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# Natezne lastnosti polipropilenskih filamentov

*Izvirni znanstveni članek*

Poslano *maj 2008* • Sprejeto *november 2008*

## *Tensile Properties of Polypropylene Filaments*

Original Scientific Paper

Received *May 2008* • Accepted *November 2008*

## Abstract

*In this work, the tensile properties of polypropylene filaments, made by a melt-spinning and drawing process optimized in order to increase the elastic modulus of the filaments, are presented. The influence of molecular weight and the molecular weight distribution of the polymer on the tensile properties of the filaments, spun from polymers of different molecular weights with various composition ranges, was investigated. It was found that polypropylene filaments spun from a fiber-grade CR-polymer have better mechanical properties, when compared with filaments spun from a plastic-grade polymer with a broader molecular weight distribution. The characteristics of the filaments produced from binary blends are mainly dependent on the prevailing polymer in the blend; however, the addition of the other polymer exerts some influence as well. In both cases, a small percentage of added polymer improves the tensile properties of the filaments.*

*Keywords: polypropylene, polymer blend, tenacity, elastic modulus.*

## Izvleček

V prispevku so prikazane natezne lastnosti polipropilenskih filamentov, izdelanih po postopku talilnega oblikovanja in nadaljnjega raztezanja, optimiziranega za doseganje visokega modula elastičnosti. Raziskan je bil vpliv molekulske mase in porazdelitve molekulskih mas polimera na natezne lastnosti filamentov, izdelanih iz dveh polimerov z različno molekulsko maso in njunih mešanic v različnem utežnem razmerju. Ugotovljeno je bilo, da imajo filamenti, oblikovani iz polimera z nižjo molekulsko maso in ozko porazdelitvijo molekulskih mas, boljše mehanske lastnosti kot filamenti, oblikovani iz polimera z višjo molekulsko maso in široko porazdelitvijo molekulskih mas. Lastnosti filamentov, oblikovanih iz mešanic, so predvsem odvisne od prevladujočega polimera, vendar ima dodatek drugega polimera določen vpliv na lastnosti. Majhen delež drugega polimera v obeh primerih izboljša natezne lastnosti filamentov.

Ključne besede: polipropilen, polimerna mešanica, trdnost, modul elastičnosti.

## 1 Uvod

Mehanske lastnosti filamentov so predvsem odvisne od njihove nadmolekulske strukture (orientacije, stopnje kristalinosti, velikosti kristalitov, števila veznih molekul, stabilnosti in pravilnosti kristaline strukture), to pa med drugim določata molekulska masa in porazdelitev molekulskih mas polimera. V preteklosti je že bilo narejenih mnogo študij o odvisnosti mehanskih lastnosti polipropilenskih (PP) filamentov od molekulske mase in porazdelitve molekulskih mas polimera [1–8]. Znano je, da z naraščа-

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## 1 Introduction

*The mechanical properties of filaments are primarily dependent on their supermolecular structure (orientation, degree of crystallinity, crystallite size, the number of tie molecules, stability and perfection of crystalline structure), which is determined by the molecular weight and molecular weight distribution of the polymer. In the past, many studies have focused on the dependence of the mechanical properties of melt-spun polypropylene (PP) filaments upon the molecular weight and molecular weight distribution of the polymer [1–8]. It is well known that the tensile strength and elastic modulus of filaments increase as the molecular weight of the polymer increases. Lu and Spruiell [4] have found that, for PP polymers of different molecular weights, a critical take-up velocity exists, below which the orientation of the macromolecules grows rapidly, whereas above it, the orientation varies slowly. They concluded that this take-up velocity is largely determined by the molecular weight of the polymer, upon which the viscosity is dependent. The viscosity determines the spinline stress of the extruded polymer, which, in turn, influences the structure of the melt-spun filaments. Minoshima [9] investigated the impact of the molecular weight distribution on the orientation of macromolecules in melt-spun filaments and found that the degree of orientation is higher in polymers with narrower distributions. The apparent spinline viscosities of filaments spun at moderate and high inline draw-down values are higher, resulting in higher stress on the solidified filaments and, consequently, higher levels of orientation. Research [10–12] has shown that the degree of crystallinity and the speed of nuclei formation are increased by lowering the molecular weight of the polymer and that the crystallization speed increases for broader molecular weight distributions. Misra and colleagues [8] found that the degree of crystallinity increases with the take-up velocity, particularly in polymers with narrower molecular weight distributions, whilst polymers with broad distributions reached high levels of crystallinity, even at low speeds. On the basis of measurements of PP filaments spun at take-up velocities of up to 7000 m/min, Shimi-*

njem molekulske mase polimera trdnost in modul elastičnosti filamentov naraščata. Lu in Spruiell [4] sta ugotovila, da za vsak PP z določeno molekulsko maso obstaja kritična navijalna hitrost, pod katero orientacija makromolekul hitro narašča, nad njo pa se le počasi spreminja. Zaključila sta, da je ta hitrost določena predvsem z utežno molekulsko maso polimera, od katere sta odvisni viskoznost in napetost oblikovanja filamentov, le-ta pa določa nadmolekulsko strukturo talilno oblikovanih filamentov. Minoshima [9], ki je raziskal vpliv porazdelitve molekulskih mas PP-polimera na orientacijo makromolekul v talilno oblikovanih filamentih, je ugotovil, da je orientacija večja pri polimerih z ozko porazdelitvijo. Navidezna viskoznost pri zmernih in velikih hitrostih razvlečenja je pri teh polimerih višja, kar se kaže kot večja napetost strjujočih se filamentov in s tem višja stopnja orientacije. Raziskave [10–12] so pokazale, da stopnja kristalinosti in hitrost tvorbe kristalizacijskih jeder naraščata z nižanjem molekulske mase polimera ter da hitrost kristalizacije narašča s širitvijo porazdelitve molekulskih mas. Misra s sodelavci [8] je ugotovil, da stopnja kristalinosti narašča z navijalno hitrostjo, in to predvsem pri polimerih z ozko in srednje široko porazdelitvijo molekulskih mas, medtem ko polimeri s široko porazdelitvijo molekulskih mas dosežejo visoko stopnjo kristalinosti že pri nizkih hitrostih. Shimizu in sodelavci [13] so na podlagi meritev dvolomnosti in modula elastičnosti PP-filamentov, oblikovanih z navijalnimi hitrostmi do 7000 m/min, ugotovili, da pri navijalni hitrosti 3000 m/min dvolomnost in modul začneta strmeje naraščati. Na krivuljah napetost/raztezek je ta navijalna hitrost meja, pri kateri ni več razvidna točka polzišča.

Molekulska masa in porazdelitev molekulskih mas polimernih molekul v talilno oblikovanih PP-filamentih vplivata tudi na raztezност teh filamentov pri nadaljnjem razteznem preoblikovanju. Raztezност filamentov narašča z molekulsko maso in doseže maksimum pri $M_v$ je 67.000–78.000, nakar se zmanjša [11]. Z molekulsko maso polimera narašča tudi optimalna temperatura preoblikovanja filamentov, medtem ko ima porazdelitev molekulskih mas manjši vpliv na temperaturno odvisnost raztezности. Raztezност filamentov s široko porazdelitvijo molekulskih mas je večja kot raztezност filamentov z ozko porazdelitvijo molekulskih mas pri temperaturah preoblikovanja, nižjih od 80 °C, pri višjih temperaturah pa je vpliv porazdelitve molekulskih mas minimalen. Andreassen in sodelavci [14] so raziskali vpliv, ki ga ima porazdelitev molekulskih mas izhodnega polimera na natezne lastnosti polipropilenskih filamentov. Ugotovili so, da polimeri z različno porazdelitvijo molekulskih mas dajejo filamente z različnimi mehanskimi lastnostmi, odvisno od velikosti uporabljenega razteznega razmerja v procesu razteznega preoblikovanja. Najvišji modul elastičnosti in najvišjo natezno trdnost so pri manjših razteznih razmerjih dosegli filamenti, oblikovani iz polimera s široko porazdelitvijo molekulskih mas. Z večanjem razteznega razmerja sta se modul elastičnosti in trdnost večala, pretržni raztezek pa manjšal za obe skupini

*zu and colleagues [13] have found that the birefringence and elastic modulus began to rise more steeply at a take-up speed of 3000 m/min. As observed from the stress / extension data, this take-up velocity is the limit, after which yield points are no longer clearly visible.*

*The molecular weight and molecular weight distribution of polymer molecules in melt-spun PP filaments also affect their stretchability in the drawing process. The drawability of filaments increases with molecular weight, reaches a maximum at Mv 67000–78000 and then reduces [11]. The optimum drawing temperature increases with the molecular weight of the polymer, while the molecular weight distribution has less impact on the temperature dependence of drawing. The drawability of filaments with broad molecular weight distributions is better than the drawability of filaments with narrow molecular weight distributions at temperatures lower than 80 °C, whereas at higher temperatures, the impact of the molecular weight distribution on the drawability is minimal. Andreassen and colleagues [14] have studied the impact of the molecular weight distribution of polymers on the tensile properties of drawn polypropylene filaments. It was found that polymers with different molecular weight distributions resulted in filaments with different mechanical properties, depending on the draw ratio applied during the drawing process. The maximum elastic modulus and tensile strength at smaller draw ratios were achieved in filaments made from a polymer with a broad molecular weight distribution. By increasing the draw ratio, the elastic modulus and tensile strength increased, whereas the breaking extension decreased for filaments formed from polymers of both narrow and broad molecular weight distributions. Since the elastic modulus grew more quickly in filaments formed from a polymer with a narrow distribution, at higher draw rates, these filaments exceeded the elastic modulus and tensile strength of filaments made from a polymer with a broad molecular weight distribution.*

*As part of a wider research attempt to produce high modulus polypropylene fibers, the impact of molecular weight and molecular weight distribution on the tensile properties of filaments was explored. PP filaments were made with a*

vzorcev, to je za filamente, oblikovane iz polimera z ozko porazdelitvijo molekulskih mas, in za filamente, oblikovane iz polimera s široko porazdelitvijo molekulskih mas. Ker pa je modul elastičnosti hitreje naraščal pri filamentih, oblikovanih iz polimera z ozko porazdelitvijo, so pri visokih razteznih razmerjih ti filamenti presegli modul elastičnosti in trdnost filamentov, oblikovanih iz polimera s široko porazdelitvijo molekulskih mas.

