ORIGINAL RESEARCH

Investigating the thickness of patterned polyethylene layers by changing the line speed and temperature in the embossing machine

Ekrem Altuncu[1,2,3] · Nilay Tuccar Kilic[2,3]

Abstract

Patterned polyethylene films are mandatory products in the rubber tire industry. They are used as protective lining to prevent contamination of the rubber. This pattern geometry (2D and 3D) prevents the rubber from sticking to each other. The film is desired to be homogeneous, precise in thickness, and have sufficient mechanical strength. The speed and the temperature of the pattern-forming machine are among the factors that determine this relationship between the thickness of the film and its mechanical properties for sustainable quality production. In this study, the effect of the speed and the temperature of the pattern machine on the pattern thickness during the creation of the pyramid-shaped pattern applied on a 100 ± 5 μm thick polyethylene film were examined. Four different machine speeds (24, 26, 28, and 30 m/min) and three different temperatures (100, 110, and 120 °C) were studied as variables. The impact of parameters on film thicknesses and tensile properties was assessed. Film thickness varied from ~375 to ~340 μm at higher machine speed, strength-at-break values decreased from 28 to 22 MPa, and elongation values dropped from 575 to 437% with the increment in speed. On the other hand, at higher temperatures, thickness rose from ~360 to ~390 μm, and elongation values reduced from 440 to 410%. Within the scope of the experimental studies, it was observed that the film thickness changes and the mechanical properties can be controlled by changing the line speed or process temperature.

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Graphical abstract
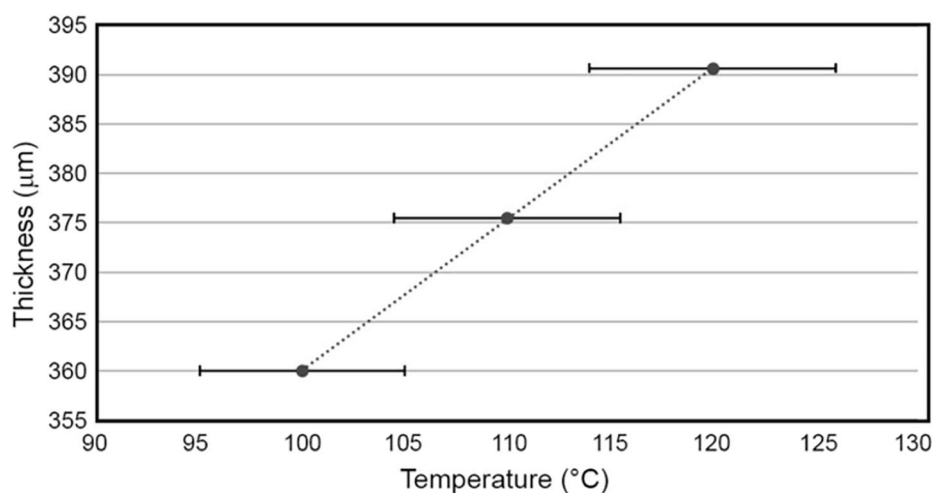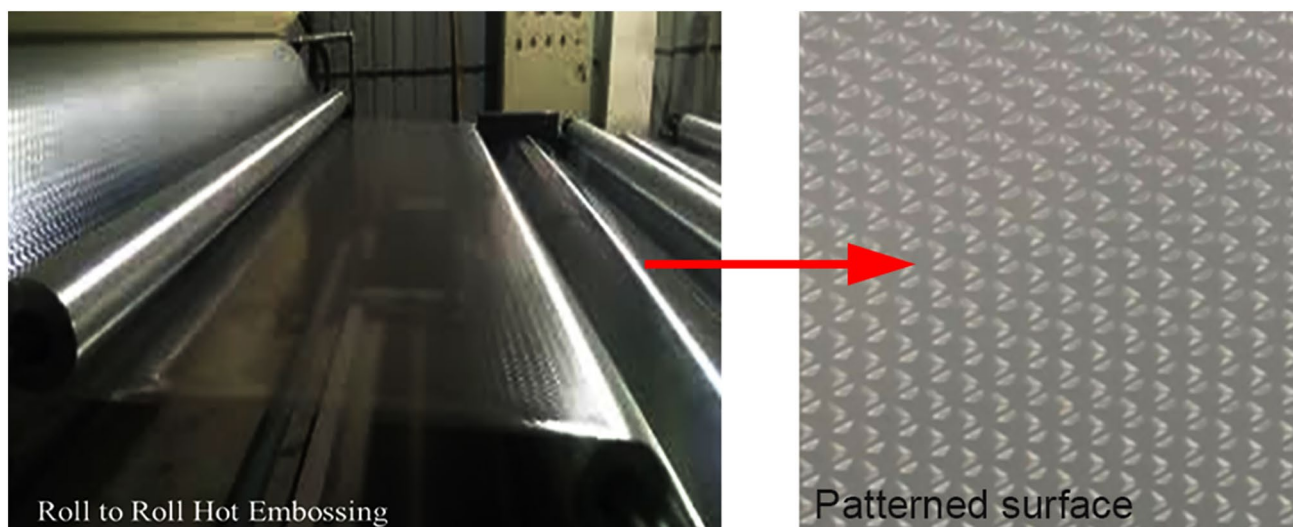
Introduction

