

## Shear-Induced Molecular Orientation of Compression Moulded PE-HD Sheets

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### ABSTRACT

The article presents experimental results on orientation of polymer macromolecules in compression moulded high density polyethylene sheets. Properties anisotropy of thermoplastic films or sheets, that are usually formed in the extrusion process, causes deformation of thermoformed products and is a serious technological problem. One of the possible solutions of this problem is application of compression moulded sheets. The paper presents the results of tests of selected strength characteristics of compression moulded PE-HD sheets. A static tensile test was performed and Charpy impact strength was determined. Additionally Chrysler's orientation test was executed. For comparison the same experiments were performed on extruded sheets. Samples were cut in directions perpendicular and parallel to the direction of polymer flow in pressing and extrusion processes. The obtained results indicate that the compression moulding technique allows the production of sheets that do not exhibit statistically significant anisotropy of the tested strength properties.

**Keywords:** PE-HD sheets, anisotropy, molecular orientation, mechanical properties, compression moulding.

### INTRODUCTION

The proper selection and use of polymers in the production of various types of products requires knowledge of the characteristics of a given material and their processing technologies. Polymeric materials are a very large and important group of materials that are currently used in almost all areas of life. Scientists subject this group of materials to constant modifications in order to improve their selected properties, be it mechanical, thermal, electrical, UV resistance as well as flame retardance by adding polymer flame retardants, ultimately these treatments also change the structure of materials [1–3]. In the thermoforming process, depending on the products requirements (mechanical strength, dimensional stability, chemical resistance, resistance to atmospheric aging, transparency, depth of thermoforming, acceptable price), sheets as semi-finished products made of various thermoplastics or their

compounds are used [4]. Apart from the composition the structure of the polymeric matrix is very important. The structure can be influenced, apart from the introduced additives, by the physical processes occurring during processing technology. One of the most important phenomena observed during processing is shear-induced orientation of macromolecules in direction of flow. The effect of macromolecules orientation is anisotropy of properties along and across the direction of flow. This structure orientation can be beneficial or troublesome. In production of fibers, bottles, films and tapes shear-induced orientation is very beneficial and is intentionally induced during manufacture. But in these technologies orientation is induced mainly by drawing with little share of shearing. In the opposite, the main problem in using extruded PE-HD sheets for thermoforming is the anisotropy of their properties [5]. Apart from the orientation, other structure characteristics of the extruded materials (e.g. conformation and morphology of the

macromolecules, content of the crystalline phase, etc.), which are determined by the manufacturing process, influence suitability to thermoforming [6]. In many extruded parts orientation can cause post-extrusion problems from non-uniform shrinkage and warpage [7,9]. In the case of anisotropic materials, different mechanical properties should be expected depending on the direction of testing in relation orientation direction [10]. The arrangement of the structure along the orientation direction results in higher strength but lower elongation at break. In the direction perpendicular to the orientation, the elongation will be greater, but the strength less. This phenomenon is very well known in extruded plates [8, 11–24]. Orientation results in thermoformed products distortion, which largely affects the quality of ready products made of extruded plates. The solution of this problem may be the use of compression moulded sheets [25]. After the pressing process, the macromolecules are oriented in different directions because of multi-directional flow. Lower orientation levels are also expected due to lower shear rates. This ought to ensure more even properties distribution and more even quality and strength characteristics, regardless of the direction of thermoforming and the load during exploitation. Additionally, due to the high pressure and the high pressing temperature, the structure of these plates is tight. Because of this, the process of production of plates by compression moulding allows to obtain the better characteristics of a product with a disordered structure. This pressing technology can produce sheets of various thicknesses, starting from 4 mm, and they are used not only for thermoformed products but in many various industries [26].

The research part of the paper describes the preparation of samples and the test program. Then, results of mechanical tests are presented: tensile strength and impact strength of high-density polyethylene (PE-HD) sheets pressed and, for comparative purposes, also extruded. The research was aimed at determining the anisotropy of the material in the area of plates produced with these two technologies. The results of conducted tests ought to answer the question whether pressed PE-HD sheets show statistically significant differences of properties in direction of flow and perpendicular to flow as a result of macromolecules orientation. The second purpose of the research is comparison of homogeneity of compression moulded sheets and extruded sheets.

