IJE TRANSACTIONS A: Basics Vol. 36 No. 04, (April 2023) 788-796

International Journal of Engineering

Journal Homepage: www.ije.ir

High Energy Release-high Retraction Smart Polymer Fibers used in Artificial Muscle Fabrication

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P A P E R I N F O

Paper history:
Received 07 January 2023
Received in revised form 23 December 2023
Accepted 06 February 2023

Keywords:
Blocked Force
Energy Release
Tensile Behavior
Smart Fiber
Shape-memory Polymer

A B S T R A C T

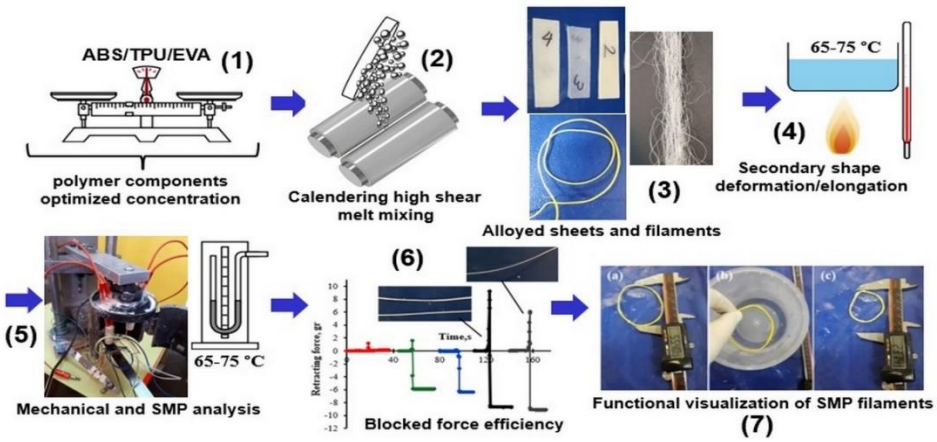
Shape-memory polymers (SMPs) could remember their original shape and then, return to their initial shape upon stimulus. So far, quantities such as fixity and recovery ratio of SMPs have been broadly reported. Nevertheless, one main issue is the existence and use of an appropriate approach to quantitatively estimate the SMPs released energy. In addition, it is hypothesized that the elastic behavior of SMPs plays an underlying role when SMP fibers need to exhibit high-tension and high-elongation capacity as required in synthetic muscles. Here, we present, for first time, SMP trinary bulk and filament systems of acrylonitrile butadiene styrene (ABS)/thermoplastic polyurethane (TPU)/ethylene vinyl acetate (EVA) fabricated under calendering intense shear mixing and hot pressing. A digital blocked force load cell was used to record specimens energy released. The results exhibited high retraction from the specimens secondary shape correlated to the tensile behavior. It was shown the blended system of 50, 25 and 25% of TPU, ABS and EVA, respectively, resulted in ~640% and 3900% increase in the elastic modulus and energy release compared to EVA/TPU systems. The double SMP filaments led to a 50% increase in the energy release compared to single fibers. Nevertheless, the blended binary specimens with the TPU/ABS ratio of 50/50 exhibited 600% increase in tensile strength. It was confirmed the elastic modulus, number of fibers and elongation at break govern the SMP stored. The findings of the research lightened a new class of SMPs to be used as fiber-based artificial muscles and orthodontic products.

***doi**: 10.5829/ije.2023.34.04a.15*

NOMENCLATURE

K	Equivalent stiffness (N/mm)	P_i	Forced applied on each fiber
K_i	Stiffness of each spring (N/mm)	W	total weight to be lifted
N	Number of fibers		

Graphical Abstract



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Please cite this article as: M. Karevan, M. A. Motalleb, A. Jannesari, M. Zeinali, High Energy Release-high Retraction Smart Polymer Fibers used in Artificial Muscle Fabrication, ***International Journal of Engineering, Transactions A: Basics***, Vol. 36, No. 04, (2023), 788-796

1. INTRODUCTION

Amongst smart materials shape-memory polymers (SMPs) could be listed due to their memory to return to their original shape with some approximation upon receiving an environmental trigger such as heat, moisture and chemicals [1, 2]. Thermal sensitive SMPs require temperature around the glass transition (T_g) or melt temperature (T_m) of the polymer components to implement the start of shape-memory cycle from the secondary to the original shape [3, 4]. Although, their mechanical performance limits their applications in structural sectors, they can find the wider exploitation in areas where mechanical response of the parts is of inferior issue. The latter has paved an avenue toward the utilization of SMPs in bio-medical parts [5, 6].