Polymer-based materials offer a wide range of physical and chemical properties and have the advantages of low cost and good processability for mass production [1–3]. Mostly neat commercial polymers are used in the industry to get easy-processible production. On the other hand, to obtain enhanced mechanical, thermal, etc., properties fiber-reinforced polymer composites are widely used [4, 5]. Hot embossing is the process of pressing a molding tool into a semi-finished polymer. This technique has the advantage of producing surface micropatterns with high quality and precision [6–8]. Micro-hot embossing is a direct and cost-effective approach to the fabrication of replicas on polymer thin films. There are three modes in the micro-hot embossing process: plate-to-plate (P2P), roll-to-plate (R2P), and roll-to-roll (R2R) [9]. Characterized by two rollers with polymer film in between, roll-to-roll hot embossing is continuous, has high throughput, and is the most efficient mode. Generally, the temperature is set above the glass transition temperature (Tg) in the roll-to-roll hot embossing process. However, due to the short embossing time and cooling time in the R2R hot embossing process, there is insufficient polymer deformation for filling mold and resilience, leading to the formation of defects and a low level of thickness. Roller temperature, roller speed, and applied force are three essential parameters determining the completion and uniformity of microstructure replication [10]. Gon et al. studied the hydrophobicity of PTFE/graphite composites produced by the hot-embossed technique. It was found that process conditions, such as

temperature and time, affected the filling mechanism of the polymer. As a result of this, the hydrophobicity of the composite changed [11]. Bouchard et al. investigated the production of micro-structured polymethylmethacrylate (PMMA) cost-effectively for white light source through plate-to-plate hot embossing. According to confocal, scanning electron microscopy combined with photospectroscopy results, the strategy was found efficient compared to conventional methods, such as direct laser writing, multiphoton polymerization, and lithography [12]. Besides, polyurethane-based vinyl ester matrixes were examined as an alternative for polymers [13]. Among conventional polymers, low-density polyethylene (LDPE) is the first commercially introduced in the polyethylene (PE) family. LDPE is a polymer with many advantageous properties (semi-rigid, transparent, gas-tight, chemical resistant, low water absorption, easy to form, and economical production capability). LDPE is the key player in many food packaging markets, especially those requiring high clarity. Moreover, LDPE has a low melting range and is relatively easy to process among the various PE types [14].

In the literature, Deshmukh et al. studied the production of superhydrophobic surfaces with a dimensional change in surface topography on different polymer substrates including polyethylene by induction-aided hot embossing technique [15]. Jiang et al. investigated the usability of polyethylene microstructured film as a self-cleaning and optical selective layer in radiative coolers. The films were produced through a scalable hot embossing lithography and the results revealed that passive daytime radiative cooling materials could be used in practical applications with minimal maintenance need [16]. In another study, hot embossing technique was used with ultra-high molecular weight polyethylene (UHMWPE) powders for obtaining high aspect ratio square prism microstructures [17].