## EXPERIMENTAL

### Materials

Pressed sheets were manufactured by the company “SZAGRU”, Studzienice, Poland. High-density polyethylene, Finathene®6002 grade 100, produced by ATOFINA S.A was applied. Table 1 shows the basic properties of the tested PE-HD polyethylene based on the manufacturer’s technical data sheet [27]. The sheets were made in three stages process. First, polymer was extruded into the accumulator using a W80/30Sz single-screw extruder (Institute of Plastics Processing in Toruń, Poland). The barrel temperatures in heating zones of the cylinder were 182, 204, 208, 215°C. The temperature of extrusion die was 185°C. Next the polymer melt was extruded into the mould and heated to a temperature of 200°C. At the next stage the melt was pressed under pressure of 30MPa for 20 minutes and in the last stage it was cooled in the mould and ejected. The mould was mounted on the PHM hydraulic press (Ponar Żywiec, Poland). The sheets with dimensions 1000x1000x6mm were tested. Tensile strength and impact strength test samples were prepared with dimensions in accordance with the applicable standards. Figures 1 and 2 show the location of the cut samples on the surface of the pressed plates. In order to compare the properties of pressed and extruded materials, samples of extruded plates were also prepared for testing. The phenomenon of property anisotropy due to orientation of extruded sheets has already been widely described in the literature, so these sheets were tested only to compare the values differences. Two extruded PE-HD grade 100 sheets of two different manufacturers were prepared for the tests: AGRU-FRANK Poland Ltd. and SIMONA Poland Ltd. Extruded sheets with dimensions 500x500x6 mm were applied. The arrangement of the cut samples is shown in Fig. 2. Out of each sheet, samples of the dumbbell shape and a flat bar-shaped samples were cut using a punch. Before the strength tests all samples were subjected to air-conditioning to remove stresses caused by machining. Temperature and time of air-conditioning were selected not too high to avoid reduction of macromolecules orientation. Specimens were conditioning at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 10\%$  relative humidity for not less than 40 h prior to test in accordance with ISO 291. Special test, called “Chrysler’s orientation test”,

determining the degree of orientation was also carried out, both for extruded and pressed sheets. The test is described in details in the literature [5]. The arrangement of samples for this test is shown in Fig. 3.

## Methodology

### Tensile strength test

For the tensile strength tests 12 samples from the first P1 plate and 15 samples from the second P2 plate were prepared. A total of 27 dumbbell-shaped samples cut from the pressed sheets and 10 samples (5 in the longitudinal flow direction and 5 in the perpendicular to flow direction) were prepared from each extrusion sheet. Subsequently, the samples were tested using HECKERT FPZ 100/1 tensile strength testing machine. The force range was 2 kN and the testing speed was 2 mm / min. Tests were carried out in accordance with EN ISO 527-2: 2012 [28] at room temperature.

### Charpy impact test

For the impact tests 15 samples from the first pressed plate (P1) and 12 samples from the second plate (P2) were prepared. A total of 27 notched bar-shaped specimens 120x10x6 mm were prepared from the pressed plates (Fig. 4) and 20 specimens (10 in the longitudinal, flow direction and 10 in the perpendicular direction) from each extruded plate. Tests were carried out at room temperature in accordance with the Polish Standard PN-EN ISO 179-1: 2010 [29] with the use of a Charpy HIT25P pendulum hammer produced by Zwick Roell (Zwick GmbH & Co.KG, Ulm, Germany).