Del širše raziskave, katere cilj je bil izdelati visokomodulna polipropilenska vlakna, je bilo raziskovanje vpliva molekulske mase in porazdelitve molekulskih mas izhodnega polimera na natezne lastnosti filamentov. PP-filamenti so bili izdelani po ločenem (diskontinuirnem) postopku, v katerem sta združeni fazi talilnega oblikovanja in tristopenjskega raztezanja potekali na laboratorijski predilno-raztezalni napravi, dodatno raztezanje v naslednji ločeni fazi pa na laboratorijski raztezalni napravi. Karakteristike obeh naprav in postopek izdelave filamentov so bili podrobneje opisani že drugje [15, 16]

## 2 Eksperimentalni del

### *2.1 Surovine in izdelava filamentov*

Za oblikovanje filamentov sta bila uporabljena Ziegler-Natta polipropilenska polimera: polimer z nizkim indeksom tečenja taline (MFI = 2 g/10 min), to je polimer z razmeroma visoko povprečno molekulsko maso in široko porazdelitvijo molekulskih mas ($\overline{M}_W$ = 280.000 g/mol, $\overline{M}_W/\overline{M}_n$ = 5), ter polimer z višjo vrednostjo MFI (18 g/10 min), to je polimer z nižjo povprečno molekulsko maso in ozko porazdelitvijo molekulskih mas ($\overline{M}_W$ = 210.000 g/mol, $\overline{M}_W/\overline{M}_n$ = 3,2), tako imenovani CR-polimer. Iz obeh polimerov so bile pripravljene mešanice v naslednjih razmerjih: 90/10, 80/20, 70/30, 60/40, 50/50, 40/60, 30/70, 20/80 in 10/90 utežnih odstotkov polimera z višjo molekulsko maso/CR-polimera z nižjo molekulsko maso.

Oblikovanje filamentov je potekalo na predilno-raztezalni napravi podjetja Extrusion Systems Ltd., in sicer s predilno hitrostjo 30,1 m/min pri vzorcih, oblikovanih pri višjem masnem pretoku, in 21,7 m/min pri vzorcih, oblikovanih pri nižjem masnem pretoku, in to skozi predilno šobo z desetimi luknjicami premera 0,35 mm. Po hitri ohladitvi ekstrudiranih filamentov z bočnim vpihovanjem zraka, ohlajenega na 3 °C, so bili filamenti neprekinjeno (kontinuirno) raztezani do optimalnega razteznega razmerja pri temperaturi 50 °C. Neprekinjenemu tristopenjskemu raztezanju na predilno-raztezalni napravi je sledilo še dodatno, ločeno (diskontinuirno) vroče raztezno preoblikovanje na laboratorijski raztezalni napravi podjetja Zimmer. Ločeno raztezanje filamentov pri dveh različnih temperaturah, 130 °C in 145 °C, je potekalo do mejnih razteznih razmerij z uporabo grelne plošče v razteznem polju. Pogoji, pri katerih je potekala izdelava PP-filamentov, so bili podrobneje opisani drugje [15, 17].

*discontinuous drawing process, where the combination of melt spinning and three-stage drawing was performed on a laboratory spin-draw device, with further additional drawing in a separate phase on a laboratory draw device. The characteristics of both devices and the manufacturing process conditions of the filaments have been described in detail previously [15, 16].*

## 2 Experimental

### 2.1 Materials and the manufacturing of filaments

*Ziegler-Natta polypropylene polymer: polymer with a low melt flow index (MFI = 2 g/10 min), this is a polymer with a relatively high average molecular weight and a broad molecular weight distribution ($\overline{M}_W$ = 280,000 g/mol, $\overline{M}_W/\overline{M}_n$ = 5); and polymer with a high MFI value (18 g/10 min), this is a polymer with a lower average molecular weight and a narrow molecular*

PP-filamenti so glede na prevladujoči delež izhodnega polimera obravnavani v dveh skupinah. V *skupini N* so tako zajeti filamenti, oblikovani iz polimera z višjo molekulsko maso in širšo porazdelitvijo molekulskih mas (Hostalen PPN 1060F) ter iz mešanic, v katerih prevladuje delež tega polimera: *100N, 90N, 80N, 70N, 60N*, ko gre za neprekinjeno, delno raztezane filamente (prva serija vzorcev), ter *100N/130, 90N/130, 80N/130, 70N/130, 60N/130 in 100N/145, 90N/145, 80N/145, 70N/145, 60N/145*, ko gre za dodatno, ločeno raztezane filamente pri dveh temperaturah, tj. 130 °C in 145 °C (druga serija vzorcev). Pri tem številka v oznaki vzorca pomeni odstotni delež tega polimera v mešanici. *Skupino U* sestavljajo filamenti, oblikovani iz polimera z nižjo molekulsko maso in z ozko porazdelitvijo molekulskih mas (Hostalen PPU 1780F2) ter iz mešanic polimerov s prevladujočim deležem tega polimera. Neprekinjeno, delno raztezani vzorci iz te skupine imajo oznake *100U, 90U, 80U, 70U, 60U, 50U* (prva serija vzorcev), dodatno, ločeno raztezani pri temperaturi 130 °C *100U/130, 90U/130, 80U/130, 70U/130, 60U/130, 50U/130* in pri temperaturi 145 °C *100U/145, 90U/145, 80U/145, 70U/145, 60U/145* in *50U/145* (druga serija vzorcev). Oznake vzorcev glede na izhodni polimer in postopek izdelave filamentov so podane v preglednici 1.

*Table 1: Samples grouped by polymer and filament manufacturing process*

| | Samples and (percent of prevailing part of polymer in polymer blend) | |
|---|---|---|
| Polymer | PPN 1060F | PPU 1780F2 |
| Group 1: | | |
| Continuously partly drawn filaments on spin-draw device, melt spun from both pure polymers and their blends | 100N (100% PPN)<br>90N (90% PPN)<br>80N (80% PPN)<br>70N (70% PPN)<br>60N (60% PPN) | 100U (100% PPU)<br>90U (90% PPU)<br>80U (80% PPU)<br>70U (70% PPU)<br>60U (60% PPU)<br>50U (50% PPU) |
| Group 2: | | |
| Additionally discontinuously drawn filaments (group 1) on drawing device at the temperature of 130°C | 100N/130<br>90N/130<br>80N/130<br>70N/130<br>60N/130 | 100U/130<br>90U/130<br>80U130<br>70U/130<br>60U/130<br>50U/130 |
| Additionally discontinuously drawn filaments (group 1) on drawing device at the temperature of 145°C | 100N/145<br>90N/145<br>80N/145<br>70N/145<br>60N/145 | 100U/145<br>90U/145<br>80U145<br>70U/145<br>60U/145<br>50U/145 |

*weight distribution ($\overline{M}_W$ = 210,000 g/mol, $\overline{M}_W/\overline{M}_n$ = 3.2), the so-called CR-polymer. These were used for the manufacturing of filaments. From both polymers, mixtures in the following proportions were prepared: 90/10, 80/20, 70/30, 60/40, 50/50, 40/60, 30/70, 20/80 and 10/90 weight percent higher molecular weight polymer / CR-polymer of lower molecular weight.*

*The manufacturing of filaments was performed on a spin-draw device (Extrusion Systems Ltd.) with a spinning speed of 30.1 m/min for samples made with the higher mass flow and 21.7 m/min for samples made with the lower mass flow, through a spinneret with ten holes, each with a diameter of 0.35 mm. After rapidly cooling the extruded filaments via crossflow air quenching at a temperature of 6°C, a continuous drawing of filaments at the optimum drawing rate at a temperature of 50°C was performed. The three-stage drawn filaments, drawn continuously at a moderate rate on the spin-draw device, were additionally hot drawn on a laboratory draw device (Zimmer). Separate drawing of filaments at two different temperatures, 130°C and 145°C, was performed with a hot plate to the limiting draw ratio. The conditions of the manufacturing process have been detailed elsewhere [15, 17].*

*The PP filaments, according to the dominant percentage of the polymer present, are divided into two groups. Group N is composed of the filaments made from a polymer with a higher molecular weight and broader molecular weight distribution (Hostalen PPN 1060F) and from polymer blends with a prevailing share of this polymer (100N, 90N, 80N, 70N, 60N, for continuously, moderately drawn filaments (first set of samples) and 100N/130, 90N/130, 80N/130, 70N/130, 60N/130 and 100N/145, 90N/145, 80N/145, 70N/145, 60N/145 for additionally, discontinuously drawn filaments at two temperatures, 130°C and 145°C (second set of samples)). In these samples, the number designates the percentage of this polymer in the mixture. The second group (group U) consists of filaments made from the lower molecular weight polymer with a narrow molecular weight distribution (Hostalen PPU 1780F2) and from polymer blends with a prevailing share of this polymer. Continuously, moderately drawn filaments from this group have the following names: 100U,*

### 2.2 Metode preiskav

Natezne lastnosti PP filamentov so bile določene v nateznem poskusu z merjenjem pretržne sile in pretržnega raztezka ter beleženjem naraščajoče obremenitve v odvisnosti od naraščajočega podaljška. Meritve so se izvajale na dinamometru Instron 6022, in sicer pri vpeti dolžini 25 cm, s hitrostjo raztezanja 5,5 mm/s za neprekinjeno, delno raztezane filamente (prva serija vzorcev) in 1,6 mm/s za dodatno, ločeno raztezane filamente (druga serija vzorcev), tako da je do pretrga prišlo v približno 20 sekundah. Določene so bile viskoelastične in pretržne lastnosti: polzišče, modul elastičnosti, pretržni raztezek, specifična pretržna napetost, specifično pretržno delo.