To develop muscle fibers, polymers of low transitional temperatures together with thermoplastic elastomers such as thermoplastic polyurethane (TPU) and ethylene vinyl acetate (EVA) blended with high stiffness polymers such as polylactic acid (PLA) have been widely reported in the literature [7-10]. The blend of such materials exhibits the network points and molecular switching phase that have been generated through covalent bonds, intermolecular interactions and cross-links [11]. The network points as hard segments serve as pivoting point for shape memory whilst the soft segment serves as an energy absorbing phase [12]. However, the ability of SMP parts to exhibit appropriate mechanical performance might be a pressing challenge as seen in the development of orthodontic filaments, surgical sutures and fibers in the devolvement of artificial muscles in humanoids or those used as scaffolds [13]. One underlying role of SMP parts derived from mechanical characteristics is the amount of energy stored in the secondary phase of SMPs to be released upon stimulus [14-16]. The higher the stored strain energy, the greater the recovery ratio of the specimens. The latter is proposed to be a criterion for SMP fibers able to lift heavier masses using two-way actuation or to tighten other objects upon being stimulated [17, 18]. The former could be correlated to the design and fabrication fiber-based muscles accommodating greater energy stored [19]. Therefore, whilst numerous studies have been elsewhere reported various SMP systems, less research so far has focused on the quality of SMP parts in energy release [14, 20]. In addition, along the ability of SMPs in energy release to perform a work by lifting masses, the extent SMP fibers could be stretched followed by their retraction (contraction) is of another requirement as seen in natural muscle fibers in human body [19]. The latter has been mostly correlated to the recovery ratio of SMPs [21]. However, the recovery ratio itself expresses the ability of SMPs to fully return to their initial shape regardless of the length difference in fibers of original shape and that in the secondary shape status. It is hypothesized that the greater the said difference, the more capable the SMP fibers to be used as artificial muscle fibers as the nature suggested [22]. Therefore, the stretchability of SMP fibers whilst simultaneously retaining their shape-memory character is a crucial factor. The soft region so far called the amorphous phase is mostly responsible for the fixation ability whilst the crystalline phase is ascribed to the SMPs recovery ratio. Whilst the apomorphs phase contributed to the stretchability upon heating, the net points are expected to enhance the recovery of SMP fibers. As understood, such effects contradicts and, thus, the formulation of SMP blends of high stored energy could be a serious challenge to be addressed [23, 24].

Mirvakili and Hunter [25] used nylon fiber actuators fabricated on the basis of highly oriented nylon filaments representing a 5% deformation with a temperature change from 25 to 140 °C with reservable amplitude. Xie et al. [26] used poly (ethylene-co-vinyl acetate)/graphene (cEVA/G) to develop shape-memory actuators. Their EVA-carbon fiber based composites (EVA/CF) resulted on a significant enhanced recovery stress. Ma et al. [27] developed a mixed-matrix membrane strategy to fabricate photo-induced SMP artificial muscles that were able to lift masses with a fast light response and the high elastomer properties. Yip and Niemeyer [28] employed conductive polyamide 6.6 sewing thread to design high-performance robotic muscles using electric heating. Xiao et al. [29] used liquid crystal polymer (LCP) in the preparation of Janus flower-like structure to design and fabricate soft robots based on the concept of order-disorder phase transition. Although the incorporation of micro- and nanofillers into polymers has been considred to improve SMP properties, the use of pure polymers of right blending ratio could be still a more reasaonable approach in terms of processability and cost [30].

