Processing Temperatures Influence of Three Types of Polyamide 6.6 Reinforced with Different Percentages of Fiber Glass on Some Mechanical Properties

GHEORGHE RADU EMIL MARIES*
University of Oradea, 1 Universităţii Str., 410087, Oradea, România

This paper analyses the influence of processing temperatures of polyamide 6.6 Grivory type HTV-3H1 noir 9205, polyamide 6.6 Grivory type HTV-45H1 noir 9205 and polyamide 6.6 Grivory type HTV-6H1 noir 9205 on some mechanical properties, when injecting items used in various technical articles. The test-specimens on PA 6.6 Grivory type HTV-3H1 noir 9205 were molded at the following injection temperatures: 310, 320, 330, 340 and 350°C. The test-specimens on PA 6.6 Grivory type HTV-45H1 noir 9205 were molded at the following injection temperatures: 320, 330, 340, 350 and 360°C. The test-specimens on PA 6.6 Grivory type HTV-6H1 noir 9205 were molded at the following injection temperatures: 330, 340, 350, 360 and 370°C. These specimens were tested using methods for determination of the following mechanical properties: tensile strength, Izod impact strength and hardness (Shore). For all these polymers, it was determined that increasing of injection temperature results in insignificant changes of hardness. The Izod impact strength decreases with increasing the injection temperature. After reaching the minimum value, Izod impact strength increases again if the processing temperature is raised further. The tensile strength at break increases with increasing the processing temperature. After reaching the maximum value, tensile strength at break decreases if the processing temperature is raised further.

Keywords: polyamide 6.6 (PA 6.6), tensile tests, Izod impact test, Shore Durometer hardness test.

Polyethylenes (PE), polyamides (PA), thermoplastic polyurethanes (TPU), polyoxymethylenes (POM), polypropylene (PP), polymethyl methacrylate (PMMA), cellulose acetate (CA), polyvinyl chloride (PVC), polystyrene (PS) and its derivatives, etc may be mentioned amongst the most frequently used polymers for manufacturing of technical parts in various industries. The most frequently used processing technology for these polymers is injection.

Polyamides have good dimensional stability, high rigidity (especially when PA is reinforced with fibre glass), good resistance to compression, wear, shocks and vibrations; they are hard materials, and maintain their hardness and tenacity at high temperatures, with no visible transformations up to 80-90°C [1,2]. PAs are semi-transparent in molded parts with thin wall and opaque in molded parts with thick wall. Reinforcing polyamides with fiber glass results in improved properties of tensile strength, bending resistance and higher values for elastic modulus and hardness. Applications: mechanical engineering (friction parts, tooth wheels, wheel bands), automotive parts (motor housings, fans, parts with sofisticated forms, fuel tanks, bushings, flexible cabling, brake fluid reservoirs), electrical and electronics parts, household items (fruit juicers, kitchen robots, handles of tableware), sport and tourism equipment (ski boots, roller skates, tents, climbing ropes and cords, protective helmets, anti-drop system for bicycle chain), footware (shoe soles) [3].

At injection molding of polymeric materials, the characteristics of molded products are highly influenced by the polymer temperature, mold temperature, cooling time and the pressure of the flowing state material during filling the mold cavity [4 - 8,13].

The present paper aims to analyze the variation of some mechanical properties with injection processing temperature through methods for determination of tensile strength at break, Izod impact strength and penetration hardness (Shore D hardness) for PA 6.6 Grivory noir 9205, type HTV-3H1, HTV-45H1 and HTV-6H1, polymers used at molding technical products in various industries.

Experimental part

The test-specimens were injection molded on the following materials: polyamide 6.6 Grivory type HTV-3H1 noir 9205 (PA 6.6 + 30% fiber glass), polyamide 6.6 Grivory type HTV-45H1 noir 9205 (PA 6.6 + 45% fiber glass) and polyamide 6.6 Grivory type HTV-6H1 noir 9205 (PA 6.6 + 60% fiber glass), using a machine ENGEL CC 100, type ES 80/50 HL, year of manufacturing 1995 (fig.1).