## 3 Rezultati in razprava

### 3.1 Pretržne natezne lastnosti

Mehanske lastnosti filamentov so najpomembnejše tehnične lastnosti, saj vplivajo na obnašanje filamentov pri predelavi in uporabi. Med mehanskimi lastnostmi so zelo pomembne natezne lastnosti, ki so posledica odziva na zunanje sile in deformacije vzdolž vlakenske osi. Pri delovanju natezne sile se v filamentih pojavi napetost – kot upor, s katerim se filamenti upirajo delovanju zunanje sile. Zaradi primerljivosti se kot merilo za trdnost filamentov uporablja specifična pretržna napetost, ki je podana z razmerjem med pretržno silo in dolžinsko maso. Če se pri izračunu specifične pretržne napetosti upošteva znižanje dolžinske mase filamentov zaradi raztezanja, se dobi korigirana, to je dejanska specifična pretržna napetost ($\sigma_{br,ac}$), ki je definirana kot razmerje med pretržno silo ($F_{br}$) in dolžinsko maso filamentov v trenutku pretrga ($T_{t,br}$) (en. 1).

$$\sigma_{br,ac} = \frac{F_{br}}{T_{t,br}} \quad ; \quad T_{t,br} = \frac{T_t \cdot 100}{100 + \varepsilon_{br}} \qquad \text{(en. 1)}$$

Vrednosti obeh pretržnih napetosti, skupaj s pretržnim raztezkom in pretržnim delom, so zbrane v preglednici 2.

Iz preglednice 2 je razvidno, da je razlika med pretržno napetostjo in dejansko pretržno napetostjo velika le pri manj raztezanih filamentih. Pri teh filamentih je dolžinska masa v trenutku pretrga bistveno manjša od izmerjene, zato deluje sila na manjši presek, kar poveča njeno napetost. Pri dodatno, do mejnih razteznih razmerij ločeno raztezanih filamentih se dolžinska masa pri nadaljnjem obremenjevanju z natezno silo le malo zmanjša, kar povzroči le majhno dejansko povečanje napetosti v filamentih.

Pretržni raztezek filamentov se z večanjem razteznega razmerja znižuje in je pri neprekinjeno, delno raztezanih filamentih med 50 in 110 %, pri dodatno, ločeno raztezanih filamentih pa med 9 in 20 %. Pretržno delo je določeno z velikostjo površine pod krivuljo

*Table 2: Breaking load ($F_{br}$), specific stress at break ($\sigma_{br}$), real specific stress at break ($\sigma_{br,ac}$), breaking extension ($\varepsilon_{br}$), specific work at break ($A_{sp,br}$), yield stress ($\sigma_y$) and elastic modulus ($E_0$) of continuously, moderately drawn and additionally, discontinuously drawn filaments.*

| SAMPLE | $F_{br}$ (N) | $\sigma_{br}$ (cN/dtex) | $\sigma_{br,ac}$ (cN/dtex) | $\varepsilon_{br}$ (%) | $A_{sp,br}$ (J/g) | $\sigma_y$ (cN/dtex) | $E_0$ (GPa) |
|---|---|---|---|---|---|---|---|
| 100N | 12.51 | 2.24 | 4.19 | 87.23 | 135.36 | 1.28 | 1.77 |
| 90N | 14.98 | 3.65 | 6.40 | 75.21 | 191.24 | 1.79 | 2.40 |
| 80N | 12.45 | 3.03 | 4.66 | 53.69 | 121.30 | 0.90 | 2.90 |
| 70N | 14.69 | 3.55 | 5.83 | 64.30 | 163.70 | 1.93 | 2.59 |
| 60N | 14.58 | 2.76 | 5.03 | 82.13 | 173.27 | 1.58 | 1.93 |
| 50U | 16.06 | 3.24 | 4.99 | 53.90 | 131.00 | 0.93 | 3.12 |
| 60U | 15.44 | 3.09 | 5.99 | 93.96 | 211.00 | 1.63 | 2.14 |
| 70U | 19.77 | 3.95 | 6.81 | 72.39 | 194.70 | 1.92 | 2.93 |
| 80U | 19.05 | 3.75 | 6.75 | 80.40 | 215.15 | 2.02 | 2.62 |
| 90U | 18.26 | 3.51 | 6.30 | 79.53 | 209.78 | 1.98 | 2.68 |
| 100U | 15.46 | 3.00 | 6.32 | 110.67 | 206.49 | 1.23 | 1.93 |
| 100N/130 | 17.53 | 5.81 | 6.95 | 19.68 | 75.42 | 0.95 | 7.15 |
| 90N/130 | 16.82 | 7.98 | 9.23 | 15.65 | 76.70 | 0.88 | 10.46 |
| 80N/130 | 17.20 | 7.18 | 8.42 | 17.25 | 75.34 | 0.82 | 9.43 |
| 70N/130 | 26.01 | 8.08 | 9.47 | 17.24 | 78.54 | 0.80 | 10.83 |
| 60N/130 | 19.86 | 7.48 | 8.77 | 17.20 | 75.34 | 0.80 | 9.46 |
| 50U/130 | 19.69 | 7.42 | 8.53 | 14.92 | 62.43 | 1.40 | 9.96 |
| 60U/130 | 18.38 | 7.76 | 9.05 | 16.62 | 68.12 | 0.90 | 10.60 |
| 70U/130 | 20.06 | 8.20 | 9.33 | 13.23 | 65.15 | 0.71 | 11.53 |
| 80U/130 | 18.52 | 7.71 | 8.64 | 12.06 | 55.74 | 0.59 | 11.56 |
| 90U/130 | 17.97 | 7.83 | 8.70 | 11.15 | 47.35 | 0.61 | 12.00 |
| 100U/130 | 15.76 | 7.77 | 8.78 | 12.95 | 51.16 | 0.65 | 12.29 |
| 100N/145 | 17.55 | 6.91 | 8.04 | 16.40 | 64.14 | 0.96 | 9.65 |
| 90N/145 | 15.61 | 8.01 | 9.16 | 14.41 | 63.29 | 0.75 | 11.82 |
| 80N/145 | 17.30 | 7.67 | 9.04 | 17.99 | 82.24 | 0.78 | 10.25 |
| 70N/145 | 16.32 | 8.13 | 9.35 | 14.95 | 66.36 | 0.71 | 11.26 |
| 60N/145 | 18.50 | 7.43 | 8.57 | 15.40 | 67.12 | 1.19 | 9.62 |
| 50U/145 | 17.75 | 7.38 | 8.29 | 12.38 | 51.05 | 0.80 | 12.51 |
| 60U/145 | 14.92 | 7.30 | 8.20 | 12.36 | 52.42 | 0.94 | 11.36 |
| 70U/145 | 17.33 | 7.82 | 8.68 | 10.94 | 50.97 | 0.76 | 13.12 |
| 80U/145 | 16.83 | 7.92 | 8.78 | 10.86 | 44.92 | 0.67 | 13.33 |
| 90U/145 | 15.47 | 7.86 | 8.57 | 9.00 | 44.70 | 0.74 | 14.80 |
| 100U/145 | 14.01 | 8.22 | 9.01 | 9.65 | 52.35 | 0.70 | 13.60 |

*90U, 80U, 70U, 60U, 50U (first set of samples), additionally, discontinuously drawn filaments at a temperature of 130°C: 100U/130, 90U/130, 80U/130, 70U/130, 60U/130, 50U/130 and at a temperature of 145°C: 100U/145, 90U/145, 80U/145, 70U/145, 60U/145 and 50U/145 (second set of samples).*

### 2.2 Testing methods

*The tensile properties of the PP filaments were established by a tensile test, by measuring the breaking load and breaking extension and recording the increasing load as a function of the increasing extension. Measurements were carried out on an Instron 6022 dynamometer, with an initial gauge length of 25 cm at a crosshead speed of 5.5 mm/s for the continuously, moderately drawn filaments (first set of samples) and at 1.6 mm/s for the additionally, discontinuously drawn filaments (second set of samples), so that the break occurred in about 20 seconds. The viscoelastic and breaking properties, i.e., the yield point, elastic modulus, stress at break, extension at break and specific work at break, were determined.*

## 3 Results and discussion

### 3.1 Tensile properties at break

*The mechanical properties of filaments are the most important technical features, influencing the behavior of filaments in both processing and use. Among the mechanical properties, the tensile properties, resulting from the response to external forces and deformation along the fiber axis, are very important. Under the action of tensile forces, stress occurs in the filaments as the filaments resist the external force. As a measure of the filament`s tenacity, the specific stress, given by the ratio between the breaking load and linear density, is used. If the thinning of the filament during stretching is taken into account in calculating the specific stress at break, the corrected or real specific stress at break ($\sigma_{br,ac}$), defined as the ratio between the breaking load ($F_{br}$) and the linear density of the filaments, determined at the moment of break ($T_{t,\,br}$) can be obtained (Eq. 1).*

*The values of both stresses at break, together with the breaking extension and work at break, are presented in Table 2.*

sila/podaljšek. Zaradi vpliva dolžinske mase in dolžine preskušanca na delo, potrebno za pretrg filamentov, je za primerjavo med vzorci primernejša uporaba specifičnega pretržnega dela ($A_{sp,br}$), ki se izračuna po enačbi 2:

$$A_{sp,br} = \frac{A_{br}}{T_t \cdot l_o} \qquad \text{(en. 2)}$$

kjer je $A_{br}$ pretržno delo, $T_t$ dolžinska masa in $l_0$ vpeta dolžina.