### Chrystler's orientation test

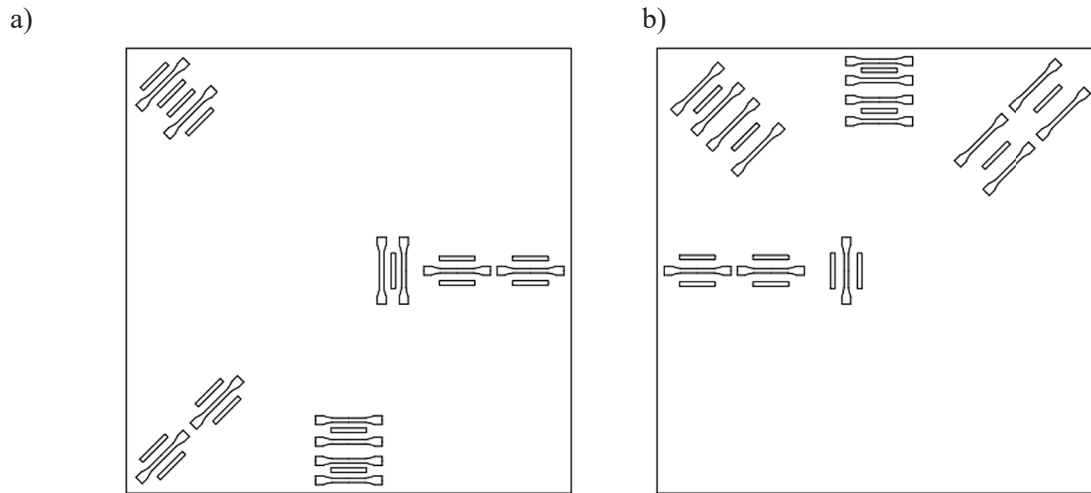
Orientation tests were performed according to the procedure described in the literature [5] and commonly known as Chrystler's orientation test. For the purpose of these tests, 3 samples were cut in two direction of material flow in given manufacturing technology and 3 samples perpendicularly to the flow direction and 3 bias to this direction. The samples were 50x180 mm in size. A series of samples was placed in a vacuum oven, then heated to the temperature of 130°C (this is the standard processing temperature of polyethylene) and kept at this temperature for 15–30 minutes (the heating time depended on the thickness of the tested sample). After the specified annealing time, the samples were removed from the dryer. After that, the samples are cooled and the deformation effects of the samples were optically assessed (Fig. 5), and the degree of deformation (as the height of distorted sample) was also measured to determine the change of shape due to warpage. The degree of dimensional and shape change indicates the comparative degree of orientation in that area or direction.

## RESULTS AND ANALYSIS

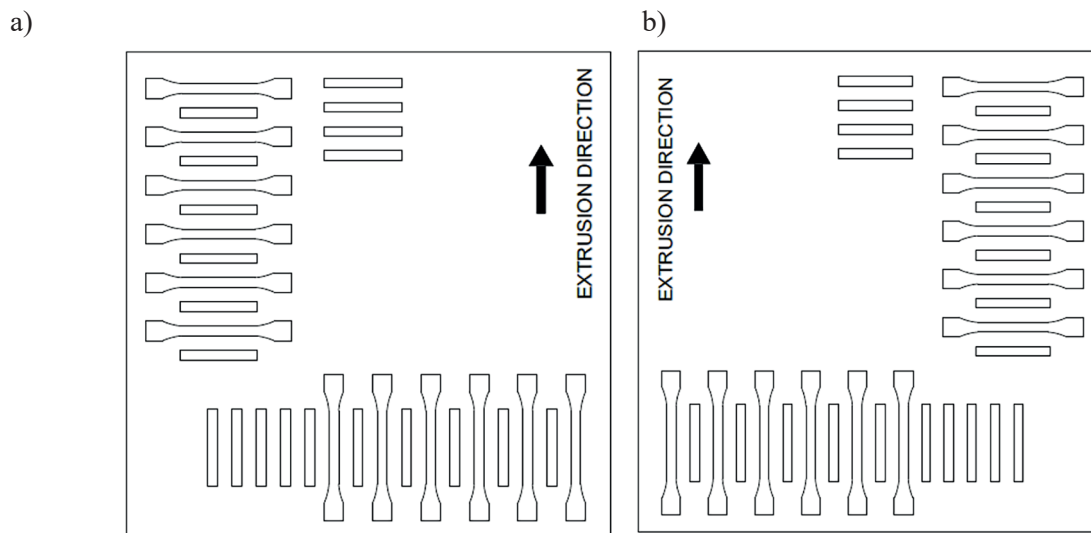
Shear-induced macromolecular orientation will be determined indirectly by evaluation of anisotropy of strength properties in flow direction and direction perpendicular to the flow. Because of this the most important will be the differences of tested properties in these two directions. Additionally results of "Chrystler,s" orientation test will be analysed.

**Table 1.** Properties of high-density polyethylene Finathene®6002 based on the ATOFINA S.A manufacturer's technical data sheet [27]