In the study of Chang et al., a microstructured pattern (microcylinder array) on different polymer substrates (polycarbonate, polymethyl methacrylate, and low-density polyethylene) was studied through a novel multilayered hot embossing process. A new facility, incorporating both graphene-based heating and multilayered hot embossing, was devised and executed [18].

Embossed polyethylene materials are generally used in the tire rubber industry for safe handling so that other processes do not affect the rubber. Embossed patterns provide easy separation of polyethylene film from rubber. The thicknesses in the patterns vary according to the materials used in rubber production and production parameters. In summary, this application improves the rubber–rubber interfacial separation with the help of surface patterns produced by the micro replica technique. The effects of process parameters on mechanical properties and film thickness in R2R micro-hot embossing have not been investigated in studies. This study aims to investigate the effects of parametric process

variables on the thickness of the LDPE layer and the pyramid patterning ability of the films for the first time in the literature.

Experimental

Materials

In this study, PE film with a high LDPE (Petkim, G03-5) ratio and a flat thickness value of 100 ± 5 μm was used.

Embossing process

Film embossing is a mechanical replica process in which a flat film is transformed into an embossed product. By transferring the pattern designed on the rollers to the polymer film, 2- or 3-dimensional functional patterns can be obtained. Depending on the pattern design, surfaces that serve the purposes of non-stickiness, easy separation, and interface cleaning attract the attention of the tire rubber industry. During the process, thermal and stress fields are applied to the polymer, causing changes in the microstructure and physical dimensions of the material [1–3]. The thermal treatment of the polymeric film was shown to be the most significant factor in the process. The important aspects influencing the thermal treatment include the radiation heater temperature, preheat roll temperature, embossing pressure, line speed, and film thickness. The presented master structures having height and width in the micrometer range could in principle be replicated by the roll-to-roll embossing process. Nevertheless, the maximum depth of the embossed holes was only about 70% of the master height [19]. As seen in Fig. 1, the pattern is formed on the film by passing the extruded polymer sheet between the rollers containing the pattern under the influence of a certain critical temperature and pressure.
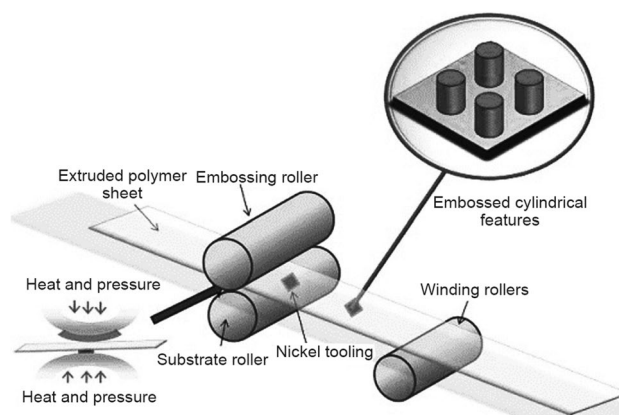
Fig. 1 Schematic representation of the hot embossing process (R2R) [26]

In the R2R hot embossing system, temperature uniformity on the roll surface is very important for better quality of patterns, because even heating is needed to get even pattern depth from identical stiffness of the substrate [20, 21]. The embossing roll serves not only to impress a pattern into the plastic film but also to cool the film to a temperature at which the embossed pattern remains fixed, i.e., below the softening point of the resin. The design of the embossing roll is very similar to that of the preheating roll. The embossing roll is cooled by the circulation of water through the core. The circulating water is usually cooled by a cooling tower. As a result of seasonal variations, the temperature of the cooling water can vary by as much as 15 °C [22]. The pressure distribution in the nip formed by the contact of the two embossing rolls is determined by the embossing pressure metal roll mechanical properties rubber layer mechanical properties and roll geometries. The two embossing rolls are pressed together using two pneumatic cylinders to control the position of the embossing roll. Air pressures commonly encountered in the industry range up to 2.4 MPa resulting in a force of 9.7 kN, assuming a cylinder head diameter of 15 cm [22].