Iz preglednice 2 je razvidno, da je specifično pretržno delo višje pri neprekinjeno, delno raztezanih filamentih in znaša med 130 in 215 J/g, pri dodatno, ločeno raztezanih filamentih pa je med 45 in 80 J/g.

#### *3.1.1 Neprekinjeno, delno raztezani filamenti*

Specifična pretržna napetost filamentov doseže po neprekinjenem, delnem raztezanju vrednosti od 2,24 cN/dtex do 3,95 cN/dtex. Izračunane vrednosti dejanske specifične pretržne napetosti filamentov, pri kateri je upoštevano stanjšanje filamentov v času preskušanja, so precej višje in se gibljejo od 4,2 do 6,8 cN/dtex. Filamenti, oblikovani iz mešanic s prevladujočim deležem polimera z višjo molekulsko maso, imajo kljub enakemu razteznemu razmerju (λ = 20,2) različno pretržno napetost, nižjo pri večjem dodatku CR-polimera. Dodatek polimera z višjo molekulsko maso k CR-polimeru z nižjo molekulsko maso pa poveča specifično pretržno napetost filamentov (*vzorci 90U, 80U* in *70U*) na 3,95 cN/dtex – skladno z večanjem dodatka – v primerjavi s filamenti, oblikovanimi iz 100 % CR-polimera (*vzorec 100U*), čeprav so bili filamenti, oblikovani iz mešanic, raztezani pri nekoliko nižjem razteznem razmerju (λ = 15,3) kot vzorec 100U (λ = 15,6).

Tristopenjsko neprekinjeno raztezani filamenti so še močno raztezni. Največji pretržni raztezek ima *vzorec 100U*, in sicer 111 %. Dodatek polimera z višjo molekulsko maso CR-polimeru z nižjo molekulsko maso zniža razteznost, tako pretržni raztezek *vzorcev 90U* in *80U* znaša 80 %, *vzorec 70U* ima 72 % raztezek in *vzorec 50U* 54 %. Pri tem so bili vsi filamenti raztezani pri enakem strojnem (λ = 15,3) in zelo podobnem dejanskem razteznem razmerju ($\lambda_d$ = 14,2–14,5) kot *vzorec 100U* ($\lambda_d$ = 13,9). Tudi pri filamentih, oblikovanih iz surovine s prevladujočim deležem polimera z višjo molekulsko maso, je razviden vpliv izhodnega polimera: *vzorec 100N* ima kljub nižjemu razteznemu razmerju (λ = 13) v primerjavi z *vzorcem 100U* (λ = 15,6) nižji pretržni raztezek, filamenti, oblikovani iz mešanic (*vzorci 90N, 80N* in *70N*), pa imajo kljub enakemu razteznemu razmerju (λ = 20,2) zelo različne pretržne raztezke; *vzorec 90N* 75 %, *vzorec 80N* 54 % in *vzorec 70N* 64 % raztezek. Najnižji pretržni raztezek 54 % imata *vzorca 50U* in *80N*. Filamenti imajo enak pretržni raztezek, čeprav je bil *vzorec 50U* raztezan z razteznim razmerjem 15,3, *vzorec 80N* pa z 20,2, kar samo potrjuje, da ima izhodna surovina vpliv na nastanek razlik v nadmolekulski strukturi, le-ta pa pogojuje pretržne lastnosti filamentov.

*From Table 2, it can be seen that a difference between the specific stress at break and the actual specific stress at break occurs only in the moderately drawn filaments. In these filaments, the linear density at the moment of break is significantly lower than the measured linear density; the load is acting on a smaller cross section, which increases the tension in the filament. In the additionally, discontinuously drawn filaments, drawn to the limiting draw ratio, the linear density during stretching under a tensile load is only slightly reduced, resulting in only a small actual increase in tension in the filaments.*

*The breaking extension of the filaments decreases with increasing drawing ratio and was measured for continuously, moderately drawn filaments to be between 50 and 110% and, for additionally, discontinuously drawn filaments, between 9 and 20%. The work at break is determined by the size of the area under the load / extension curve. Due to the influence of the length and linear density of the sample on the work necessary to break the filaments, it is more appropriate to use the specific work at break, ($A_{sp,br}$), for comparison between the samples, which is calculated by Eq. 2, where $A_{br}$ is the work at break, $T_t$ is the linear density, and $l_0$ is the initial length of the filament.*

*From Table 2, it can be seen that the specific work at break is higher in the continuously, moderately drawn filaments and is between 130 and 215 J/g; whereas, in the additionally, discontinuously drawn filaments, the specific work at break is between 45 and 80 J/g.*

### *3.1.1 Continuously, moderately drawn filaments*

*The specific breaking stress of filaments after continuous moderate drawing reaches values between 2.24 cN/dtex and 3.95 cN/dtex. The calculated values of the actual specific stress at break, which take into account the thinning of the filaments during tensile testing, are much higher and range from 4.2 to 6.8 cN/dtex. The filaments made from the polymer blends with a prevailing share of the higher molecular weight polymer have different values of specific stress at break, despite having the same draw ratio (λ = 20.2); the values are lower for higher additions*

Pretržno delo in specifično pretržno delo sta pri neprekinjeno, delno raztezanih filamentih visoki, kar pomeni, da so filamenti precej žilavi. Višje vrednosti imajo filamenti, oblikovani iz surovine s prevladujočim deležem CR-polimera z nižjo molekulsko maso.

### *3.1.2 Dodatno, ločeno raztezani filamenti*

Dodatno, ločeno raztezno preoblikovanje pri visokih temperaturah specifično pretržno napetost filamentov še močno poveča. Pri določenem razteznem razmerju (λ nad 32) dosežejo filamenti trdnosti nad 7 cN/dtex, ki se z nadaljnjim večanjem razteznega razmerja le zmerno povečujejo – do 8,2 cN/dtex (sl. 1). Vrednosti dejanske specifične pretržne napetosti dodatno raztezanih filamentov so še za 9 do 14 % višje in se gibljejo med 8 in 9,5 cN/dtex. Najvišjo specifično pretržno napetost ($\sigma_{br}$ = 8,22 cN/dtex) imajo filamenti, oblikovani iz CR-polimera z nižjo molekulsko maso, dodatno raztezani pri 145 °C (*vzorec100U/145*). Najvišjo dejansko specifično pretržno napetost ($\sigma_{br,ac}$ nad 9,4 cN/dtex) pa imajo dodatno, ločeno raztezani filamenti, oblikovani iz mešanice 70/30 utežnih odstotkov polimera z višjo molekulsko maso/CR-polimera z nižjo molekulsko maso (*vzorca 70N/130* in *70N/145*).

Pretržni raztezki dodatno raztezanih filamentov so nizki, pri filamentih, raztezanih pri temperaturi 130 °C, so v območju od 11 do 20 % in pri filamentih, raztezanih pri temperaturi 145 °C, od 9 do 18 %. Filamenti, oblikovani iz CR-polimera z nižjo molekulsko maso in iz mešanic polimerov s prevladujočim deležem tega polimera, imajo bistveno nižje pretržne raztezke ($\varepsilon_{br}$ med 9 in 15 %) kot filamenti, oblikovani iz polimera z višjo molekulsko maso in mešanic s prevladujočim deležem tega polimera ($\varepsilon_{br}$ med 14,4 in 19,7 %), kljub temu da niso bili vsi raztezani z višjimi razteznimi razmerji. S tem je vpliv izhodnega polimera razviden tudi po dodatnem raztezanju filamentov do mejnih razteznih razmerij. Najnižji pretržni raztezek ($\varepsilon_{br}$ = 9 %) imajo filamenti, oblikovani iz polimerne mešanice, sestavljene iz 90 % CR-polimera z nižjo molekulsko maso in 10 % polimera z višjo molekulsko maso ter široko porazdelitvijo molekulskih mas (*vzorec 90U/145*).

Z dodatnim raztezanjem pri visokih temperaturah se vrednosti pretržnega in specifičnega pretržnega dela močno znižajo, še posebej pri filamentih iz *skupine vzorcev U*, ki imajo po dodatnem raztezanju nižje pretržno in specifično pretržno delo od *vzorcev* iz *skupine N*. Filamenti, raztezani pri 145 °C, oblikovani iz mešanice 90/10 CR-polimera z nižjo molekulsko maso/polimera z višjo molekulsko maso (*vzorec 90U/145*), so najmanj raztezni in žilavi med vsemi vzorci ($A_{sp,br}$ = 44,7 J/g).

## *3.2 Krivulja specifična napetost/raztezek*

Oblika krivulje specifična napetost/raztezek je v glavnem odvisna od molekulske strukture materiala. Modul elastičnosti, ki je podan z razmerjem specifične napetosti in relativnega podaljška oz. s tangensom kota med začetnim delom krivulje specifična napetost/raztezek in absciso, je pomembna karakteristika materiala.

*of the CR-polymer. The addition of the higher molecular weight polymer to the lower molecular weight CR-polymer increases the specific stress at break of the filaments (samples 90U, 80U and 70U) to 3.95 cN/dtex. This is consistent with the increase in added polymer, compared with the filaments made from 100% CR-polymer (sample 100U), although the filaments made from the polymer blends were drawn at a lower draw ratio (λ = 15.3) than sample 100U (λ = 15.6)*

*The three-stage continuously drawn filaments are still very extensible. The highest breaking extension, 111%, was observed for sample 100U. The addition of the higher molecular weight polymer to the lower molecular weight CR-polymer lowers the stretchability; the breaking extension of samples 90U and 80U was 80%, sample 70U had a 72% extension, and sample 50U had an extension of 54%. All the samples were drawn at the same draw ratio (λ = 15.3) as sample 100U ($\lambda_{ac}$ = 13.9), with very similar actual draw ratios (determined from the ratio of linear densities) ($\lambda_{ac}$= 14.2–14.5). In addition, in the filaments made from the polymer blends with a prevailing share of higher molecular weight polymer, the impact of the polymer is seen. Despite having a lower draw ratio (λ = 13) than sample 100U (λ = 15.6), sample 100N exhibited a lower extension at break, the filaments made from the polymer blends (samples 90N, 70N and 80N) have very different extensions at break at the same draw ratio (λ = 20.2): sample 90N, 75%; sample 80N, 54%; and sample 70N, 64%. The lowest extension of 54% was observed for samples 50U and 80N. The filaments had the same breaking extension, although sample 50U was drawn with a draw ratio of 15.3, while sample 80N was drawn at a ratio of 20.2, which confirms that the raw material (polymer) has an impact on the formation of the supramolecular structure, which, in turn, determines the breaking properties of the filament.*

*Both the work at break and specific work at break of continuously, moderately drawn filaments are high, which indicates that the filaments are not very resistant. Higher values were measured for the filaments made from polymer blends with a prevailing share of the lower molecular weight CR-polymer.*

Pove, kakšen je začetni upor filamentov proti raztezanju. Vrednosti modula, podane v *preglednici 2*, so bile določene na začetnem premo sorazmernem območju iz maksimuma prvega odvoda krivulje [18].