Here, we proposed a shape-memory trinary blend system of Acrylonitrile butadiene styrene (ABS)/thermoplastic polyurethane (TPU)/Ethylene vinyl acetate (EVA) bulk and fiber fabricated through melt mixing method on a two-roller mixing calendering followed by hot pressing. The filaments were manually fabricated by applying tensile tension on the hot melt pieces of alloyed strips. Blocked force method and tensile testing were performed to determine the optimized level of polymer components in terms of energy recovery and tensile response of the parts. The findings of the research expressed a methodology in formulation and fabrication high energy release SMPs containing an optimized level of polymer ratios which are capable of being transformed to filaments. The latter may suggest application of the formulated compounds in design and fabrication of polymer fibers to be used in design of artificial muscles due to their high elastic stored energy.

2. EXPERIMENTAL

2. 1. Material In the current work, PLA (KAS GmBh, Austria), TPU (EPAFLEX, Italy) and EVA granules (28%, EPAFLEX, Italy) were used as the thermoplastic matrices. No additional treatment and chemicals modification was performed on the polymer granules.

2. 2. Shape-memory Film and Fiber Fabrication To fabricate SMP alloyed films, a two-roller calendering device was utilized to melt mixed the polymer components of TPU (0 to 70%), ABS (0 to 70%) and EVA (0 to 50%) under heat and intense shear forces. To decide on the loading fraction of each polymer phase, expected mechanical properties of each polymer was taken into consideration followed by a simple rule of mixtures method to estimate overall mechanical behavior of the parts. It was hypothesized that the higher the fraction of the EVA and TPU part, the greater the elongation and processability of the alloyed systems [31, 32]. On the other hand, it was expected that the ABS would contribute to the tensile strength and modulus of the fabricated parts. The temperature of rollers in the calendering machine was set to ~140° to 160°C at 5-10 rpm and the granules were first gradually fed into the rollers gap to ensure the melting state onto the heated rolls under intense shear forces. To fabricate the films, the gap between the rollers then was decreased to the desired sheet thickness followed by gradual temperature decrease of the rollers in air. The melt alloyed compound was removed from the rollers surface for the hot press processing under the temperature of 150°C to fabricate the films. The fabricated specimens in form of sheets are displayed in Figure 1.

The strips (bulk specimens) per ASTM D882 standard were cut from the sheets of less than 1 mm for the tensile and energy release characterization steps as displayed in Figure 2. The same dimensions were used for the specimens in the blocked force measurement analysis. The SMP fibers were fabricated using a pre-tension onto the melt compound of the trinary polymer systems at the melt state of the compound collecting from the roller's surface drawn so that a diameter of 200 to 800 µm is achieved for blocked force measurements as shown in Figure 3.

Figure 1. Blended polymer systems onto the calendering rolls in form of thin layer sheets after hot pressing

Figure 2. Polymer blended strips (bulk) of ABS/TPU/EVA

Figure 3. Fabricated SMP fibers of ABS/TPU/EVA

Figure 4 represents the fabricated fibers (filaments) that are in the form of single, multiple bundled, twisted or numerous paralleled fibers from Figures 4(a) to 4(d), respectively.

It is hypothesized that the number of fibers in the form of yarn or bundled filaments affects the amount of energy stored in the softer phases of the alloyed systems to be returned to the fibers initial length upon heating influenced by the presence of net points (hard regions) in either polymer phases [15, 33].

It is noted that multiple fibers parallelly aligned would behave similar to a stronger muscle containing fibers which mimic elastic springs whose equivalent stiffness (K) is the sum of the stiffness of each spring (k_i).

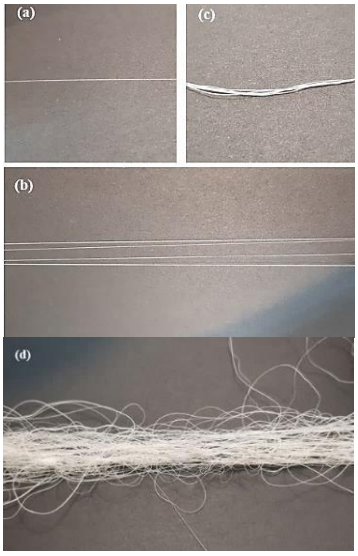

Figure 4. Polymer blended fibers in the form of a) single, b) multiple aligned (bundled), c) twisted (yarn) and d) numerous paralleled