The following machining conditions have been proposed for hot embossing of polymer parts with micron-sized properties [23–25]: (1) the thermal cycle (defined as the temperature range between embossing and de-embossing temperatures) should be 25–40 °C in order to minimize the thermally induced stresses, and (2) the embossing pressure should be around 0.5–2 kN/cm^2.

The film used in the study was produced flat in a 3-layer (3 extrusion screw) (Fig. 2) blown line with an automatic dosing system. Then, the study was carried out with the R2R (roll to roll) hot embossing machine. The machine speed started at 20 m/min and increased gradually to 34 m/min. The variation of the thickness on the pyramid pattern and the mechanical variations in the film structure were analyzed accordingly. Cylinder temperatures and pressure were kept constant. The aim here is to better understand machine speed, one of the process parameters that strongly influence the result of the hot embossing process. The machine speed and the pressure were kept constant in the second experimental study. The temperature was gradually increased. The variation of the thickness on the pyramid pattern and the mechanical changes in the film structure were analyzed. Blown extrusion and roll-to-roll hot embossing process are illustrated in Fig. 2.

The most preferred pattern type in the rubber industry is the pyramid pattern with different geometric dimensions [22]. In this study, the pyramid pattern was applied to the flat film of 100 μm thickness with the help of an embossing roller and rubber roller in the hot embossing process. It is expected that the final film thickness will vary between 320 and 400 μm depending on the pyramid pattern formed and the machine speed.

Measurements and characterizations

Test samples of 2.5 cm × 15 cm (Fig. 3) sections were taken from the film coil surface for each machine's speed parameters and temperature parameters. The thickness of the samples was measured with a bearing micrometer at 2 cm intervals, and the sample weight was checked with a precision balance. The flat thickness value (ASTM D6988) of the film was calculated using Eq. 1. Density is accepted as 0.95 g/cm^3.

$$\text{Film thickness} = \frac{10 \times \text{film weight}}{\text{surface area} \times \text{film density}} \tag{1}$$

Strength tests were carried out based on the ASTM D 882 standard, with a tensile speed of 500 mm/min. Zwick brand tensile-rupture device was used for the tests, and the software of the device gave the following tests by automatically calculating the standard deviations of the drawn samples.
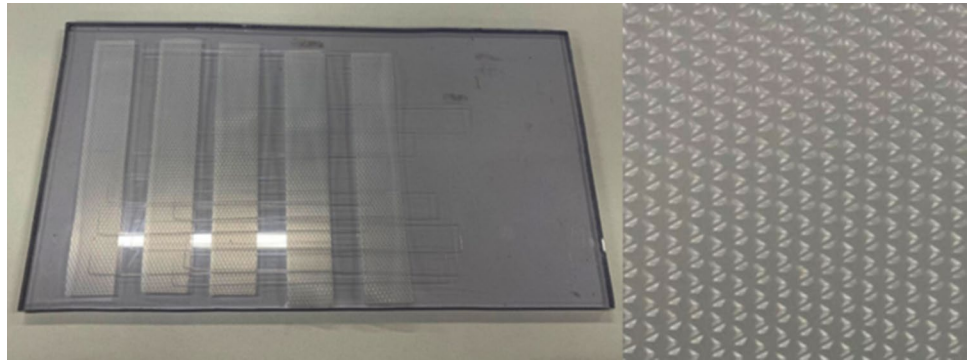
Results and discussion

### Effect of machine speed parameters on thickness

A pyramid pattern was applied to the polyethylene film, which was produced flat with a thickness of 100 μm by