Druga pomembna karakteristika, ki jo lahko odčitamo iz krivulje specifična napetost/raztezek, je polzišče. Polzišče je točka na krivulji specifična napetost/raztezek za proporcionalnim elastičnim območjem, na kateri pride do prve trajne deformacije filamentov. Definirano je s pripadajočima napetostjo in raztezkom. Vrednosti specifične napetosti v polzišču so podane v *preglednici 2*. Napetost v polzišču je višja pri kontinuirno raztezanih filamentih, pri katerih je material še bolj raztezen. Polzišče nastane pri raztezkih med 7 in 8 %. Pri močno raztezanih filamentih pa se trajna deformacija začne že pri zelo nizkih raztezkih, in sicer pri 0,5 do 1,5 %.

### *3.2.1 Neprekinjeno, delno raztezani filamenti*

Krivulje neprekinjeno, delno raztezanih PP-filamentov kažejo pri obremenjevanju v nateznem poskusu začetno elastično obnašanje, izrazito polzišče in sledeče viskoelastično, predvsem plastično obnašanje s počasi naraščajočo napetostjo do pretrga. Prvi odvod, ki predstavlja potek modula, najprej strmo pade, po minimumu pa počasi narašča do pretrga. Na *sliki 1* so prikazane krivulje neprekinjeno, delno raztezanih filamentov obeh skupin. Oblika krivulj je na celotnem območju deformacij in napetosti podobna pri vseh vzorcih, še posebej pri filamentih, oblikovanih iz mešanic obeh polimerov. Podoben potek krivulj pomeni, da se bodo ti filamenti na delovanje natezne sile odzvali podobno, ne glede na mešanico, iz katere so bili izdelani. Filamenti, oblikovani iz mešanic, se razlikujejo od filamentov, oblikovanih iz 100 % izhodnih polimerov, po tem, da dosežejo višje vrednosti specifične napetosti pri enakih raztezkih v primerjavi z *vzorcema 100U* in *100N*. Slednja se razlikujeta tudi po tem, da so filamenti, oblikovani iz CR-polimera z nižjo molekulsko maso (*vzorec 100U*), bolj raztezni kot drugi vzorci in se pretrgajo pri bistveno višjem raztezku, filamenti, oblikovani iz polimera z višjo molekulsko maso (*vzorec 100N*), pa ne

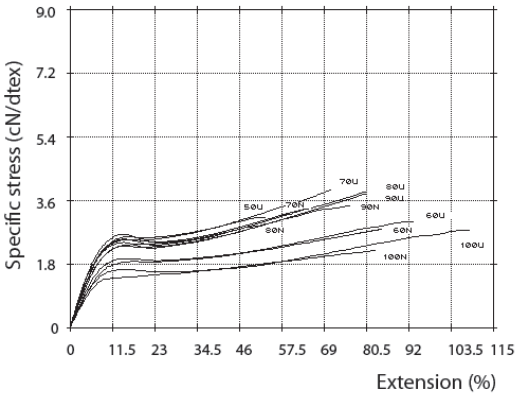


*Figure 1: Specific stress/extension curves of continuously, moderately drawn filaments.*

### *3.1.2 Additionally, discontinuously drawn filaments*

*Additional, separate drawing at high temperature strongly increases the specific stress at break of filaments. At a specific draw ratio (λ over 32), the filaments withstood stress values greater than 7 cN/dtex; by further increasing the draw ratio, only a moderate increase in the specific stress, up to 8.2 cN/dtex, was achieved (Fig. 1). The values of the real specific stress at break of the additionally, discontinuously drawn filaments are between 8 and 9.5 cN/dtex, and are 9 to 14% higher than values of the specific stress. The highest specific stress at break ($\sigma_{br}$ = 8.22 cN/dtex) was observed in the filaments made from the lower molecular weight CR-polymer, additionally drawn at 145°C (sample 100U/145). The maximum real specific stress at break ($\sigma_{br,ac}$ over 9.4 cN/dtex) was measured in the additionally, discontinuously drawn filaments made from a blend of 70/30 weight percent of higher molecular weight polymer / lower molecular weight CR-polymer (samples 70N/130 and 70N/145).*

*The breaking extensions of the additionally, discontinuously drawn filaments are low; the filaments drawn at a temperature of 130°C range from 11 to 20%, and the filaments drawn at a temperature of 145°C range from 9 to 18%. Filaments made from the lower molecular weight CR-polymer and polymer blends with a prevailing share of this polymer have a significantly lower breaking extension ($\varepsilon_{br}$ between 9 and 15%) than filaments made from the higher molecular weight polymer and polymer blends with a prevailing share of the latter ($\varepsilon_{br}$ between 14.4 and 19.7%), despite the fact that not all were drawn at a higher draw ratio. This implies that the influence of the polymer is also seen in additionally drawn filaments drawn at the limiting draw ratio. The lowest extension ($\varepsilon_{br}$ = 9%) was observed in filaments made from the polymer blend consisting of 90% of the lower molecular weight CR-polymer and 10% of the higher molecular weight polymer with a broad molecular weight distribution (sample 90U/145).*

*As additional drawing is carried out at high temperatures, the values of the work at break and specific work at break are significant reduced, particularly those of the samples from*

kažejo tako izrazitega padca napetosti za točko polzišča in obenem izkazujejo manjše naraščanje napetosti do pretrga od drugih filamentov. Izstopajo tudi filamenti, izdelani s 40 % deležem drugega polimera (*vzorca 60N* in *60U*). Krivulji teh dveh vzorcev ležita med krivuljama *vzorcev 100U* in *100N* ter krivuljami vzorcev, oblikovanih iz drugih polimernih mešanic.

Moduli elastičnosti neprekinjeno, delno raztezanih filamentov so nizki, njihove vrednosti so od 1,77 do 3,12 GPa. Korelacija z raztezni razmerjem, določena po fazah izdelave, je nizka, ker pri filamentih, oblikovanih iz mešanic polimerov, dodatek polimera z višjo molekulsko maso k CR-polimeru z nižjo molekulsko maso poveča modul elastičnosti v primerjavi z *vzorcem 100U* z 1,93 na 3,12 GPa, in to kljub nekoliko manjšemu raztezni razmerju. Prav tako večji utežni dodatek CR-polimera k polimeru z višjo molekulsko maso poveča modul elastičnosti, *vzorec 80N* ima tako večji modul ($E_o$ = 2,9 GPa) v primerjavi z *vzorcem 90N* ($E_o$ = 2,4 GPa), četudi sta bila oba vzorca izdelana z enakim razteznim razmerjem.

Specifična napetost v polzišču z vrednostmi od 0,9 do 2 cN/dtex je visoka, saj znaša okoli 50 % specifične pretržne napetosti. Pri vseh neprekinjeno, delno raztezanih filamentih se za polziščem odpor proti raztezanju močno zmanjša. Takoj za polziščem se filamenti deformirajo brez povečanja napetosti. Pri večini vzorcev pride celo do znižanja napetosti z deformacijo in modul v prvi obračalni točki ($E_1$), ki predstavlja najnižjo vrednost modula v področju za polziščem, zato doseže negativno vrednost. Pri nadaljnjem obremenjevanju je prirastek raztezka veliko večji kot prirastek napetosti in po obliki krivulje je razvidno, da je večina tega raztezka nepovratna. Bistvene utrditve materiala med potekom nateznega preskusa ni zaslediti, kar potrjuje tudi odsotnost maksimuma prvega odvoda, ki predstavlja modul v drugi obračalni točki ($E_2$). Nizke vrednosti modula elastičnosti in modula v prvi obračalni točki ($E_1$) ter celotna oblika krivulje kažejo, da se filamenti slabo upirajo deformiranju pod vplivom natezne sile in so še močno raztezni.

### *3.2.2 Dodatno, ločeno raztezani filamenti*

Potek krivulj dodatno, ločeno raztezanih filamentov je tipičen za močno raztezana vlakna s kratkim začetnim elastičnim področjem, brez jasno izraženega polzišča in prehoda v viskoelastično obnašanje, z enakomernim naraščanjem specifične napetosti in raztezka vse do pretrga. Prvi odvod krivulje se najprej strmo zniža, potem pa se počasi konstantno znižuje do točke pretrga.