It is clearly understood that each spring (fiber) tolerates a portion of weight collectively applied as expressed by Equation (1):

$$P_i = \frac{W}{N} \quad \text{and} \quad \text{i=1 to N} \tag{1}$$

where P_i is the forced applied on each fiber, N the number of fibers in a bundle or yarn and W the total weight to be lifted. Therefore, the overall stiffness of the bundle (multiple fibers) as given by Equation (2) as follows:

$$K = K_1 + K_2 + \cdots K_N \tag{2}$$

2. 3. Characterization of ABS/TPU/EVA SMPs

2. 3. 1. Tensile Testing The tensile response of the ABS/TPU/EVA alloyed systems including the Young's modulus, elongation and tensile strength was determined based on ASTM D882 using a universal tensile testing machine (Sanaf Co., Iran). Specimens measuring the length of 100 mm and width of 10 mm with the thickness of less than 1 mm were cut from the blended sheets as mentioned earlier. The gage length of 50 mm was considered in the experiment as the grip separation. Measurements were obtained at the deformation rate (stroke speed) of 12.5 mm/min at the ambient temperature.

2. 3. 2. Blocked Force The shape-memory recovery measuring the released energy stored in the specimens in the second SMP cycle (heating-deformation-cooling) at their secondary state above their transitional temperature was measured using the blocked force method. The measurements were conducted on both fibers and strips to understand the correlations amongst the mass, shape, number of fibers and the energy stored in the SMPs. The heat was applied onto their temporary shape and the force applied by specimens release energy was assessed using a home-made strain gage transducer. To apply heat, two parallel heating elements within a cylindrical chamber surrounding the specimens as the built-in accessory of the load cell was used. The transition temperature of the specimens was specified once the load cell started showing changes in its output current. Due to the low average T_g values of EVA and TPU (being around ~ -25 to – 30 °C and -50 to 10 °C, respectively [34, 35]), it was found that the specimens started contraction at temperatures in the range of 65-75 °C as their SMP transition temperature. To perform the test, two ends of the pre-tensioned SMP wires/yarn were initially fixed at two rigid and deflectable supports of the blocked force system. Upon fibers or strips shortening, the single cantilever beam deflected and linked to the applied force made by the SMP fibers or strips. The block force apparatus was initially calibrated to gain the input-output slope as shown in Figure 5.

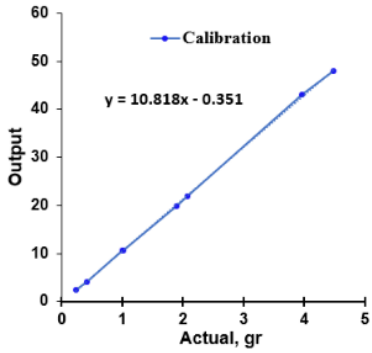

Figure 5. Calibration curves exhibiting input vs output values

The SMP filament and bulk specimens and the used loadcell setup showing the specimens fixed are illustrated in Figure 6(a) and (b). Figure 6(c) further represents the bulk flexed specimens in the secondary SMP shape upon fixation in the cold water to be activated by thermal stimulus on the blocked force set-up (also revisit Figure 2). Based on the former information provided as the transition temperature initiation, the bulk specimens were placed into water with the temperature in the range of 65-75 °C followed by quick mechanical deformation around a curved surface to obtain their secondary fixed shape (temporary shape). In case of fibers, it is noted that the fibers were initially stretched in water with the said temperature up to a diameter reduction of around 25% compared to their original diameter (~200 to 800 µm). It is concluded that considering the total contraction of the fibers (above 50%) as explained in next part, a diameter swelling of ~ 12% is expected upon thermal stimulation.