Fig. 2 Schematic of the blown extrusion [22] and roll-to-roll hot embossing process
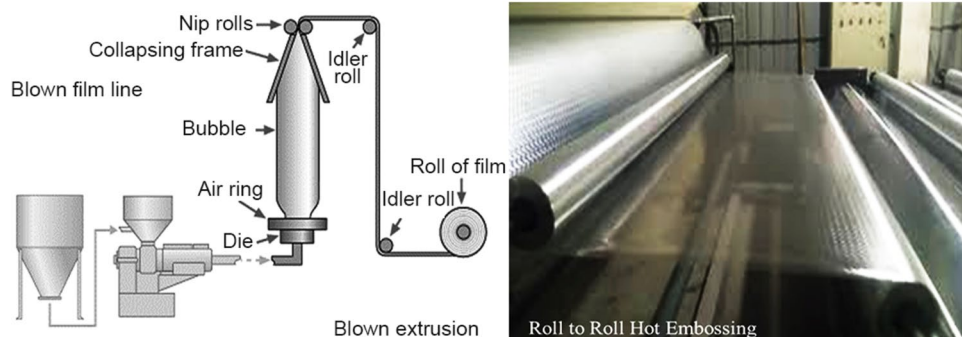
Fig. 3 Test samples produced with different machine speeds

Table 1 Average thickness change depending on machine speed

Machine speed (m/min)	Thickness measurement results of film samples								Sample weight (g)	1 m^2 weight (g)	Flat thick (μm)
	1	2	3	4	5	6	7	AVG			
24	378	373	370	370	374	375	373	373.29	0.3630	96.80	0.102
	379	379	373	376	372	377	375	375.86	0.3593	95.81	0.101
26	367	371	366	360	363	362	361	364.29	0.3576	95.36	0.101
	364	363	367	363	363	366	366	364.57	0.3620	96.53	0.102
28	356	358	360	365	365	362	366	361.71	0.3735	99.60	0.105
	356	360	363	365	361	362	362	361.29	0.3675	98.00	0.103
30	336	337	341	343	345	344	340	340.86	0.3637	96.99	0.102
	343	344	336	349	338	346	341	342.42	0.3644	97.17	0.102
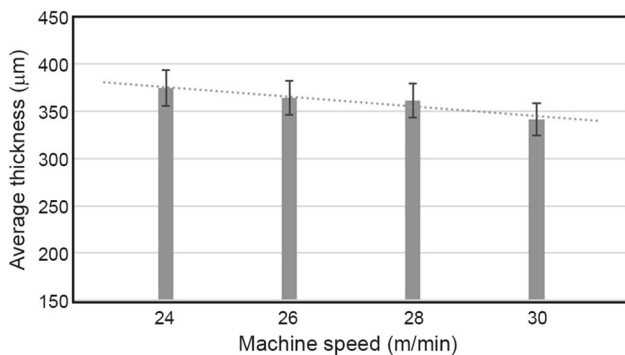
**Fig. 4** Film thickness changes by machine speed

the same operators in the same blow machine, in the same embossing machine. Cylinder temperatures and pressures were kept constant. While the pyramid pattern thickness applied at a speed of 24 m/min is 375 μm on average, the pattern thickness is in the band of 340 μm at a speed of 30 m/min. There is an average of 30 μm thickness variation between the speed of 24 m/min and the machine speed of 30 m/min. Flat thickness calculation results are given in Table 1. It was observed that the film thickness decreased as the machine speed increased (Fig. 4). It was observed that the thickness applied after the pyramid pattern decreased as the machine speed increased. When the speed of the

machine increased, the response of the polymer chains was significantly affected. As the rate of speed rises, the mobility of the polymer molecules decreases, causing the material to become less flexible. This results the change in the film properties of the polymer [27–29].

Test samples are produced at different line speeds and the resulting measurement data of mechanical properties: the average yield strength values were measured as 12.3 MPa, the breaking strength was measured as 24.8 MPa, and the elongation value was 505% on average. Tests were performed on 2 different sets of sample groups (6 samples in total) and their averages were taken. The machine speed affects the mechanical properties and elongation ratio according to Figs. 5 and 6, and Table 2. Especially in the elongation ratios and breaking strengths, significant differences are observed (Fig. 6). These differences could be occurred because of the molecular mobility changes of the polymer chains as mentioned above.