The temperature of the flowing state material was measured using thermocouples placed on the plastification-injection cylinder. The test-specimens on PA 6.6 Grivory type HTV-3H1 noir 9205 were molded at the following injection temperatures: 310, 320, 330, 340 and 350°C. The test-specimens on PA 6.6 Grivory type HTV-45H1 noir 9205 were molded at the following injection

Fig.1 Injection molding machine ENGEL CC 100 Type ES 80/50 HL

* email: maries.radu@rdslink.ro; Tel.: 0745825804

Fig. 2 Polyamide test-specimens made by injection moulding

temperatures: 320, 330, 340, 350 and 360°C. The test-specimens on PA 6.6 Grivory type HTV-6H1 noir 9205 were molded at the following injection temperatures: 330, 340, 350, 360 and 370°C. The injection molded test-specimens are shaped according to figure 2.

During the injection molding of the test-specimens on PA 6.6 Grivory noir 9205, type HTV-3H1,, HTV-45H1 and HTV-6H1, the parameters that influence the injection cycles were maintained constant excepting the processing temperature.

All the test-specimens made by injection molding were tested in order to determine the following mechanical properties: Shore D hardness, Izod impact strength and tensile strength at break.

Measurement of the Shore D hardness

The Shore D hardness was determined with a durometer type D, model SAUTER HB/Germany (fig.3). The method consisted in measuring the initial penetration (depth) on test-specimen and instant recording (less than 1s from pressing) of the values indicated by the device.

Fig.3. Shore D durometer, model SAUTER HB/ Germany

The tests were carried out in accordance with the European Standard SR EN ISO 868:2003 [9] on test-specimens with forms and dimensions such as those illustrated in figure 4. A batch of twenty-five (25) samples was molded at each processing setting and then was tested in order to determine a final result as the arithmetic mean of the measured values.

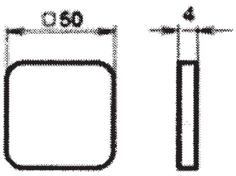

Fig.4. Test-specimen for the hardness determination

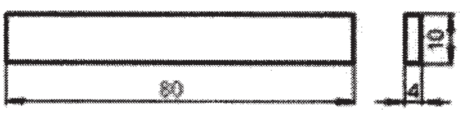

Fig.5. Test-specimen for the Izod impact determination

Measurement of the Izod impact strength

The Izod impact test was performed on unnotched specimens (fig.5).

The tests were carried out in accordance with the European Standard SR EN ISO 180 [10] using a pendulum impact tester model PENDOLO P400, manufactured by HAMMEL/ England (fig.6)

Fig.6. Izod impact tester, model PENDOLO P400, HAMMEL/ England

Fig.7. Tensile testing machine, WPM – VEB Thuringer Industrie werk, Ranenstein great R 37, Typ 2092

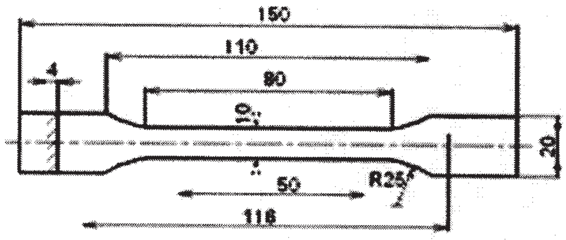

Fig.8.. Test-specimen for tensile strength determination

According to the user manual, the pendulum arm at the initial angle of 150° has an initial potential energy of 7,5 J.

The Izod impact strength of unnotched specimens (a_{iU}) according to SR EN ISO 180 is calculated using the following equation:

$$a_{iU} = \frac{E_c}{h \cdot b} \times 10^3 , \quad [kJ/m^2] \qquad (1)$$

where

E_c - energy (in J) absorbed at breaking the specimen

h - specimen thickness (in mm)

b – specimen width (in mm)

The software of the PENDOLO P400 device displays automatically the value of the energy absorbed at breaking the test-specimen. The test-specimens were fixed in parallel mode. A batch of ten (10) samples was molded at each processing setting and then was tested in order to determine a final result as arithmetic mean of the measured values.

Measurement of the tensile strength at break

Determination of the tensile strength at break was made using a tensile testing machine WPM – VEB Thuringer Industrie werk, Ranenstein gerat R 37, Typ 2092 (fig.7).