Filamenti, raztezani pri temperaturi 130 °C (*sl. 2*), imajo podobno obliko in potek krivulj kot filamenti, raztezno preoblikovani pri temperaturi 145 °C (*sl. 3*). Slednji, ki so bili raztezani z večjim razteznim razmerjem, se bolj upirajo delovanju natezne sile in so manj raztezni kot filamenti, oblikovani iz enake surovine, dodatno raztezani pri nižji temperaturi in z nižjim razteznim razmerjem. Krivulje filamentov, oblikovanih iz polimera z višjo molekulsko maso (*vzorci skupine N*), se med seboj razlikujejo bolj, kot se

*group U. The samples from group U have lower values of work at break and specific work at break than the samples from group N. The filaments drawn at a temperature of 145°C made from the polymer blend of 90/10 lower molecular weight CR-polymer / higher molecular weight polymer (sample 90U/145) are the least extensible and of low toughness of all the samples ($A_{sp,br}$ = 44.7 J/g).*

### 3.2 Specific stress/extension curves

*The shape of a specific load/extension curve is largely dependent on the molecular structure of the material. The elastic modulus, which is given by the ratio of the specific stress and the relative extension, i.e., by the tangent of the angle between the initial region of the specific stress/extension curve and the abscissae, is an important characteristic of a material. It determines the initial resistance of the filament to stretching. The elastic modulus values given in Table 2 were determined from the initial region of the curve, as the maximum of the first derivative of the curve [18].*

*Another important characteristic that can be determined from the specific stress/extension curve is the yield point. The yield point is a point on the specific stress/extension curve that is observed beyond the proportional elastic region, where permanent deformation of the filament is initiated. It is defined by the stress and extension. The yield stress values are given in Table 2. The yield stress is higher in the continuously, moderately drawn filaments, where the material is still very extensible. The onset of yield occurs at an extension of 7–8%. In the additionally drawn filaments that are drawn to the limit, the permanent deformation begins at low extensions of 0.5 to 1.5%.*

#### *3.2.1 Continuously, moderately drawn filaments*

*The curves of the continuously, moderately drawn PP filaments show a marked yield point in the initial region of elastic behavior, followed by viscoelastic, particularly plastic, behavior, with the stress slowly growing until the break point. The first derivative, which represents the modulus, first falls sharply and then, after reaching a minimum, slowly grows until*

med seboj razlikujejo filamenti, oblikovani iz surovine s prevladujočim deležem CR-polimera z nižjo molekulsko maso (*vzorci skupine U*). Krivulje so pri slednjih strmejše in krajše kot krivulje prvih, čeprav so bili nekateri filamenti iz *vzorcev skupine U* raztezani z nižjimi razteznimi razmerji kot filamenti iz vzorcev skupine *N*. Pri obeh temperaturah raztezanja so krivulje filamentov, oblikovanih s 100 %, 90 %, 80 % in 70 % deležem CR-polimera z nižjo molekulsko maso, najstrmejše in imajo podoben potek. Tej skupini sledi druga, ki jo sestavljajo filamenti, oblikovani iz 60/40, 50/50, 30/70 in 10/90 CR-polimera z nižjo molekulsko maso/polimera z višjo molekulsko maso, ki dosežejo podobno visoke pretržne napetosti pri višjih raztezkih. Potek krivulj je pri teh filamentih zelo podoben, kar pomeni, da se bodo podobno odzvali na delovanje natezne sile, čeprav se močno razlikujejo v razteznih razmerjih, s katerimi so bili izdelani.

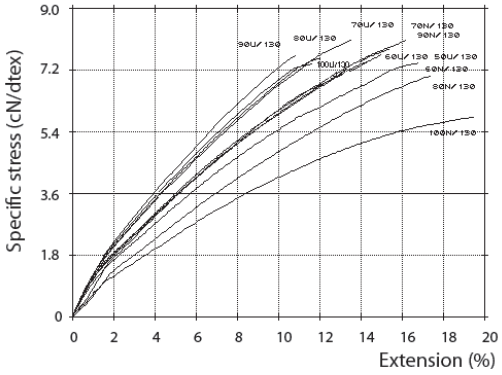


*Figure 2: Specific stress/extension curves of additionally, discontinuously drawn filaments, drawn at a temperature of 130 °C.*

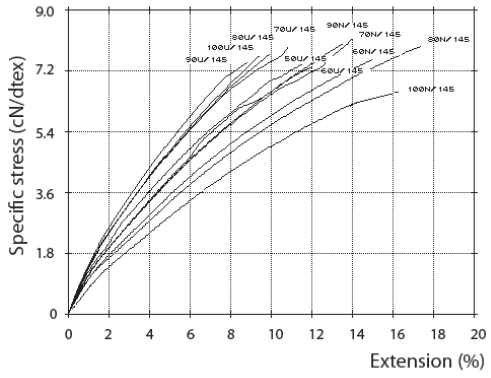


*Figure 3: Specific stress/extension curves of additionally discontinuously drawn filaments, drawn at the temperature of 145 °C.*

Dodatno raztezanje pri visokih temperaturah, ki bistveno utrdi filamente in zmanjša njihovo razteznost, močno poveča tudi modul elastičnosti. PP-filamenti, raztezani pri temperaturi 130 °C, dosežejo vrednosti od 7,15 do 12,3 GPa, filamenti, raztezani pri tem-

*the break point. Figure 1 below shows curves of continuously, moderately drawn filaments from both sample groups. The shapes of the curves are similar for all the samples throughout the entire deformation range, especially for the filaments formed from polymer blends. The similarity of the curves indicates that the filaments have similar responses to tensile load, regardless of the polymer blend from which they were made. The filaments made from polymer blends differ from the filaments made from 100% pure polymer in that they reach higher levels of specific stress at the same extension, as compared with samples 100U and 100N. The latter two samples differ in that the filaments made from the lower molecular weight CR-polymer (sample 100U) are more extensible than the other samples and break at significantly higher extensions. The filaments formed from the higher molecular weight polymer (sample 100N) do not exhibit such an abrupt decrease in the specific stress beyond the yield point and show a smaller stress increase, as compared to the other filaments until the break point. The curves of filaments consisting of a 40% share of the second polymer in the blend (samples 60N and 60U) differ from those of the other samples. The curves of these two filaments lie between the curves of samples 100U and 100N and the curves of the filaments made from the other polymer blends.*

*The elastic moduli of the continuously, moderately drawn filaments are low, with values from 1.77 to 3.12 GPa. Correlation with the draw ratio, determined by the stages of production, is low. For the filaments made from the polymer blends consisting of higher molecular weight polymer added to the lower molecular weight CR-polymer, the elastic modulus increased, as compared to sample 100U, from 1.93 to 3.12 GPa, despite being formed at a slightly lower drawing ratio. In addition, the elastic modulus increased with the addition of the CR-polymer to the higher molecular weight polymer; sample 80N has a higher modulus (Eo = 2.9 GPa) than sample 90N (Eo = 2.4 GPa), even though the two samples were drawn at the same draw ratio.*

*The specific stress at yield, with values ranging from 0.9 to 2 cN/dtex, is high, as it is near 50% of the specific stress at break. For all the continuously, moderately drawn filaments, the resist-*

peraturi 145 °C, pa od 9,65 do 14,8 GPa. Moduli so pri obeh temperaturah raztezanja višji pri filamentih, oblikovanih iz surovine s prevladujočim deležem CR-polimera z nižjo molekulsko maso, in v nekaterih primerih celo dosežejo višje vrednosti, kot jih dosežejo filamenti, oblikovani iz surovine s prevladujočim deležem polimera z višjo molekulsko maso, čeprav so bili slednji raztezani pri večjih raztezalnih razmerjih. Najvišji modul elastičnosti ($E_o$ = 14,8 GPa) imajo pri temperaturi 145 °C dodatno raztezani filamenti, oblikovani iz polimerne mešanice, sestavljene iz 90 % CR-polimera z nižjo molekulsko maso in 10 % polimera z višjo molekulsko maso ter široko porazdelitvijo molekulskih mas (*vzorec 90U/145*). Dodatno raztezani filamenti pri 130 °C in pri 145 °C nimajo izrazitega prehoda iz elastičnega v viskoelastično obnašanje, razvidno iz krivulje, izrazitega znižanja upora proti raztezanju, ki ga podaja modul v prvi obračalni točki ($E_1$), in ne kažejo značilne utrditve in povečanja upora proti raztezanju, podanega z modulom v drugi obračalni točki ($E_2$). Specifična napetost polzišča in pripadajoči raztezek imata nižje vrednosti, kot so le-te pri neprekinjeno, delno raztezanih filamentih (*pregl. 2*). Tako je specifična napetost od 0,6 do 1,4 cN/dtex, kar ne predstavlja niti 15 % vrednosti specifične pretržne napetosti. Krivulja specifična napetost/raztezek se ves čas strmo dviga, prirastek napetosti na prirastek raztezka je velik do pretrga.

### *3.3 Medsebojna odvisnost nateznih lastnosti in razteznega razmerja*

Linearna korelacija specifične pretržne napetosti dodatno, ločeno raztezanih filamentov s strojnim in dejanskim razteznim razmerjem je dokaj visoka pri obeh temperaturah raztezanja ($r_{xy}$ od 0,69 do 0,88), medtem ko pri samo neprekinjeno, delno raztezanih filamentih ne obstaja. Podobno je pri korelaciji med dejansko specifično pretržno napetostjo in razteznim razmerjem, le da so vrednosti korelacijskega koeficienta nekoliko nižje. S slike 4 je

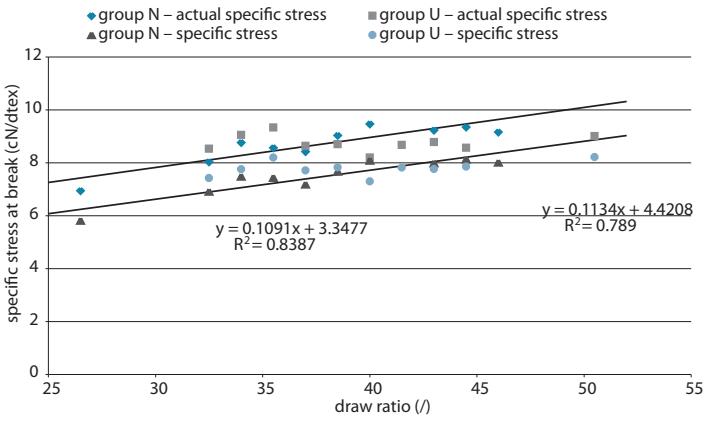


*Figure 4: The specific stress at break of the additionally, discontinuously drawn PP filaments, depending on the draw ratio (nominal and actual).*

