult	Difficult	Normal

TABLE III
Spinnability in Different Processing Parameters

Grade	381G	151G	151G	151G	151G	151G
Temperature (°C)	400	400	385	370	400	400
Capillary diameter (mm)	1.0	1.0	1.0	1.0	0.75	1.0
Length of the spinning path (cm)	40	40	40	40	40	5
Shear rate (1/s)	19.0	4.6	4.8	7.9	18.0	4.0
Lowest stable piston speed (mm/s)	0.0165	0.0040	0.0042	0.0069	0.0066	0.0035
Theoretical mass throughput (g/min)(1)	0.15	0.036	0.038	0.062	0.059	0.031
Theoretical diameter (μm)(2)	39	19	19	25	24	18
Theoretical tex-number (dtex)(3)	15.4	3.7	3.7	6.5	6.2	3.3

impossible because the process was stable for only a few seconds at best. The reason for this is most likely the high temperature air PEEK encounters when using short capillaries. If a full-length capillary is not used then PEEK comes out inside a hot tube (the capillary is inside the barrel). The second problem was the gravitational self-flow of PEEK when using very large capillaries (>1 mm). With a 1.5 mm capillary, the process was stable without the piston movement. Therefore, only two capillaries were tested: 1.0 and 0.75 mm. Spinnability turned out to be considerably better with a 1.0 mm capillary.

The third tested parameter was the length of the spinning path. Normally, the spinning path is several meters long, but there was not much room under the capillary in the used experimental setup. Therefore, spinning path length could not affect the spinnability in the system used significantly. Most of the tests were made with a 40-cm spinning path, but one test was made with very short spinning path and it improved spinning stability even further. Very thin fibers cool down rapidly, so very short spinning paths can be used.

As a conclusion, the best processing parameters in the system used were the 400°C processing temperature, 30/1 mm capillary, and very short spinning path. The best spun fibers were 18 μm in average diameter. According to these tests, process optimization should be started from the material grade, because it affects more to the viscosity than any other parameter. Mass throughput of grade 381G had to be increased by 317% to get the process as stable as grade 151G is in similar spinning conditions. Processing temperature should be above 385°C because then the increase in stable mass throughput is only 6% from 400 to 385°C but 72% from 400 to 370°C. Changing capillary diameter from 1 to 0.75 mm worsens spinnability nearly as much as decreasing temperature from 400 to 370°C. The length of the spinning path is the least important of these processing parameters because the increase in stable mass throughput is only 16% from 5 to 40 cm. In industrial scale systems, the spinning path is longer thus affecting more to the spinnability. Obtained spun PEEK fibers can be seen in the optical micrographs in Figure 6. The optical microscope confirms the huge variations in fiber thickness, the average being 25 μm and the standard deviation 5.2 μm. The minimum detected value was 14 μm and maximum 36 μm. Variations can also be seen in the more detailed SEM micrograph in Figure 7.

Figure 6 Spun PEEK filaments. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Mechanical properties and fineness

The obtained filaments were tested to evaluate their fineness and tenacity. For the mechanical tests, a

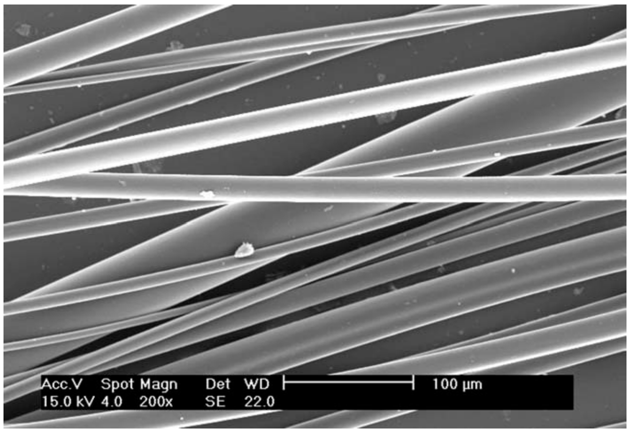

Figure 7 SEM photo of PEEK fibers.

sufficient amount of 25-μm diameter fiber was manufactured according to the otherwise optimal processing parameters (Victrex 151G, 400°C, 30/1 mm, 5 cm, and 100 m/min). The following can be concluded from the test data: The obtained fiber diameter is not homogenous, as Tex numbers vary from 3.1 to 9.9 dtex, with an average of 6.2 dtex. The tensile strength for the filaments is 2.2 cN/dtex, corresponding to about 280 MPa (ratio is 100ρ). This is slightly below the expectations based on previously reported studies made with PEEK.[7] The standard deviation of tensile strength is very small, only 0.24 cN/dtex, which indicates good experimental reproducibility. Elongation at break, however, varies more, the average being 150%, which is much more than anticipated. It is evident that thick fibers have higher elongation at break. This could mean that there is a limit in thickness near 10 μm where PEEK fibers break. Fiber parts that have stretched relatively more during the winding cannot stretch more during the tensile testing and vice versa. Young's modulus at 1% elongation is higher with thinner fibers, the average being about 19.7 cN/dtex (2.6 GPa).

