Injection moulding: properties customization by varying process conditions

Injection Moulding - Properties Customisation by Varying Process Conditions

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Abstract. The process of injection moulding (IM) is one of the most widely employed methods for manufacturing an extremely diverse range of polymeric parts of varying size and complexity. To match the final application requirements a base material is often modified by certain additives to enhance the material performance. However, the objective of the current research is to explore the effect of different process parameters on the toughness properties of IM polyamide (PA) materials with the aim of customizing the material to meet specific needs. Material suppliers provide their customers with discrete values regarding the mechanical properties, but they only suggest broad windows when specifying processing conditions like the melt temperature. Test samples produced within, as well as below and above the recommendations were produced and tested regarding their quasi-static tensile and instrumented impact behaviour, showing significant differences in performance.

Keywords. Injection moulding, material properties, process parameter.

1. Introduction

Improving the mechanical properties of polymeric materials is key to many final applications. To fulfil required industrial standards, the addition of certain chemical additives, depending on the favoured mechanical properties, to the base material is a popular approach. Polyamides (PAs), a polymer type in high demand for many applications, such as electrical, automotive and sporting goods, are often improved in this way, with different types of additives used, frequently aiming to improve their toughness. Within the literature you find publications investigating the toughening of PAs through the addition of rubber particles [1], [2], glass fibres [3]–[5] or even graphene [6]. The aim of this work is to highlight the change in toughness properties which can be achieved without the use of additional additives, induced only by varying the melt temperature during the injection moulding process. Material suppliers provide their customers with discrete values regarding the mechanical properties, but these note a range of recommended settings when specifying processing conditions. Three different types of PAs, PA-6, PA-12 and PA-610, were injection moulded within and outside of

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the temperature process window suggested and the resulting property changes were stated with focus on the toughness. The influence of different process parameters on the properties of PA materials has been investigated previously [7], [8] mostly with focus on PA-6 and PA-66. A change in mechanical behaviour of injection moulded PAs due to different applied process temperatures is directly related to the resulting material morphology, highlighted by differences in the skin-to-core ratio [9]. PAs are hygroscopic materials and their properties are dependent on the amount of moisture they contain [10], [11] due to the effect on their intermolecular chain mobility. To highlight the difference in the tested PA materials, featuring a different CH_2 to NHCO ratio, tests were conducted on dry and conditioned samples. Those two states were chosen to highlight the difference in material properties immediately after the ejection of the part (dry) and after moisture saturation (conditioned state). Moisture saturation can take up to several months, if passive conditioned, depending on the PA type [12].

2. Experimental

2.1. Selected Materials and Processing Parameter

Three commercially available PA materials were selected, namely two homopolymers, PA-6 (impact modified) and PA-12, and a copolymer, PA-610. The materials were injected at four different melt temperatures using a consistent mould temperature of 60°C, as presented in Table 1.

Table 1. Injection moulding process parameter: melt and mould temperature

Process parameter	Mod 1	Mod 2	Mod 3	Mod 4
Melt temperature [°C]	230	260	290	320
Mould temperature [°C]	60	60	60	60

The suggested process windows from the respective material supplier are 240-300°C for the PA-6, 220-250°C for PA-12, and 260-290°C for the PA-610.

2.2. Sample Preparation

All tests were performed on 2 mm thick samples, press cut, with different geometries according to the applied testing standards (BS ISO 527 and 6603). All test specimens were either conditioned, to simulate moisture saturated samples, at 70°C and 62% relative humidity to accelerate the moisture intake, or dried out at 70°C. All the requested conditioning times and changes in weight are presented in Table 2 and all test specimens were conditioned accordingly, to ensure reproducible test results.

Table 2. Conditioning study different PA materials: time and weight increase

Conditioning study	PA-6	PA-12	PA-610
Conditioning time [hours]	240	96	216
Drying time [hours]	216	96	144
Weight difference [%]	3.17	1.01	1.59

The difference in conditioning time and weight (indicating the equilibrium moisture uptake data) are in accordance to the CH_2 to NHCO ratio of the different PA types.

2.3. Mechanical Testing

Quasi-static tensile test data was obtained using an Instron 3366 tensometer operating at a speed of 1 mm/min during the first 0.3% strain and 50 mm/min afterwards with an initial clamp separation of 115 mm. ISO BS 527-2 Type 1A samples were tested in the flow direction to determine tensile properties. Values for the tensile modulus and toughness (indicating stress and strain at break) were further investigated. Instrumented falling weight impact tests were conducted on an Instron Dynatup 9250 using a 20 mm diameter striker at an impact speed of 4.4 m/s providing an overall impact energy of around 197 J. Circular specimens with 52 mm diameter were tested in accordance with the ISO BS 6603-2 standard. For the instrumented impact results the focus was on the detected peak force, the total energy and the differences in puncture deflection.