3. RESULTS AND DISCUSSION

3. 1. Tensile Properties Figure 7 represents the tensile testing results on the ABS/TPU/EVA specimens as a function of the polymer ratios used. It is illustrated

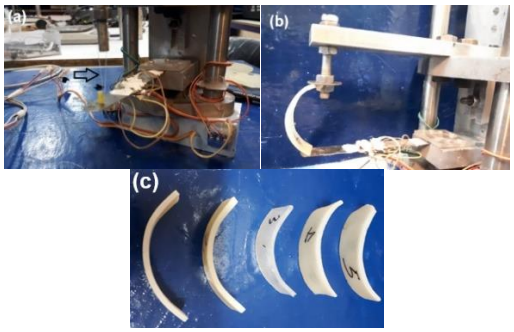

Figure 6. Blocked force load cell measuring the energy released by (a) fibers, (b) bulk specimens and (c) the bulk specimens in the secondary (flexed) shape before thermal activation

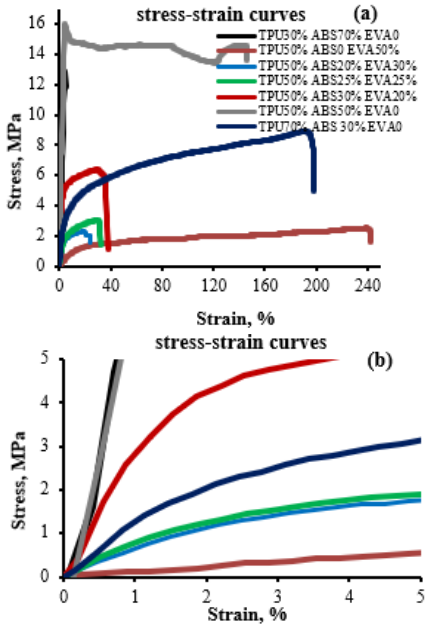

Figure 7. (a) Tensile stress-strain curves of ABS/TPU/EVA alloyed systems and (b) of which the inset at low-strain regions

in Figure 7(a) that the presence of ABS as the harder polymer significantly affects the overall tensile strength as expected as shown by the gray and black curves [36].

Further, it is confirmed that when EVA is incorporated into the alloyed systems, the elongation of the sheets increases, and the more the EVA content, the greater strain at break of the parts which is in good agreement with results reported elsewhere [31]. It is clearly shown in the figure that lower amount of ABS and greater fraction of EVA result in low tensile behavior of the specimens as exhibited by ABS content less than 25% and that in the case of EVA above 20%. It is shown, however, that lack of EVA in the specimens as seen in the case of TPU/ABS ratio of 70/30 leads to an optimized level of both tensile elongation and strength (dark blue line). It is shown that upon the addition of EVA replaced by the same fraction of TPU, the elongation and tensile strength decrease as shown by the red curve. To better examine the effect of the polymer ratios on the elastic modulus of the specimens, an inset of the graph was prepared at low strain regions as demonstrated in Figure 7(b).

It is clearly shown that similar to the tensile strength performance of the specimens, the addition of ABS phase leads to greater modulus values. Nevertheless, when EVA is added into the blends, the modulus of the specimens sharply drops shown by lower slop of the curves represented in the inset graph. It could be understood that both EVA and TPU negatively contribute to the tensile modulus whilst the former more noticeably influences the tensile behavior of the fabricated specimens. To compare the obtained results, the tensile strength and modulus values are reported and summarized in Figures 8 and 9, respectively. It is shown that the high loading of EVA in overall results in the lowest tensile strength of the specimens unlike addition of ABS (~2.3 MPa). It is understood that equal fraction of ABS and TPU leads to the greatest strength value of ~ 16 MPa. It is demonstrated in Figure 8 that the blended system containing 50, 50 and 0% of TPU, ABS and EVA, respectively, exhibits the tensile strength above 600% greater that than in the case of parts with the loading of 50, 20 and 30% of TPU, ABS and EVA, respectively, as the worse blending scenario. In meantime, blended specimens composing of 50, 25 and 25% of TPU, ABS and EVA, respectively, lead to the greatest elastic modulus 640% higher than that in parts with the mixture of only 50 and 50% of TPU and EVA, respectively, as understood from modulus values in Figure 9. The former is hypothesized to exhibit favorable SMP energy release, too. As shown in Figure 7(a), the latter system results in the greatest elongation at break of 240% as expected.