Effect of temperature parameters on film thicknesses

A pyramid pattern was applied to the polyethylene film, which was produced flat with a thickness of 100 microns by the same operators in the same blow machine, in the same embossing machine. Machine speed and pressure

Fig. 5 Strength of film change by machine speed
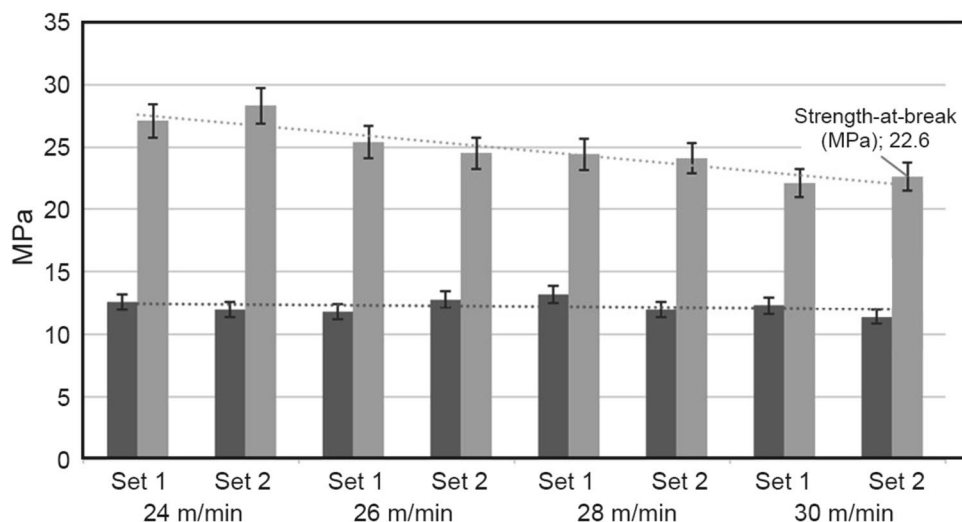
Fig. 6 Film elongation ratio change by machine speed
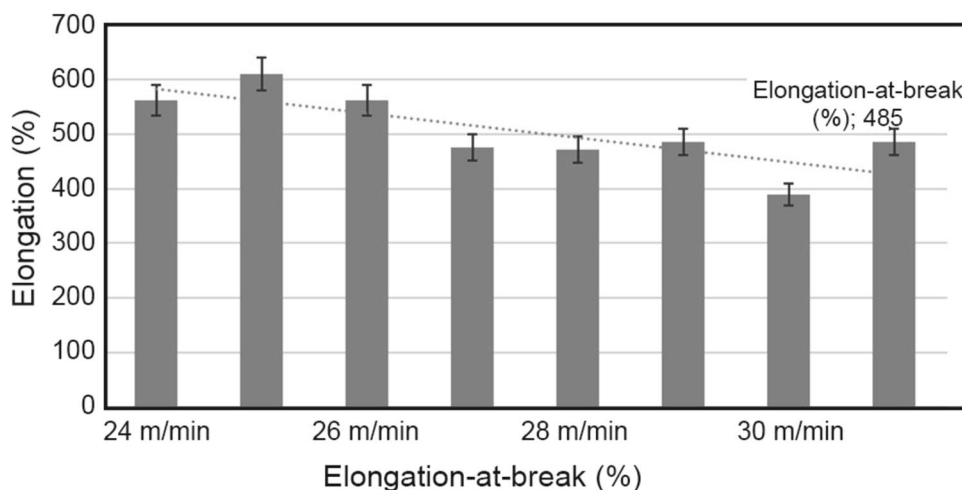
Table 2 Results of tensile test measurements and comparative graphics

Tests	24 m/min		26 m/min		28 m/min		30 m/min	
Set number of samples	Set 1	Set 2	Set 1	Set 2	Set 1	Set 2	Set 1	Set 2
Strength-at-yield (MPa)	12.6	12	11.8	12.8	13.2	12	12.3	11.4
Strength-at-break (MPa)	27.1	28.3	25.4	24.5	24.4	24.1	22.1	22.6
Elongation-at-break (%)	562.1	609.6	561.5	474.9	470.7	485.2	388.9	485.0

were kept constant (Fig. 7). Pyramid pattern thickness at 100 °C is 360 μm on average, while 120 °C is 390 μm. There is an average thickness change of 30 μm between temperatures of 100–120 °C. Flat thickness calculation results are given in Table 3. It was observed that the thickness applied after the pyramid pattern rose as the temperature increased. The properties of polymers are greatly influenced by temperature. As the temperature rises, the molecular chains become more flexible and can deform in the direction of applied stress. Molecular motions are constrained by physical bonds, such as those caused by

Van der Waals, hydrogen, or dipole–dipole interactions [27–29].