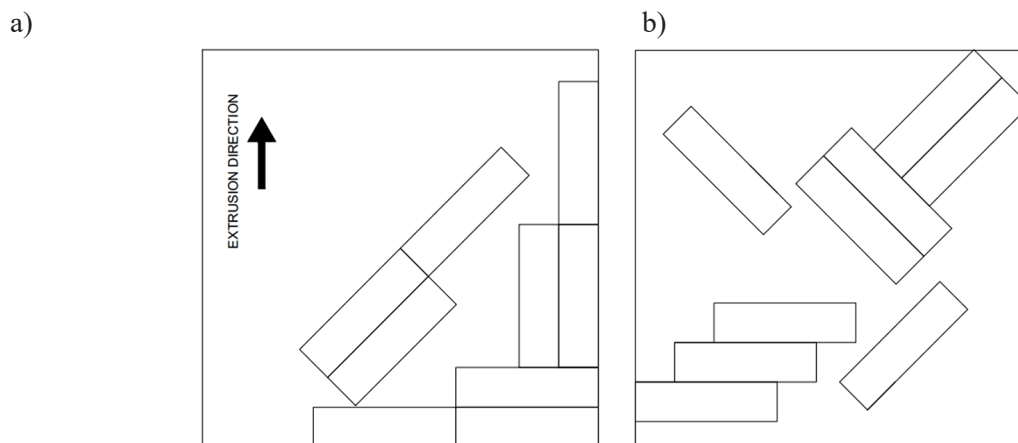
Properties	Value
Density at 23°C [g/cm <sup>3</sup> ]	0.959
MFR [g/10 min] T = 190 °C	21.6 kg
	5 kg
	2.16 kg
Vicat softening point [°C]	128
Tensile strength, yield [MPa]	28
Elongation at yield [%]	8
Tensile strength at break [MPa]	26
Elongation at break [%]	>700
Tensile impact strength [kJ/m <sup>2</sup> ], 23 °C	230



**Fig. 1.** Arrangement of the cut samples on the surface of the pressed sheets: the first P1 (a) and the second P2 (b)



**Fig. 2.** Arrangement of the cut samples on the surface of the extruded sheets: a) the first W1, b) and the second W2



**Fig. 3.** Arrangement of the samples for orientation tests: a) extruded sheet, b) pressed sheet



Fig. 4. Exemplary samples after Charpy impact strength test.

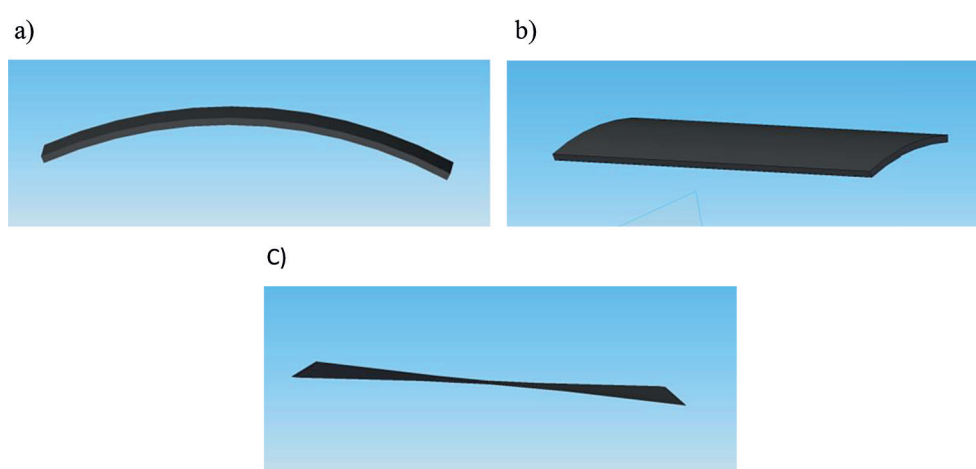


Fig. 5. Typical distortions observed after orientation test of samples cut: a) in extrusion direction, b) traverse to extrusion direction c) slant to extrusion direction

The STATISTICA computer software was used to perform statistical calculations of experimental results.

### Analysis of tensile strength results

Yield stress,  $Re$ , and strain at break,  $\varepsilon_x$ , were determined in tensile strength test. The results are presented in Fig. 6 and 7.

Tensile strength tests were carried out for two different pressed plates. Additionally, samples cut from extruded plates were tested (Fig. 6 and 7).

Obtained mean values of yield stress (Fig.6) show that in the case of pressed and extruded sheets very minor differences were observed for two directions of testing. Because differences were not very pronounced, statistical significance test was performed to verify whether observed mean values differences were statistically significant or not. For the accepted probability level  $p=0,05$  the

values of the arithmetic mean ( $\bar{x}$ ) and standard deviation (SD) were calculated. Next two values of the Student's t-statistics were determined,  $t_0$  for tested samples and  $t_{\alpha, n_1+n_2-2}$  – critical value of Student's t-statistic. In some level of simplification it may be accepted that when  $t_0 < t_{\alpha, n_1+n_2-2}$  the test indicates that mean values differences are not statistically significant [30].