*ance to drawing was greatly reduced after the yield point. Immediately after the yield point, the filaments are deformed without increasing the stress. In most of the samples, even a reduction in stress is present with the deformation, and the modulus at the first turning point (E1), which represents the lowest value of the modulus after the yield point, reaches a negative value. As the deformation process continues, the increase in extension is much higher than the increase in stress; from the shape of the curve, it can be seen that the majority of this deformation is non-recoverable. The obvious strengthening of the material that occurs during the tension test is not present, and the absence of a maximum of the first derivative, which represents the modulus at the second turning point (E2), i.e., the modulus of strengthening, confirms this. The low values of the elastic modulus and the modulus at the first turning point (E1) and the overall curve show that the filaments exhibit little resistance to deformation under the influence of tensile load and are still very extensible.*

### *3.2.2 Additionally, discontinuously drawn filaments*

*The shapes of the curves of additionally, discontinuously drawn filaments are typical for strongly drawn filaments, with a short initial elastic area without a clearly marked yield point and a transition to viscoelastic behavior with a steeper increase in specific stress occurring with the extension until the break point. The first derivative of the curve sharply drops, then slowly and constantly decreases until the break point.*

*The filaments drawn at a temperature of 130°C (Fig. 2) show curves similar to those of the filaments drawn at a temperature of 145°C (Fig. 3). The latter, which were drawn at a higher draw ratio, are more resistant to the action of the tensile load and are less extensible than the filaments made from the same polymer blend, additionally drawn at a lower temperature and lower draw ratio. The curves of the filaments made from the higher molecular weight polymer (samples from group N) differ more with each other than the filaments made from the polymer blends with a dominant share of the lower molecular weight CR-polymer (samples from group U). The curves of the latter are steeper and short-*

razvidno, da pri dodatno, ločeno raztezanih filamentih specifična pretržna napetost in dejanska pretržna napetost linearno naraščata z razteznim razmerjem pri *vzorcih skupine N*, medtem ko *vzorci skupine U* že pri nižjih razteznih razmerjih dosežejo visoko specifično pretržno napetost in potem kažejo le rahel trend naraščanja z razteznim razmerjem.

Linearna korelacija pretržnega raztezka filamentov z razteznim razmerjem je po posameznih fazah izdelave zelo nizka ($r_{xy}$ med –0,44 in –0,52), saj na pretržni raztezek filamentov ne vpliva samo raztezno razmerje, temveč tudi molekulska struktura izhodnega polimera, pogoji oblikovanja in s tem povezana nadmolekulska struktura filamentov. S slike 5 pa je razvidno, da pri dodatno, ločeno raztezanih filamentih pretržni raztezek linearno pada z razteznim razmerjem.

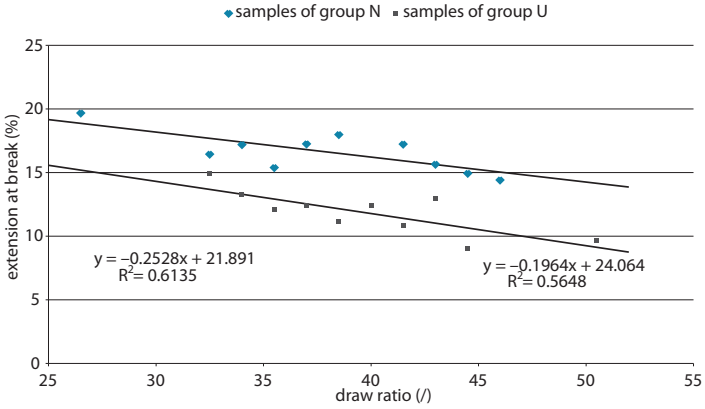


*Figure 5: The breaking extension of the additionally, discontinuously drawn PP filaments, depending on the draw ratio.*

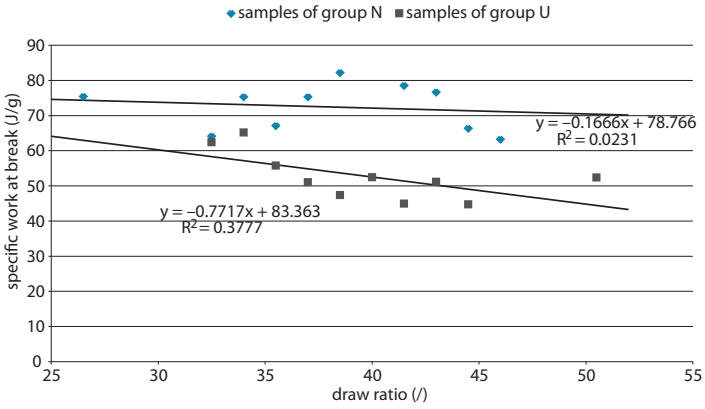


*Figure 6: The specific work at break of the additionally, discontinuously drawn PP filaments, depending on the draw ratio.*

Tudi med specifičnim pretržnim delom in razteznim razmerjem linearne korelacije, določene po posameznih fazah izdelave, ni. S slike 6 je razvidno, da pri dodatno, ločeno raztezanih filamentih iz

*er than the curves of the first group, although some filaments in group U were drawn at a lower draw ratio than the filaments of group N. At both drawing temperatures, the curves of the filaments made from polymer blends of 100%, 90%, 80% and 70% lower molecular weight CR-polymer are steeper and have a similar shape. This group is followed by a second group, which is made up of filaments formed from 60/40, 50/50, 30/70 and 10/90 lower molecular weight CR-polymer / higher molecular weight polymer, which achieve similar high values of stress at higher extensions. The shapes of the curves of these filaments are similar. That indicates that they will exhibit similar responses to the action of a tensile load, although the draw ratios with which they were made vary greatly.*

*Additional drawing at high temperature, which significantly reinforces the filaments and reduces their stretchability, greatly increases the elastic moduli of the filaments. PP filaments drawn at a temperature of 130°C have modulus values of 7.15 to 12.3 GPa, while filaments drawn at a temperature of 145°C have modulus values ranging from 9.65 to 14.8 GPa. For both drawing temperatures, the moduli are higher in the filaments made from the polymer blends with a prevailing share of the lower molecular weight CR-polymer and, in some cases, even reach higher values than those achieved in filaments made from the polymer blends with a dominant share of the higher molecular weight polymer, although the latter were drawn at a higher draw ratio. The highest elastic modulus (Eo = 14.8 GPa) was observed in the filaments drawn at a temperature of 145°C, made from the polymer blend consisting of 90% of the lower molecular weight CR-polymer and 10% of the higher molecular weight polymer with a broad molecular weight distribution (sample 90U/145).*

*Additionally drawn filaments drawn at 130 and 145°C do not have an apparent yield point or an abrupt transition from elastic to viscoelastic behavior in the curves, with a sharp reduction in resistance to extension, which is determined by the modulus at the first turning point (E1), and do not show significant strengthening or increased resistance to extension, given by the modulus at the second turning point (E2). The specific stress and associated extension are*

*skupine U* specifično pretržno delo pada z razteznim razmerjem, medtem ko pri vzorcih *skupine N* specifično pretržno delo ne kaže soodvisnosti z razteznim razmerjem.

Modul elastičnosti z večanjem raztezнega razmerja narašča. Pri obeh skupinah vzorcev je medsebojna odvisnost naraščanja modula elastičnosti ter večanja strojnega in dejanskega raztezнega razmerja, računana ločeno za posamezno skupino vzorcev, vključujoč vse faze izdelave pri posamezni skupini, zelo visoka ($r_{xy}$ = 0,97). Linearna korelacija, določena po stopnjah izdelave, pa podobno kot pri specifični pretržni napetosti pri neprekinjeno raztezanih filamentih ne obstaja, pri dodatno, ločeno raztezanih filamentih pa ima raztezno razmerje večji vpliv, zato je razvidna določena soodvisnost ($r_{xy}$ = 0,7 pri temperaturi raztezanja 130 °C in $r_{xy}$ = 0,6 pri temperaturi raztezanja 145 °C). Modul elastičnosti dodatno, ločeno raztezanih filamentov narašča z naraščanjem raztezнega razmerja pri obeh skupinah vzorcev (sl. 7). Visoka linearna korelacija obstaja med modulom elastičnosti in faktorjem povprečne orien-

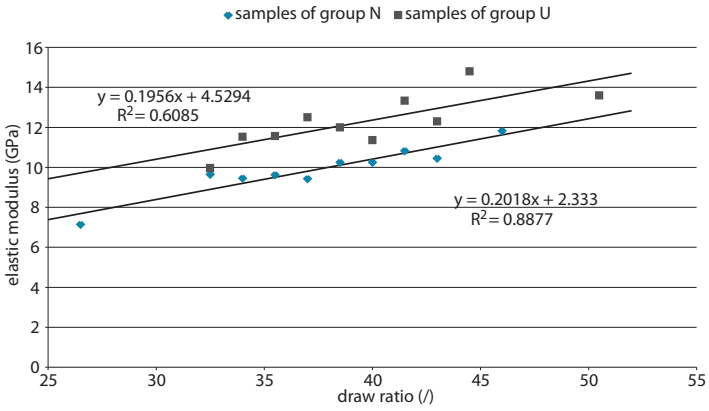


*Figure 7: Elastic modulus of the additionally, discontinuously drawn PP filaments, depending on the draw ratio.*