3. Results and Discussion

The final properties of an injection moulded part are influenced by the melt temperature applied during production, which has a direct effect on melt viscosity and also influences the simultaneous loss of heat during the injection phase. PA samples produced as presented in Table 1 were tested and compared regarding their quasi-static and instrumented impact properties. The data obtained for all three PA types is presented below, starting with the quasi-static tensile test results for PA-6 tested.

Table 3. Test results quasi-static tensile: PA-6 dry ¦ conditioned

Mod	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]
1 - 230°C	2670 ± 20	61.4 ± 8.4	7.5 ± 1.9	903 ± 24	55.0 ± 0.7	216 ± 1
2 - 260°C	2410 ± 20	47.9 ± 13.6	10.1 ± 2.9	704 ± 37	52.8 ± 3.4	212 ± 21
3 - 290°C	2470 ± 80	31.5 ± 11.2	41.4 ± 25.2	653 ± 20	52.2 ± 2.6	244 ± 15
4 - 320°C	2450 ± 60	44.8 ± 3.1	50.0 ± 26.2	638 ± 28	62.1 ± 7.9	317 ± 43

The tensile toughness of PA-6 samples tested in a dry state were found to be highly influenced by the applied melt temperature. The deviation in stress (31.5 - 61.4 MPa) and strain at break (7.5 - 50%) is obvious, with samples produced at low melt temperatures (Mod 1 and 2) failing mostly within the yield region, prior to gross material necking. An obvious influence on the modulus data was detected for samples produced at the lowest melt temperature settings (Mod 1 - 230°C) in both conditioning states. Superior tensile toughness properties were found for the 320°C (Mod 4) samples due to higher elongation at break. The influence of moisture led to a consistent decrease in modulus, by 71% ± 3% on average, due to the reduction in intermolecular attractions. The strain at break results showed a relative increase by up to 2775 % for the 230°C samples (Mod 1), which was due to the early failure within the yield region when tested dry. This also explains the recorded stress at break decrease from 61.4 MPa to 55.0 MPa, while it increased for all the other process modifications. The tensile results for the PA-12, featuring a longer methylene backbone, are shown in Table 4 for dry and conditioned samples.

Table 4. Test results quasi-static tensile: PA-12 dry ¦ conditioned

Mod	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]
1 - 230°C	1090 ± 64	52.1 ± 2.0	187 ± 8	567 ± 33	52.2 ± 4.0	225 ± 18
2 - 260°C	1050 ± 87	61.1 ± 4.7	257 ± 25	562 ± 29	52.1 ± 5.8	248 ± 32
3 - 290°C	1030 ± 58	53.2 ± 1.8	252 ± 16	569 ± 6	50.6 ± 4.8	267 ± 29
4 - 320°C	1010 ± 68	62.9 ± 2.7	334 ± 22	584 ± 22	60.4 ± 1.1	366 ± 16

Comparable to the PA-6 findings, the PA-12 shows a decrease in tensile toughness when injected at a low melt temperature (Mod 1 - 230°C) and superior properties at 320°C (Mod 4). The modulus decreased consistently due to the influence of moisture, by 45% ± 2% on average and a reduced moisture sensitivity due to the higher CH_2 to NHCO ratio was confirmed. The overall excellent toughness properties for which PA-12 is often specified, were highlighted by strain at break values above 180% for all modifications, even when tested dry. Table 5 shows the tensile data for the dry and conditioned tested PA-610 copolymer samples.

Table 5. Test results quasi-static tensile: PA-610 dry ¦ conditioned

Mod	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]	Modulus [MPa]	Stress at break [MPa]	Strain at break [%]
1 - 230°C	2420 ± 10	49.6 ± 0.4	17.8 ± 1.1	1170 ± 28	42.6 ± 0.8	52 ± 9
2 - 260°C	2320 ± 8	48.8 ± 0.6	37.8 ± 8.2	1080 ± 12	40.4 ± 0.6	58 ± 15
3 - 290°C	2408 ± 3	47.0 ± 0.9	24.9 ± 3.6	1134 ± 13	62.3 ± 4.2	253 ± 7
4 - 320°C	2527 ± 29	61.0 ± 5.1	15.3 ± 2.2	1248 ± 42	58.6 ± 2.6	258 ± 13