Results of the significance tests for mean values of tensile yield stresses are shown in Table 2.

The test shows that for the accepted probability level there are no significant differences between the average values of the tensile stress at the yield point in both tested directions in the case of extruded and pressed plates. It can be concluded that differences in yield stresses are alone not a good measure of degree of shear-induced macromolecular orientation. Much more research is needed to confirm this conclusion.



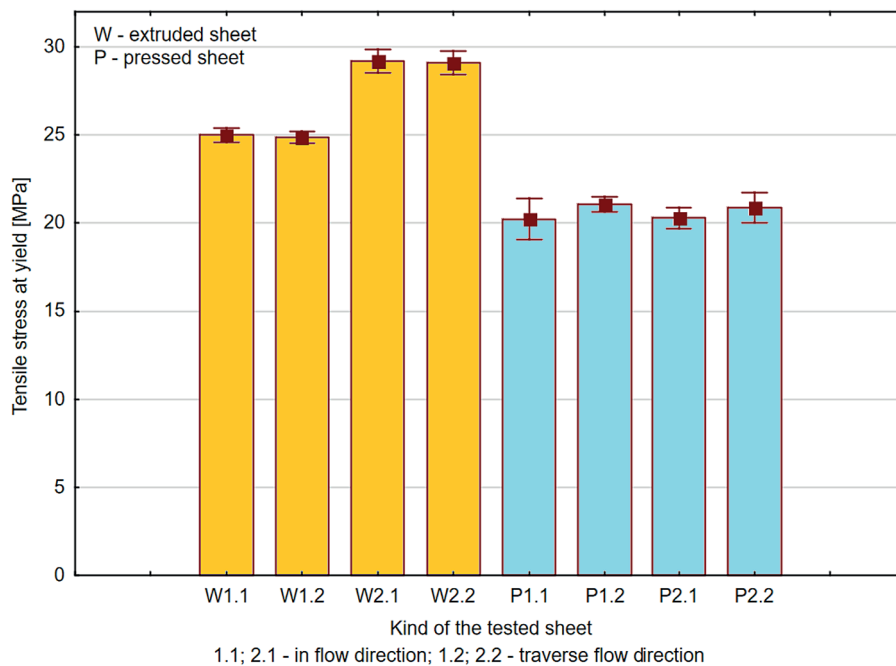


Fig. 6. Comparison of the tensile stress at yield point depending on the sheet production technology and the direction of testing

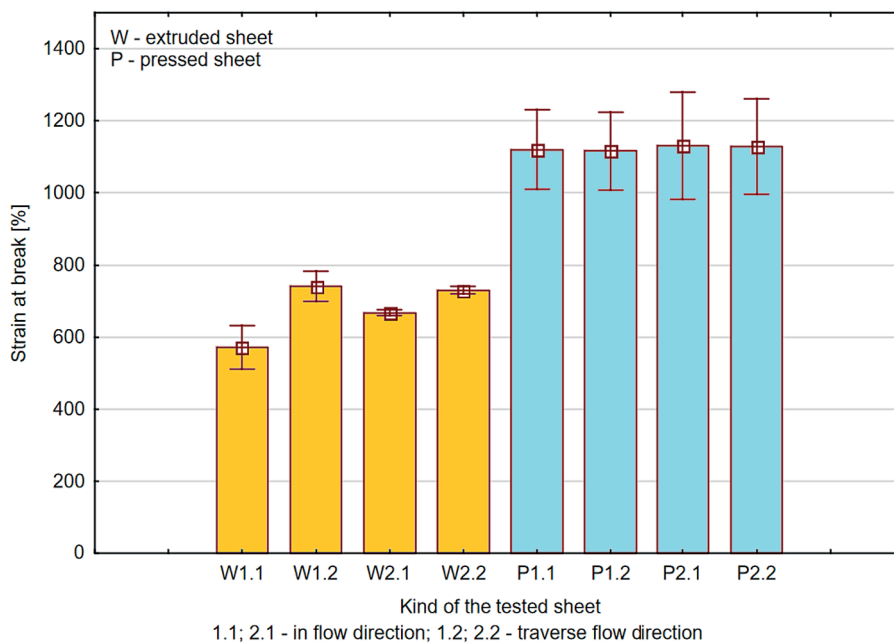


Fig. 7. Comparison of the strain at break depending on the sheet production technology and the direction of testing