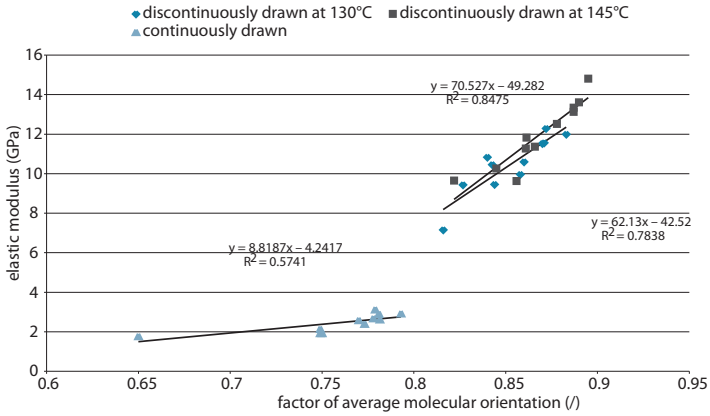


*Figure 8: Elastic modulus of the continuously, moderately and of additionally, discontinuously drawn PP filaments, depending on the molecular orienatation.*

*lower than those of the continuously, moderately drawn filaments (Fig. 2). Thus, the specific stress ranges from from 0.6 to 1.4 cN/dtex, which does not constitute 15% of the value of specific stress at break. The specific stress / extension curves constantly rise; the increase in specific stress is rapid until the break point.*

### 3.3 Correlation between tensile properties and drawing ratio

*The linear correlation of the specific stress of the additionally, discontinuously drawn filaments with the draw ratio and the actual draw ratio is relatively high ($r_{xy}$ from 0.69 to 0.88) at both drawing temperatures, while for the continuously, moderately drawn filaments, no correlation is found. A similar situation occurs for the correlation between the real specific stress at break and the draw ratio, except that the correlation coefficient is slightly lower. From Figure 4, it can be seen that, for the additionally, discontinuously drawn filaments, the specific stress at break and the real specific stress at break increase linearly with the draw ratio for the samples from group N, while the samples from group U achieve a high specific stress even at lower draw ratios and then show only a slight increase with the draw ratio.*

*The liner correlation of the breaking extension of the filaments with the draw ratio at each processing stage is very low ($r_{xy}$ between –0.44 and –0.52), since the breaking extension of the filaments depends not only on the draw ratio, but also on the molecular structure of the polymer, the processing conditions and the related supramolecular structure of the filaments. From Figure 5, it is clear that for the additionally, discontinuously drawn filaments, the breaking extension decreases linearly with the draw ratio.*

*In addition, a linear correlation between the specific work at break and the draw ratio at each processing stage is not found. From Figure 6, it can be seen that for the additionally, discontinuously drawn filaments from the samples from group U, the specific work at break decreases with the draw ratio, while those of the samples from group N show no correlation with the draw ratio.*

*The elastic modulus of the filaments increases with increasing draw ratio. In both groups*

tacije polimernih molekul, kar je razvidno s slike 8. Modul elastičnosti filamentov se povečuje predvsem z naraščanjem molekularne orientacije, stopnja kristalinosti pa ima manjši vpliv.

## 4 Zaključek

Filamenti, oblikovani iz polimerne mešanice s prevladujočim deležem CR-polimera z nižjo molekulsko maso (*vzorci skupine U*), prenesejo visoke pretržne napetosti in se pri tem manj raztezajo. Filamenti, oblikovani iz polimerne mešanice s prevladujočim deležem polimera z višjo molekulsko maso in širšo porazdelitvijo molekulskih mas (*vzorci skupine N*), dosežejo podobno visoke pretržne napetosti, vendar pri višjih pretržnih raztezkih. Razlike v mehanskih lastnostih filamentov, oblikovanih iz različnih polimerov, so velike pri obeh temperaturah raztezanja. Tako doseže *vzorec 100N/145* pri trdnosti 6,91 cN/dtex in 16,4 % pretržnem raztezku modul elastičnosti 9,65 GPa, *vzorec 100U/145* pa pri 9,65 % pretržnem raztezku trdnost 8,22 cN/dtex in modul elastičnosti 13,6 GPa, ki je za okoli 4 GPa višji kot pri *vzorcu 100N/145*. To vzorec 100U/145 uvršča med visokomodulna, visokotrdna PP-vlakna. Dodatek drugega polimera v obeh primerih izboljša mehanske lastnosti filamentov. Maksimalno možno raztezno razmerje, struktura in lastnosti filamentov so predvsem odvisni od prevladujočega polimera, vendar pa ima določen vpliv tudi delež dodanega polimera. Vzorcem iz prevladujočega polimera z višjo molekulsko maso in široko porazdelitvijo molekulskih mas (*vzorci skupine N*) se z dodatkom CR-polimera z nižjo molekulsko maso poveča mejno raztezno razmerje. To povečanje je pri obeh temperaturah raztezanja višje pri 10 % dodatku CR-polimera. Skladno z večjim razteznim razmerjem so boljše tudi mehanske lastnosti; povečata se pretržna napetost in modul elastičnosti. Pri vzorcih iz prevladujočega CR-polimera z nižjo molekulsko maso (*vzorci skupine U*) majhen dodatek polimera z višjo molekulsko maso (10 in 20 %) ne zmanjša bistveno mejnega raztteznega razmerja. Izboljšanje lastnosti je pri manjšem, to je pri 10 %, dodatku polimera z višjo molekulsko maso k CR-polimeru večje kot pri 20 % dodatku. Po modulu elastičnosti najbolj izstopa *vzorec 90U/145*, ki z vrednostjo 14,8 GPa predstavlja najvišje vrednosti, ki smo jih dosegli v okviru raziskav.

Raziskava je potrdila, da je struktura raztezno preoblikovanih PP filamentov oblikovanih iz taline odvisna od molekulskih mas in od porazdelitve molekulskih mas izhodnega polimera. Ugotovljeno je bilo:

- da je orientacija makromolekul večja pri filamentih, oblikovanih iz polimera z ozko porazdelitvijo molekulskih mas,
- da je razteznost filamentov, oblikovanih iz polimera s široko porazdelitvijo molekulskih mas pri nižjih temperaturah preoblikovanja večja, kot razteznost filamentov oblikovanih iz polimera z ozko porazdelitvijo molekulskih mas, pri višjih temperaturah preoblikovanja pa je vpliv porazdelitve molekulskih mas minimalen,

*of samples, the mutual dependence of the elastic modulus and the draw ratio, calculated separately for each group of samples, including all phases of production in each group, is very high ($r_{xy}$ = 0.97). Similar to the specific stress at break, the linear correlation determined at each level of the production process does not exist for the continuously, moderately drawn filaments; in the additionally, discontinuously drawn filaments, the draw ratio has a greater impact, it exhibits a correlation with the elastic modulus: $r_{xy}$ = 0.7 at a temperature of 130°C and $r_{xy}$ = 0.6 at a temperature of 145°C. The elastic modulus of the additionally, discontinuously drawn filaments increases with increasing draw ratio for both groups of samples (Fig. 7). High linear correlation exists between the elastic modulus and factor of average molecular orientation, which can be seen in Figure 8. As the orientation of macromolecules increases the elastic modulus of filaments increases too.*

## 4 Conclusion

*Filaments made from a polymer blend with a prevailing share of the lower molecular weight CR-polymer (samples from group U) exhibited a high elastic modulus and specific stress and a low extension at break. Filaments made from polymer blends with a prevailing share of the higher molecular weight polymer with a broader molecular weight distribution (samples from group N) achieved a similar high specific stress at break but at a higher breaking extension.*

*The differences in the mechanical properties of the filaments made from the different polymers are great for both drawing temperatures. Sample 100N/145, with a tensile tenacity of 6.91 cN/dtex and a breaking extension of 16.4%, exhibited an elastic modulus of 9.65 GPa; sample 100U/145, at a breaking extension of 9.65%, showed a tenacity of 8.22 cN/dtex and an elastic modulus of 13.6 GPa, which is about 4 GPa greater than the modulus of sample 100N/145. Thus, sample 100U/145 can be regarded as a high-modulus, high-tenacity polypropylene filament. In both cases, the addition of another polymer improves the mechanical properties of the filaments. The maximum attainable draw ratio, the structure and the properties of the*

– da so pri visokih razteznih razmerjih filamenti oblikovani iz polimera z ozko porazdelitvijo molekulskih mas presegli modul elastičnosti in natezno trdnost filamentov, oblikovanih iz polimera s široko porazdelitvijo molekulskih mas.

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*filaments are primarily dependent on the prevailing polymer, though the added polymer has some influence. For the filaments made from a blend with a prevailing share of the higher molecular weight polymer with a broader molecular weight distribution (samples from group N), the draw ratio increased significally with the addition of the lower molecular weight CR-polymer. This increase, observed at both drawing temperatures, is higher at a 10% addition of the latter polymer. Consistent with the higher draw ratio, the mechanical properties of the filaments are improved; the specific stress at break and the elastic modulus increase. For the filaments made from blends with a prevailing share of the lower molecular weight CR-polymer (samples from group U), a small addition of the higher molecular weight polymer (10 and 20%) did not significantly reduce the draw ratio. Greater improvement of the filament`s properties occurred with the smaller (10%) addition of the higher molecular weight polymer to the lower molecular weight CR-polymer, as compared to a 20% addition. Sample 90U/145 is distinct from the other samples in its elastic modulus value. With a value of 14.8 GPa, this sample represents the highest elastic modulus achieved in the context of this study.*

*Our research has confirmed that the structure of drawn melt-spun PP filaments depends on the molecular weight and molecular weight distribution of the polymer. It was established:*

- *that the orientation of macromolecules is higher in the filaments made from the polymer with narrow molecular weight distribution,*
- *that the drawability of the filaments made from the polymer with broad molecular weight distribution is greater than drawability of the filaments made from the polymer with narrow molecular weight distribution at the drawing temperature of 50°C, whereas the impact of the molecular weight distribution at 130 and 145°C is minimal,*
- *that at high draw ratio the filaments made from the polymer with narrow molecular weight distribution exceed the elastic modulus and tensile strength of the filaments, made from the polymer with broad molecular weight distribution.*

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