Figure 9 illustrates that EVA can help to increase modulus at specific fractions of ABS and TPU unlike its general effects on the tensile performance (addition of 25% of EVA into 50% and 25% of TPU and ABS, respectively). However, both EVA and TPU as soft

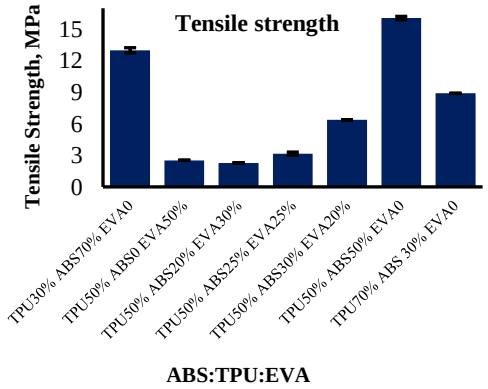

Figure 8. Tensile strength of the blended specimens

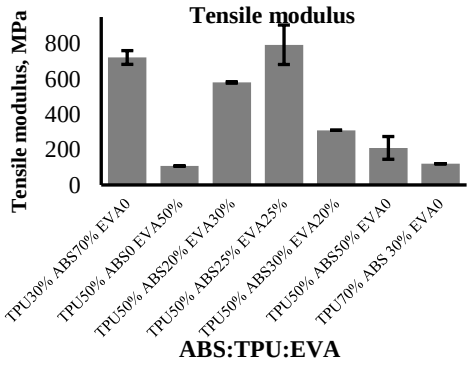

Figure 9. Tensile modulus of the blended specimens

regions compared to the ABS phase exhibit adverse effect on the modulus of the specimens as widely reported in literature [37].

3. 2. Blocked Force Energy Release The blocked force values of the alloyed specimens are shown in Figure 10. The results represent that the amount of released energy upon activation of the specimens is markedly sensitive to the type and percentage of polymer component used. It is clearly shown that TPU and high ABS ratios would lead to minimal energy released. However, it is exhibited that when 50% of TPU is mixed with ABS and EVA of the same ratio the greatest degree of energy released (blocked force) is obtained shown by the black solid curve.

It is concluded that the number and quality of net points (represented by rigid regions within the amorphous phases) significantly contribute to the amount of stored energy in the fixation cycle (cooling and secondary shape) of SMPs used in this study as reported elsewhere in other studies [38, 39]. Nevertheless, it is shown that the larger the loading of softer polymers such as EVA and soft TPU, the lower the level of the energy released upon thermal activation of bulk specimens (shown by green and blue curves).

It is noted that the drop displayed on the curved vs time is correlated to the soft nature of polymer components upon heating and a contraction due to the forced applied by the elastic and rigid supports in the load cell. It is clearly shown that the ABS/TPU ratio of 50/50 still results in low energy released due to lack of enough soft region to be recoiled upon actuation. One needs to keep in mind that the flexed bulk specimens as shown in Figure 6(c) are placed between two load cell supports when the parts are in their secondary bent state. Upon heating, the specimens incline to recover to their original straight state and thus exerting deflection onto the elastic single-cantilever support of the blocked force system. It is noted based on Figure 10 that the higher the modulus of the specimens, the greater the energy released [40, 41].

As easily understood from the figure, bulk specimens resulting in the greatest modulus (50, 25 and 25% of TPU, ABS and EVA) also exhibit the greatest energy release of ~6 grf that is above 3900% greater than the blended SMP systems showing the least energy release. When double filaments are used, the energy release increases by 50% with respect to the single wire case (9.25 grf vs 6 grf) which is in overall 6000% greater than blended systems with the TPU/EVA ratio of 50/50 with absence of ABS phase.