Table 2. Significance test of the two mean tensile stresses at the yield point for the probability level  $p = 0.05$

Parameters	W1.1	W1.2	W2.1	W2.2	P1.1	P1.2	P2.1	P2.2
$\bar{x}$	24.99	24.89	29.21	29.13	20.23	21.08	20.29	20.87
SD	0.38	0.34	0.66	0.67	1.16	0.42	0.60	0.85
$t_0$	0.4196		0.1969		1.5222		1.3469	
$t_{0; n_1 + n_2 - 2}$	2.2281		2.2281		2.2281		2.1604	

Dissimilar results were obtained in the case of elongation at break (Fig. 7). Differences in mean values of elongation of pressed test pieces were very small but of extruded were much more pronounced. As expected elongation in flow direction was smaller than in direction perpendicular to the flow. It is the effect of macromolecular orientation in the flow direction. To confirm the significance of measured mean values differences once statistical significance test was performed. Results are presented in Table 3.

The statistical test confirmed that there were significant differences in the relative elongation at break in the tested extruded plates, but as expected, there are no significant differences in the case of pressed plates.

### Impact test results

The results of impact tests of samples cut in the earlier described directions for compression moulded and extruded sheets are summarized and compared in Figure 8.

Similar results as in the case of elongation at break were obtained. Differences in mean values of impact strength of pressed test pieces were very small but of extruded were much more pronounced. Impact strength in flow direction was smaller than in direction perpendicular to the flow. It is the effect of stiffening of polymer due to macromolecular orientation in the flow direction. To confirm the significance observed mean values differences third statistical significance test was performed. Results are presented in Table 4.

The test shows that there are no significant differences between the average values of the notched impact strength in both tested directions in the case of pressed sheets, while there are significant differences in impact toughness in the tested extruded sheets. Again it demonstrates that shear-induced orientation of pressed sheets is significantly lower than orientation of extruded sheets.

### Orientation test results

The shapes of test pieces after thermal orientation test (“Chrystler’s test”) of extruded and pressed plates are shown in Fig. 9. It can be seen that pressed sheets deformed very little and it was not possible to measure their deformation according to earlier described procedure. Deformation of extruded sheets was measured and is presented in Table 5.

Samples cut from extruded sheets exhibit moderate orientation stresses. In the flow direction and in the perpendicular to flow direction they curl to a medium extent. The greatest deformations occurred in the case of samples cut slant and along the flow direction. On the other hand, the samples of pressed sheets did not deform, i.e. they did not show any stresses due to orientation.

### Summary of the analysis of the results

Significant differences in the anisotropy of the properties of extruded and pressed plates result from differences in the history of flows during their formation. In the extrusion process, orientation occurs mainly during the unidirectional flow of the plasticized material through the slit channel in the extrusion die. It is called shear-induced orientation. The second stage of orientation is due to the differences in the speed of the individual calender rolls causing drawing of the sheet. In manufacturing of sheets dedicated to thermoforming producers try to minimize the drawing of sheets in order to minimize molecular orientation. In the pressing process, the flow is directed in different directions, radially from the centre of the plate. In addition, after the plate is formed, while it is cooling in the mould at a temperature higher than the ambient temperature, stress relaxation occurs, reducing the effects of macromolecule orientation. Differences in shear rates during forming with both technologies are also important. Typically, in extrusion processes, the shear rates at the

**Table 3.** Significance test of the two mean relative elongation at break for the probability level  $p = 0.05$

Parameters	W1.1	W1.2	W2.1	W2.2	P1.1	P1.2	P2.1	P2.2
$\bar{x}$	571.46	741.44	667.6	726.6	1119.9	1116.2	1130.2	1129.4
SD	60.43	41.66	8.89	4.94	110.06	107.43	148.61	132.06
$t_0$	5.1783		12.9674		0.0547		0.0095	
$t_{\alpha; n_1 + n_2 - 2}$	2.2281		2.2281		2.2281		2.2281	