The mechanical properties of the test films produced at different temperatures are shown in Figs. 8 and 9. The test results were obtained depending on the temperature variable: the average yield strength values were measured as 13.3 MPa. The breaking strength was measured 33.9 Mpa, and the elongation value was 427% on average. These differences could be occurred because of the molecular mobility changes of the polymer chains due to physical bonds as discussed before.
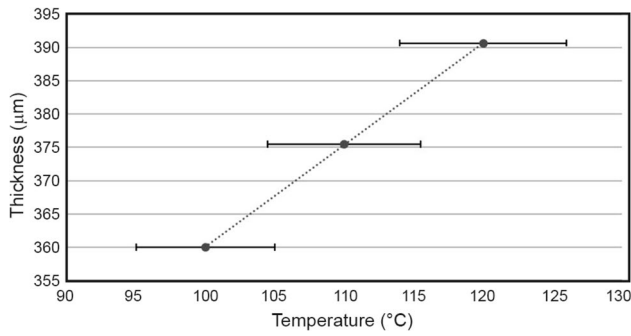
Fig. 7 Thickness change by temperature

Conclusion

Patterned (embossed) polyethylene films are mandatory products in the rubber tire industry. To maintain the purity of the rubber, these embossed films are utilized as a protective lining during transportation and storage in tire production, preventing any foreign elements from causing contamination. The two- and three-dimensional pattern geometry keeps the rubber from adhering to one another. It is intended that this film possesses adequate mechanical strength and a specific, uniform thickness. Process parameters in the pattern-forming machines especially machine speed and temperature are the most important factors to specify the affair between the film thickness and mechanical properties. In this study, the changes of the pyramid pattern thickness and mechanical properties were investigated according to machine speed and temperature parameters in the R2R hot embossing process. It was observed that film thickness varied from ~375 to ~340 μm at higher machine speed, and strength-at-break values decreased from 28 to 22 MPa, and elongation values dropped from 575 to 437% with the increment in speed. A correlation was established for pattern thickness with the mechanical test results. Therefore, the mechanical properties vary according to the thickness of the film. On the other hand, at higher temperature thickness rose from ~360 to ~390 μm, and elongation values reduced from 440 to 410%. This reduction could be reviewed because of the molecular chain construction and deformation under the applied stress. The film can be produced following the relevant standards based on temperature and speed

Table 3 Thickness change depending on the temperature

Temperature (°C)	Thickness (μm)								Sample weight (g)	1 m^2 Weight (g)	Flat thick (μm)
	1	2	3	4	5	6	7	AVG			
100	340	338	339	340	341	339	340	339.57	0.3974	105.97	0.111
	380	383	381	379	378	380	382	380.42	0.3950	105.33	0.110
110	350	354	355	352	349	347	349	350.85	0.3990	106.40	0.112
	400	402	403	399	397	398	402	400.14	0.3502	93.39	0.098
120	370	374	375	371	368	364	374	370.85	0.3730	99.47	0.104
	410	412	415	410	409	407	411	410.57	0.3761	100.29	0.105