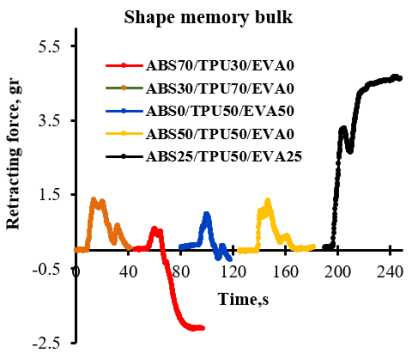

Figure 10. Blocked force values as a function of ABS based alloyed systems and TPU in bulk specimens

The same measurements were conducted on the alloyed fibers of the same polymer components as illustrated in Figure 11. Based on tensile and blocked force values on bulk specimens demonstrated in Figure 7 and 10, respectively. It was decided to use specific fractions of polymers in the fabrication of the fibers followed by their blocked force analysis. One main limitation is the capability of polymer alloys to be converted to filament. Therefore, an optimized level of polymer components needs to be incorporated into the blends. As shown in the figure, the binary alloyed systems result in minimal released energy upon heating and consequent shortening (contracting) of the fibers as shown by the green, blue and red solid curve [12, 16]. However, ABS systems loaded with 50 and 25wt% of TPU and EVA, respectively, lead to greater energy released as shown by the gray and black solid curve. The results are in good agreement with the bulk specimens of the same trinary systems. Nevertheless, by the use of a single fiber specimen, the amount of force applied on the load cells decreases (gray curve) as shown by the inset image.

To better understand the effect of parallel springs constants, two wires were used and their SMP released energy was determined. It is clearly shown that such filaments result in a greater energy release. It is demonstrated that the mass of fibers as well as their secondary length significantly contribute to the applied force onto the load cell cantilever upon stimulation. The greater the contraction of the fibers, the greater the energy released due to the increased overall stiffness of the material as proved by relations 1 and 2 stated earlier. To better show the retraction of the fibers fabricated, visual performance of the fibers was recorded as presented in next part.

3. 3. Visual Performance Figure 12 demonstrates the length difference between secondary temporary shape and shortened filaments before and after thermal activation. It is clearly illustrated that upon activation, the SMP fiber is shortened as shown by the scale ruler (a retraction above 50% was observed). The difference between two stages governs the generated forces and energy released as discussed earlier. The more the filament is retracted, the higher the energy released [42]. Factors including the ratio and type of polymers, activation temperature and the degree of net points define the energy released [43]. Figure 13 rephrases the length difference in Figure 12 whilst the filament is circled

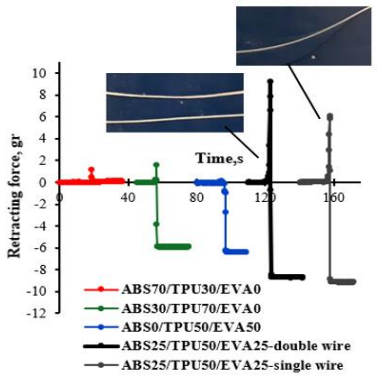

Figure 11. Blocked force values as a function of ABS based alloyed systems and TPU in filament specimens. The insets represent the number of fibers tested

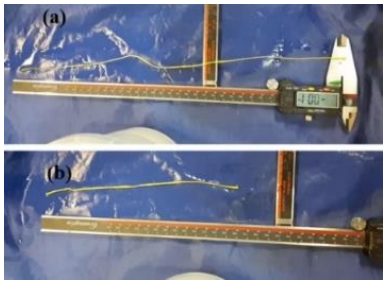

Figure 12. SMP filaments (a) long before and (b) contracted (shortened) upon thermal activation

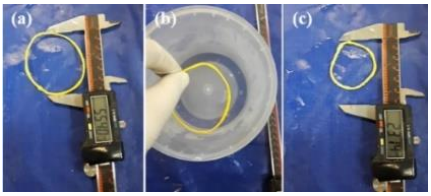

Figure 13. (a) SMP circle filament of larger diameter before activation, (b) thermal activation in warm water and (c) smaller diameter after recovery

mimicking a knot. It is shown that upon activation, the diameter of the circle decreases. The representation of the diameter change is used in simulation of a self-tightening suture knot over an open wound which is tightened upon an activation as another potential application of the current research [44]. The latter conveys that the difference in fiber lengths causes an internal exerted force that is able to close the wound edges as an external force [45].