Tests were carried out in accordance with the European Standard SR EN ISO 527-1:2000 [11] and SR EN ISO 527-2:2000 [12] on test-specimens with forms and dimensions such as those illustrated in figure 8.

The testing speed for all samples was 200 mm/min. A batch of ten (10) samples was molded at each processing setting and then was tested in order to determine a final result as arithmetic mean of the measured values.

The tensile strength at break was calculated using the following equation:

$$\sigma = F/A, \quad [MPa] \qquad (2)$$

where

F - force [N] measured at breaking the specimen

A – initial cross-sectional area [mm^2] of the test-specimen

PA 6.6 Grivory, type HTV-3H1 noir 9205		PA 6.6 Grivory, type HTV-45H1 noir 9205		PA 6.6 Grivory, type HTV-6H1 noir 9205	
Processing temperature [°C]	Shore D hardness [N/mm²]	Processing temperature [°C]	Shore D hardness [N/mm²]	Processing temperature [°C]	Shore D hardness [N/mm²]
310	88.618	320	91.748	330	92.914
320	88.766	330	92.002	340	93.093
330	89.332	340	92.552	350	93.655
340	89.223	350	92.426	360	93.455
350	89.023	360	92.210	370	93.300

Table 1
PROCESSING TEMPERATURE DEPENDENCE OF PENETRATION HARDNESS FOR INJECTION MOLDED TEST-SPECIMENS ON PA 6.6 GRIVORY NOIR 9205, TYPES HTV-3H1, HTV-45H1 AND HTV-6H1

Materials								
PA 6.6 Grivory, type HTV-3H1 noir 9205			PA 6.6 Grivory, type HTV-45H1 noir 9205			PA 6.6 Grivory, type HTV-6H1 noir 9205		
Processing temperature [°C]	E_c [J]	a_{iU} [kJ/m²]	Processing temperature [°C]	E_c [J]	a_{iU} [kJ/m²]	Processing temperature [°C]	E_c [J]	a_{iU} [kJ/m²]
310	1.568	39.200	320	1.917	47.925	330	1.838	45.950
320	1.380	34.500	330	1.749	43.725	340	1.665	41.625
330	1.340	33.500	340	1.699	42.475	350	1.630	40.750
340	1.243	31.075	350	1.622	40.550	360	1.533	38.325
350	1.314	32.850	360	1.673	41.825	370	1.594	39.850

Table 2
PROCESSING TEMPERATURE DEPENDENCE OF IZOD IMPACT STRENGTH AND ABSORBED ENERGY AT BREAKING UNNOTCHED TEST-SPECIMENS ON PA 6.6 GRIVORY NOIR 9205, TYPES HTV-3H1, HTV-45H1 AND HTV-6H1

Results and discussions

The test-specimens on PA 6.6 Grivory noir 9205, types HTV-3H1, HTV-45H1 and HTV-6H1 were tested using a durometer type D in order to determine the penetration hardness (table 1).

It can be noted that increasing the processing temperature of PA 6.6 Grivory, type HTV-3H1 noir 9205 from 310 to 330°C results in a slightly increased hardness. The maximum hardness was 89.332 N/mm^2 measured at 330°C. The further raise in processing temperature results in a decrease of hardness at 89.023 N/mm^2.

Increase in processing temperature of PA 6.6 Grivory, type HTV-45H1 noir 9205 from 320 to 340°C results in a slightly increase of hardness. The maximum hardness was 92.552 N/mm^2 measured at 340°C. The further raise in processing temperature results in a decrease of hardness at 92.210 N/mm^2.

Increasing the processing temperature of PA 6.6 Grivory, type HTV-6H1 noir 9205 from 330 to 350°C results in a slightly increase of hardness. The maximum hardness was 93.655 N/mm^2 measured at 350°C. The further raise in processing temperature results in a decrease of hardness at 93.300 N/mm^2.