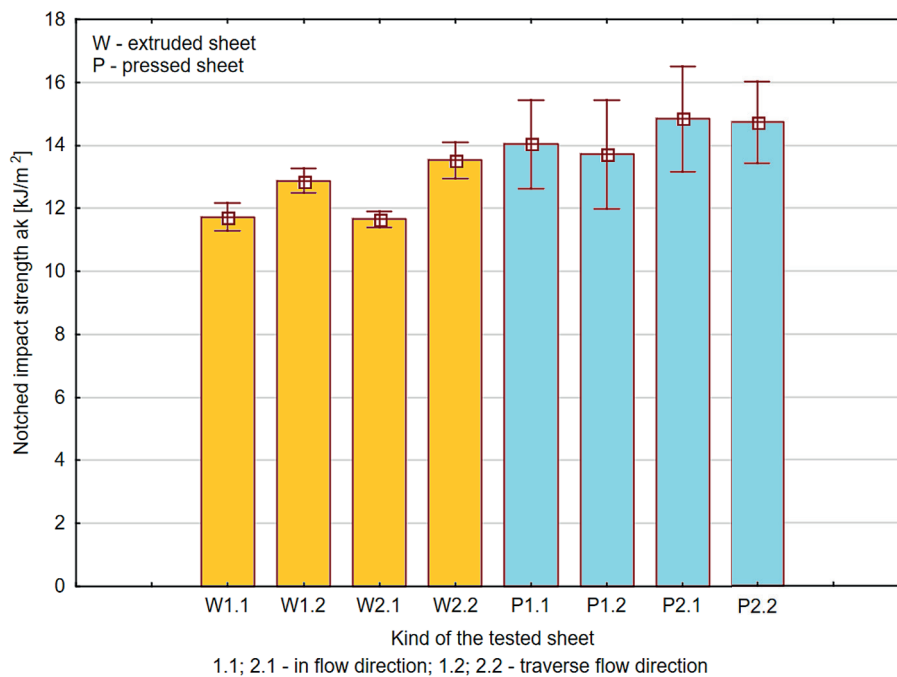


Fig. 8. Influence of manufacturing technology on the plates impact strength

die wall are in the range 10 to 1000 s<sup>-1</sup> [31]. The highest shear rates are obtained in film extrusion processes. In the processes of extrusion of plates with a thickness of 5mm to 10 mm, shear rates at walls of the head are usually higher than 100s<sup>-1</sup> but not higher than 300 s<sup>-1</sup>. It is not possible to evaluate precise values of shear rates applied in the production of plates used in the experiment because supplier does not provide information on extrusion rates. In the pressing process, the shear rates are most often in the range 0.001–10 s<sup>-1</sup> [31].

As in the process of producing the plates used in the tests, their formation begins with the production of a cylinder in the plastic accumulator and then the cylinder is pressed, the shear rates at the beginning of pressing are very low and increase to the maximum values in its final stage. There are no literature data on shear rates occurring in this kind of technology. In order to estimate the maximum shear values, the following calculations were made:

1. Assuming the principle of constant volume is fulfilled, it was calculated what is the increase in the radius of the pressed cylinder ( $\Delta R$ ) as a result of its height reduction ( $\Delta H$ ) (1).

$$\Delta H = R \left( \sqrt{\frac{H}{H - \Delta H}} - 1 \right) \quad (1)$$

where:  $H$  – initial height of the cylinder;  
 $R$  – the initial radius of the cylinder

2. Relating the height and radius increments to the time in which they occur, the speed of the radius increment (VR) was calculated on the basis of the known pressing speed (VH). Based on the analysis of the determined function of the velocity change in relation to the height of the pressed cylinder, it was found that the highest speed of the radius growth takes place at the final pressing moment.

Table 4. Significance test of two mean notched impact strength for a probability level of p = 0.05

ParameterS	W1.1	W1.2	W2.1	W2.2	P1.1	P1.2	P2.1	P2.2
$\bar{x}$	11.72	12.88	11.65	13.53	14.53	12.65	17.05	18.84
SD	0.44	0.39	0.25	0.57	1.74	1.86	5.61	4.01
$t_0$	5.9019		9.0344		1.8309		0.5806	
$t_{\alpha, n_1+n_2-2}$	2.1009		2.1009		2.1604		2.2281	