The Victrex grade 151G has a very low viscosity probably as a result of a small molecular weight. This means that many of the mechanical properties are inferior compared to higher viscosity grade PEEK's.[16] Another reason for the low mechanical properties is the low degree of crystallisation of PEEK fibers measured by a DSC test (at 100% crystallization level $\Delta H_F = 130$ J/g).[17] According to this DSC test, 25 μm fiber had only 11% degree of crystallization which is very low compared with the theoretical maximum of about 40%. Golzar's tests show that increasing the take-up velocity or decreasing the mass throughput increases the degree of crystallization.[7] In the spinning system used, the bottleneck is the spinning motor which is not capable of higher take-up speeds than 100 m/min.

CONCLUSIONS

Fiber spinning of PEEK is relatively easy despite the high melt temperatures needed. The results of thermal tests confirm the very unique thermal properties of PEEK polymers. During the fiber spinning tests, the best achieved fibers were 18 μm in average diameter. There are several processing parameters affecting the limits of spinnability. The most important parameters were estimated to be viscosity related; material grade and processing temperature. Spinnability improved when the material viscosity decreased. However, viscosity could not be decreased limitlessly, because at some point, PEEK started to flow out from the capillary faster than piston moved.

The biggest drawbacks of the melt spinning system were related to Göttfert's structure which is designed for viscosity measurements, not for fiber spinning. A typical conventional spinning line cannot be used with PEEK because of the high processing temperatures needed. The biggest downside of the system used is the resulting high variations in obtained fiber thickness. This most definitely affects the limits of spinnability because the thinnest fiber parts were about 10 μm in diameter. The second reason for the big variations may be the motor system used. Although easy to control and use, it is still a bit old-fashioned. The requirements for very thin filaments are high and they do not allow any rough acceleration.

If the setup were to be optimized further then the first step would be a new motor system. The structure of Göttfert cannot be changed, except by replacing the used capillaries with capillaries of another size. However, a basic problem with process is the absence of mixing, which is present in all piston type melt spinning systems.

One important processing parameter which could not be tested in this study was the windup motor speed's effect on the limits of spinnability. The motor system used had no RPM screen and therefore had to be used at full speed. Further and more precise experimental trials would require a more advanced motor system such as a precise AC servo motor.[18] It would also be useful to know the mechanical properties of fibers of a same diameter manufactured at different take-up velocities.

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References

1. Platt, D. K.Engineering and High Performance Plastics Market Report; Smithers Rapra Technology: Shawbury, 2003.
2. Sabu, T.; Visakh, P. M. Handbook of Engineering and Speciality Thermoplastics: Polyethers and Polyesters; New York: Wiley, 2011; Vol. 3, Chapter 3, p 55.
3. Zyex Ltd. Manufacturer of PEEK fibers. Available at: http://www.zyex.com. Accessed June 13, 2011.
4. Ziabicki, A.Fundamentals of Fiber Formation; Wiley-Interscience: London, 1976.
5. Shekar, R. I.; Kotresh, T. M.; Rao, P. M. D.; Kumar. K. J Appl Polym Sci 2009, 112, 2497.
6. Brünig, H.; Beyreuther, R.; Vogel, R.; Tändler, B. J Mat Sci 2003, 38, 2149.
7. Golzar, M.Melt spinning of the fine PEEK filaments. Dissertation, Technische Universität Dresden, Dresden, 2004.
8. Gupta, V. B.; Kothari, V. K.Manufactured Fibre Technology; Springer: London, 1997.

9. Fourné, F. Synthetic fibers: Machines and Equipment, Manufacture, Properties: Handbook; Hanser Gardner: Munich, 1999.
10. Gupta, V. B.; Mondal, S. A.; Bhuvanesh, Y. C. J Appl Polym Sci 1997, 65, 1773.
11. White, J. L. Fiber and Yarn Processing; Wiley: New York, 1975.
12. Repkin, Y. S. Fiber Chem 1972, 3, 377.
13. Shimizu, J.; Kikutani, T. J Appl Polym Sci 2002, 83, 539.
14. Victrex, PLC. Leading manufacturer of PEEK. Available at: http://www.victrex.com. Accessed June 14, 2011.
15. Mylläri, V. Production of filament yarns made of PEEK. Master of Science Thesis, Tampere University of Technology, Tampere, April 2011.
16. Yuan, M.; Galloway, J. A.; Hoffman, R. J.; Bhatt, S. Polym Eng Sci 2011, 51, 94.
17. Blundell, D. J.; Osborn, B. N. Polymer 1983, 24, 953.
18. Firoozian, R.; Servo Motors and Industrial Control Theory; Springer: New York, 2008.