For all these PA 6.6 polymers, it can be noted that the lowest hardness is measured at the lowest processing temperatures (310°C for type HTV-3H1, 320°C for type HTV-45H1 and 330°C for type HTV-6H1), caused by the mechanical degradations of polymer which occur inside the plastification-injection cylinder. At the highest processing temperatures (350°C for type HTV-3H1, 360°C for type HTV-45H1 and 370°C for type HTV-6H1) thermal degradations of polymer occur and lower the hardness as well.

The unnotched test-specimens on PA 6.6 Grivory noir 9205, types HTV-3H1, HTV-45H1 and HTV-6H1 were tested in order to determine the izod impact strength (a_{iu}) and absorbed energy at break (E_c) (table 2):

As for the PA 6.6 Grivory, type HTV-3H1 noir 9205 increasing the processing temperature from 310 to 340°C results in a decrease of impact strength from 39.200 kJ/m^2 to 31.075 kJ/m^2. Raising the processing temperature further at 350°C results in an increase of impact strength at 32.850 kJ/m^2.

As for the PA 6.6 Grivory, type HTV-45H1 noir 9205 increasing the processing temperature from 320 to 350°C results in a decrease of impact strength from 47.925 kJ/m^2 to 40.550 kJ/m^2. Raising the processing temperature further at 360°C results in an increase of impact strength at 41.825 kJ/m^2.

As for the PA 6.6 Grivory, type HTV-3H1 noir 9205, increasing the processing temperature from 330 to 360°C results in a decrease of impact strength from 45.950 kJ/m^2 to 38.325 kJ/m^2. Raising the processing temperature further at 370°C results in an increase of impact strength at 39.850 kJ/m^2.

The test-specimens on PA 6.6 Grivory noir 9205, types HTV-3H1, HTV-45H1 and HTV-6H1 were tested in order to determine the breaking force (F) and tensile strength at break (σ) (table 3).

As for the PA 6.6 Grivory, type HTV-3H1 noir 9205 increasing the processing temperature from 310 to 340°C

Materials								
Polyamide 6.6 Grivory, type HTV-3H1 noir 9205			Polyamide 6.6 Grivory, type HTV-45H1 noir 9205			Polyamide 6.6 Grivory, type HTV-6H1 noir 9205		
Processing temperature [°C]	F [N]	σ [MPa]	Processing temperature [°C]	F [N]	σ [MPa]	Processing temperature [°C]	F [N]	σ [MPa]
310	5031.20	125.78	320	6223.39	155.58	330	6923.07	173.07
320	5189.20	129.73	330	6430.39	160.75	340	7107.07	177.67
330	5305.20	132.63	340	6525.39	163.13	350	7215.07	180.37
340	5868.00	146.70	350	7008.19	175.20	360	7707.87	192.69
350	5592.20	139.80	360	6811.39	170.28	370	7501.07	187.52

Table 3
PROCESSING TEMPERATURE DEPENDENCE OF BREAKING FORCE (F) AND TENSILE STRENGTH AT BREAK (σ) FOR THE TEST-SPECIMENS ON PA 6.6 GRIVORY NOIR 9205, TYPES HTV-3H1, HTV-45H1 AND HTV-6H1

results both in an increase of the force required to break the specimen from 5031,20 N to 5868.00 N and of the tensile strength at break (σ) from 125.78 MPa to 146.70 MPa. The further raise in processing temperature at 350°C results in a decrease both of the force required to break the specimen and of the tensile strength at break.

As for the PA 6.6 Grivory, type HTV-45H1 noir 9205 increasing the processing temperature from 320 to 350°C results in an increase both of the force required to break the specimen from 6223.39 N to 7008.19 N and of the tensile strength at break (σ) from 155,58 MPa to 175.20 MPa. The further raise in processing temperature at 360°C results in a decrease both of the force required to break the specimen and of the tensile strength at break.

As for the PA 6.6 Grivory, type HTV-6H noir 9205, increasing the processing temperature from 330 to 360°C results in an increase both of the force required to break the specimen from 6923,07 N to 7707,87 N and of the tensile strength at break (ó) from 173,07 MPa to 192,69 MPa. The further raise in processing temperature at 370°C results in a decrease both of the force required to break the specimen and of the tensile strength at break.