4. CONCLUSIONS

Successful fabrication of trinary SMPs was demonstrated using ABS/TPU/EVA blended systems fabricated in bulk and filament geometries. To ensure complete blending of the polymers component, high intense shear mixing on a calendering device was employed. Hot press was used to fabricated sheets SMPs after the melt mixing process. Fibers were produced between the two calendering rolls during the melt mixing process and were able to be collected due to their high surface tension. Blocked forced measurement was performed to quantitatively estimate the SMP stored energy of the bulk and fiber specimens. The results confirmed tight correlations amongst the tensile behavior, retraction and energy release of SMPs upon thermal actuation. It was shown specific fractions of the trinary blended SMPs containing 50, 25 and 25% of TPU, ABS and EVA, respectively, led to the greatest elastic modulus and recovery energy release by 640% and 3900% increase, respectively, with respect to binary systems. The dependence of the energy release values on the number of fibers was also represented. The latter showed governing synergistic effect of SMPs parallelly aligned as parallel springs contributing to an increase in the overall spring constant of the system. To better understand the effect of polymer fractions, contraction (retraction) of fabricated filaments upon thermal stimulus was further visually demonstrated. The latter together with the obtained high shape-memory strain stored in the SMPs suggested the application of the trinary formulation of the blends in the design and development of fibers used as synthetic muscles elements or self-tightening surgical sutures.

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Persian Abstract

چکیده

پلیمر ها با حافظه شکلی قادر به خاطرآوری شکل اولیه خود بوده و پس تحریک می توانند به آن شکل اولیه بازگردند. تاکنون مقادیری نظیر نسبت تثبیت و بازیابی بسیار گزارش شده است. با اینوجود، یک چالش اصلی وجود و استفاده از یک روش مناسب برای تخمین کمی مقدار انرژی رهایش حافظه داری است. بعلاوه، اینگونه فرضیه سازی می شود که خواص الاستیک الیاف حافظه دار نقش کلیدی در الیاف موقعی که نیاز است رفتار با درصد تغییر طول زیاد و کشش زیاد داشته باشد دارد همانطور که در ماهیچه های مصنوعی مورد نیاز است. در این تحقیق، ما برای اولین با، ترکیب پلیمرهای حافظه دار بصورت بالک و یا فیلامنت بصورت دو و یا سه جزئی حاصل از ترکیبات پلیمرهای اکریل.بوتادین استایرن (ABS)، پلی یورتان ترموپلاستیک (TPU) و اتیلین وینیل استات (EVA) از طریق ترکیب مذاب با غلتک کلندرینگ تحت تنش برشی شدید و پرس گرم را ارائه می دهیم. یک لودسل دیجیتال برای بررسی نیروی مقید و تثبت نتایج انرژی رهایش استفاده شد. نتایج رفتار حافظه داری عالی را که توسط اندازه گیری نیروی مقید و انقابض از حالت ثانویه را در ارتباط با رفتار کششی نمایش دارد. نشان داده شد که ترکیب پلیمری حافظه دار TPU، ABS و TPU به ترتیب با درصدهای ۵۰٪، ۲۰٪ و ۲۵٪ منجر به افزایش ٦٤٠ درصدی و ۳۹۰۰ درصدی در مدول الاستیک و مقدار انرژی رهایش حافظه داری نسبت به سامانه های ترکیبی دوجزئی تنها با ترکیات EVA و TPU می شود. همچنین نشان داده شد که نمونه های دو فیلامنتی منجر به افزایش ۵۰ درصدی در انرژی رهایش نسبت به تک سیمی شد. با اینوجود، ترکیب دوجزئی TPT/ABS با درصد ترکیب ۵۰/۵۰ افزایش ٦۰۰ درصدی در استحکام کششی در مقایسه با سامانه های سه جزئی گردید. تائید شد که مدول الاستیک، تعداد الیاف و درصد ازدیاد طول در شکست به طور چشمگیری حاکم بر خواص حافظه داری در خصوص مقدار کرنش حافظه داری ذخیره شده است. یافته های این تحقیق گروه جدیدی از پلیمرهای حافظه دار را نمایش داد که می تواند در طراحی و ساخت مواد با رهایش انرژی بالا و انقباض زیاد به منظور کاربردها نظیر ماهیچه مصنوعی پایه الیاف و محصولات ارتودونسی استفاده گردد.