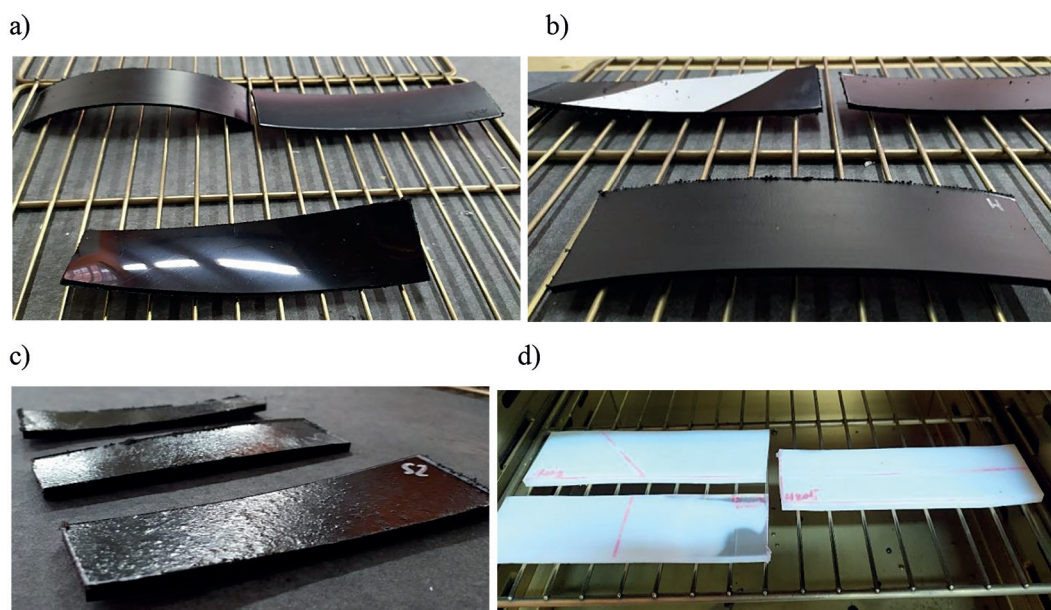


Fig. 9. Pictures of samples after orientation tests: a) W1, b) W2, c) P1, d) P2

3. Knowing the average flow rate of the material through the gap between the surfaces ( $V_R$ ), the shear rate on the walls of the mould was determined [32, 33] (2):

$$\dot{\gamma}_{wa} = \frac{6Q}{H^2 \cdot w} = \frac{6V_R \cdot w \cdot H}{H^2 \cdot w} = \frac{6V_R}{H} \quad (2)$$

where:  $Q$  – volumetric flow rate;  
 $w$  – the width of the slot through which the material flows;  
 $\dot{\gamma}_{wa}$  – uncorrected shear rate on the mould wall.

Taking into account the non-Newtonian nature of melted polymers, the corrected shear rate was determined (3):

$$\dot{\gamma}_w = \dot{\gamma}_{wa} \cdot \left( \frac{2n + 1}{3n} \right) \quad (3)$$

where:

$$n = \frac{d \ln \tau_w}{d \ln \dot{\gamma}_{wa}} \quad (4)$$

For PE, the value of  $n$  is in the range 0.4 to 0.7.

The process of pressing was observed in the company “Szagru” (Studzienice, Poland) and for PEHD sheets ( $n = 0.5$  was adopted) with a thickness of 6 mm to 15 mm, approximate values of the shear rate on the mould wall were obtained in the range from 8 to 17  $s^{-1}$ . These values are close to those reported in the literature for classical pressing technology [31] but, most importantly, are much lower than the values observed for the extrusion of sheets through slit dies. The differences in shear rates explain the observed significantly lower orientation effects for pressed sheets than for extruded sheets.

Table 5. The results of measurements of the deformation of extruded samples in the orientation test

Sheet W1 flow direction	Deformation [mm]						Average [mm]
	1	2	3	4	5	6	
In the direction	19.4	19.4	19.6	19.5	19.4	19.45	19.46
Traverse direction	5.9	6.63	6.81	5.92	6.75	6.4	6.40
Slant direction	33	32.82	33.51	33.67	32.92	33.48	33.23
Sheet W2 flow direction							
In the direction	18.85	18.82	18.69	19.19	18.85	18.8	18.87
Traverse direction	5.53	5.57	5.28	5.22	4.93	4.94	5.25
Slant direction	24.2	23.97	23.44	22.97	23.35	23.5	23.57

## CONCLUSIONS

Based on the research performed, the following conclusions can be drawn. The conducted tests have shown that the tested properties: tensile strength properties and impact strength of high-density polyethylene both extruded and pressed sheet are within the range of literature values. The obtained results show that the pressing technique allows for the production of sheets that do not show a statistically significant anisotropy of the tested strength properties. The sheets produced in compression moulding technology did not deform in the “Chrystler’s” orientation test what demonstrates that their shear-induced orientation was insignificant. Because of insignificant molecular orientation compression moulded sheets are more suitable for thermoforming than extruded one.

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