The conclusion is that the lowest values of tensile strength at break were measured at the lowest and at the highest processing temperatures. At these extreme temperature limits, mechanical degradations (at low temperatures) and thermal degradations (at high temperatures) may occur wîthin the mass of injection molded polymers that influence their tensile strength at break.

Conclusions

The object of the present study is the analysis of the modifications on mechanical properties with injection temperature of PA 6.6 Grivory noir 9205, type HTV-3H1, HTV-45H1 and HTV-6H1, polymers used at molding technical products in various industries. The test-specimens on PA 6.6 Grivory type HTV-3H1 noir 9205 were molded at the following injection temperatures: 310, 320, 330, 340 and 350°C. The test-specimens on PA 6.6 Grivory type HTV-45H1 noir 9205 were molded at the following injection temperatures: 320, 330, 340, 350 and 360°C. The test-specimens on PA 6.6 Grivory type HTV-6H1 noir 9205 were molded at the following injection temperatures: 330, 340, 350, 360 and 370°C. The test-specimens were molded by an injection machine ENGEL CC 100 Type ES 80/50 HL.

The determination of Shore D penetration hardness with a durometer type D, model SAUTER HB/Germany revealed that increasing the processing temperature resulted in insignificant changes of polymer hardness.

The Izod impact test was carried out on unnotched test-specimens using a pendulum impact tester, model PENDOLO P400, manufactured by HAMMEL, England. The Izod impact strength decreases with increasing the injection temperature. After reaching the minimum value, Izod impact strength increases again if the processing temperature is raised further.

The tests of tensile strength at break were carried out using a tensile testing machine WPM – VEB Thuringer Industrie werk, Ranenstein gerat R 37, type 2092. The tensile strength at break is influenced by the processing temperature. The highest value of tensile strength at break for PA 6.6 Grivory type HTV-3H1 noir 9205 was 146.70 MPa at 340°C; for PA 6.6 Grivory, type HTV-45H1 noir 9205 was 175.20 MPa at 350°C; for PA 6.6 Grivory, type HTV-6H1 noir 9205 was 192.69 MPa at 360°C. The lowest values of tensile strength at break were measured at the lowest and at the highest processing temperatures.

References

1. MARSAVINA L., CERNESCU A., LINUL E., SCURTU D., CHIRITA C., Mat. Plast., **47**, no.1, 2010, p.85.
2. ŞEREŞ I., Materiale termoplastice pentru injectare, tehnologie, încercări, Editura Imprimeriei de Vest, Oradea, 2002, p.92-104.
3. MĂRIEŞ GH., R., E., Materiale plastice în designul de produs, Ed. Universităţii din Oradea, Oradea, 2008, p.62-71.
4. MANOVICIU V., MĂRIEŞ GH., R., E., Materiale compozite cu matrice organică, Ed. Universităţii din Oradea, Oradea, 2005, p.283-284.
5. PICHON J., F., Injection des matieres plastiques, Dunod, Paris, 2001, p.121-154.
6. MĂRIEŞ GH., R., E., MANOVICIU I., BANDUR G., RUSU G., PODE V., Mat. Plast., **44**, no.4, 2007, p.289.
7. MĂRIEŞ GH., R., E., MANOVICIU I., BANDUR G., RUSU G., PODE V., Mat. Plast., **46**, no.1, 2009, p.58.
8. NEDELCU D., FETECĂU C., CIOFU C., MINDRU D., Mat. Plast., **46**, no.3, 2009, p.269.
9. *** SR EN ISO 868:2003, Materiale plastice şi ebonită. Determinarea durităţii prin penetrare cu un durometru (duritate Shore).
10*** SR EN ISO 180:2001, Materiale plastice. Determinarea proprietăţilor de şoc Izod.
11. *** SR EN ISO 527-1:2000, Materiale plastice. Determinarea proprietăţilor de tracţiune. Partea 1: Principii generale
12. *** SR EN ISO 527- 2:2000, Materiale plastice. Determinarea proprietăţilor de tracţiune. Partea 2: Condiţii de încercare a materialelor plastice pentru injecţie şi extrudare
13. FETECĂU C., COSMA L., STAN F., Mat. Plast., **44**, no.2, 2007, p.163

Manuscript received: 3.04.2014