Fig. 8 Strength of the film change by temperature
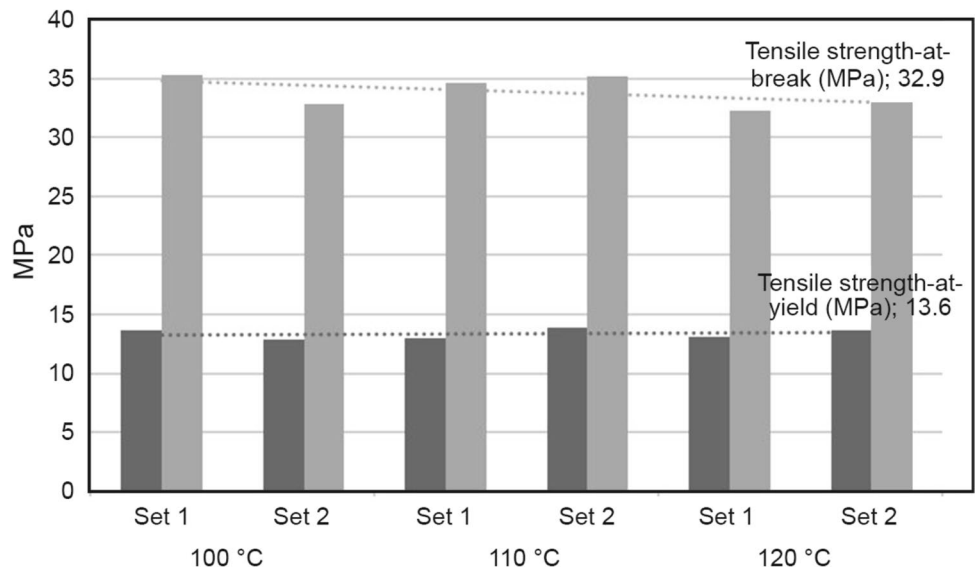
Fig. 9 Elongation of film change by temperature
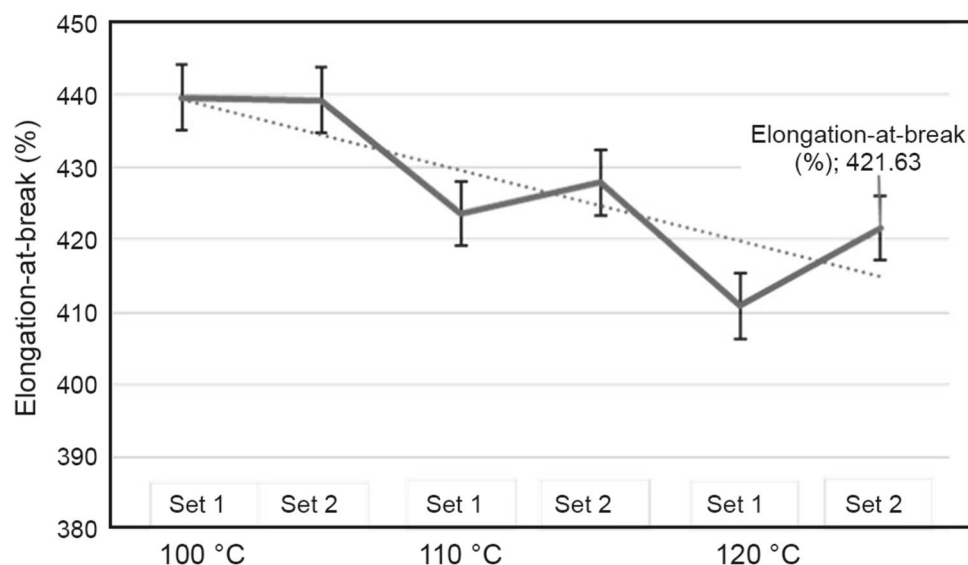
optimization. Different formulations of rubber are used in the rubber industry, especially in different groups of summer and winter tires. In the face of increasing raw material energy costs, recycled rubber products can be used in rubber compound mixtures. Sealing elements, connection hoses, industrial textile products (work gloves, work shoes, etc.) sectoral applications are observed. In addition, there is a significant market potential in future applications, especially in vibration damping and earthquake isolators. Therefore, this work is promising especially for the rubber sector.

Data availability The dataset generated and analyzed during the present study "https://doi.org/10.1007/s13726-024-01293-x" is available from the corresponding author upon reasonable request. In this study, systematic experimental studies within the scope of an R&D study for an industrial application were carried out and validated both on a laboratory scale and on an industrial scale, and the results of the experiments were discussed scientifically and then formatted into an article suitable for your journal.

Declarations

Conflict of interest No conflict of interest was declared by the authors.

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