While the highest applied melt temperatures (Mod 4 - 320°C) had a consistently positive influence on the tensile toughness properties for both homopolymers, several samples of the PA-610 copolymer failed within yield region, combined with the highest modulus findings, when tested dry. When tested in the conditioned state, superior tensile toughness properties were found for samples injected at both 290°C (Mod 3) and 320°C (Mod 4), the high process melt temperature modifications. Considering the recommended melt temperature window of 260 - 290°C by the material supplier, the tensile toughness properties of the a final part can very between 40.4 MPa and 62.3 MPa with regards to the stress and 58 % to 253 % in terms of strain readings, depending on which end of the recommended temperature range the part was injected at.

The corresponding instrumented impact test results are shown below, with Table 6 summarizing the PA-6 test samples.

Table 6. Test results instrumented impact: PA-6 dry ¦ conditioned

Mod	Peak force [N]	Puncture deflection [mm]	Total energy [J]	Peak force [N]	Puncture deflection [mm]	Total energy [J]
1 - 230°C	1870 ± 1400	5.2 ± 2.65	5.8 ± 5.6	3830 ± 152	19.4 ± 0.82	47.9 ± 2.2
2 - 260°C	1320 ± 715	4.1 ± 1.41	3.1 ± 1.9	3690 ± 110	19.6 ± 0.16	46.8 ± 1.3
3 - 290°C	4130 ± 1420	16.5 ± 6.05	50.6 ± 22.7	3500 ± 41	19.6 ± 0.06	46.0 ± 0.6
4 - 320°C	4560 ± 68	17.8 ± 2.66	55.4 ± 12.6	3560 ± 116	19.5 ± 0.18	46.6 ± 1.5

When injection moulded at lower melt temperatures (230°C and 260°C) all samples tested dry experienced brittle failure by fracture and crack growth, which explains the low values in all relevant categories. Higher temperatures (290°C and 320°C) lead to a significant improvement in impact properties, with only one sample in each condition failing by brittle fracture, while all the others showed ductile yielding behaviour. Clearly the conditioned state of this PA has a critical influence on the ductile-brittle transition, under high velocity loading. These findings correlate with the tensile toughness results for dry samples stated in Table 3. When conditioned, all samples showed ductile behaviour with higher peak force readings when produced at 230°C (Mod 1). The detected impact properties for the PA-12 are presented in Table 7.

Table 7. Test results instrumented impact: PA-12 dry ¦ conditioned

Mod	Peak force [N]	Puncture deflection [mm	Total energy [J]	Peak force [N]	Puncture deflection [mm]	Total energy [J]
1 - 230°C	3630 ± 88	23.0 ± 0.52	51.4 ± 2.1	3390 ± 65	22.0 ± 0.51	47.1 ± 1.3
2 - 260°C	3570 ± 86	22.8 ± 0.25	50.9 ± 1.7	3320 ± 62	22.1 ± 0.37	46.8 ± 1.0
3 - 290°C	3390 ± 47	22.5 ± 0.19	47.7 ± 0.9	3160 ± 44	21.8 ± 0.56	43.7 ± 1.3
4 - 320°C	3390 ± 20	22.2 ± 0.47	47.2 ± 0.5	3140 ± 35	22.0 ± 0.38	43.6 ± 0.8

In contrast to the observed trends for PA-6, all PA-12 samples showed exclusively ductile properties with an obvious offset in peak force and the resulting total energy between the lower (230°C and 260°C) and the higher melt temperatures (290°C and 320°C). Due to the moisture intake the properties decreased consistently by around 7% for peak force and 8% for the resulting total energy, while no obvious trend was observed in puncture deflection. Table 8 summarizes the outcomes from the testing of PA-610.

Table 8. Test results instrumented impact: PA-610 dry ¦ conditioned

Mod	Peak force [N]	Puncture deflection [mm	Total energy [J]	Peak force [N]	Puncture deflection [mm	Total energy [J]
1 - 230°C	4680 ± 103	18.3 ± 0.31	58.1 ± 1.3	4440 ± 168	18.7 ± 0.20	55.5 ± 2.3
2 - 260°C	4730 ± 91	18.8 ± 0.15	59.5 ± 1.5	4330 ± 145	19.2 ± 0.26	55.0 ± 2.2
3 - 290°C	4660 ± 33	18.6 ± 0.28	58.1 ± 0.9	4270 ± 133	18.9 ± 0.25	54.4 ± 1.9
4 - 320°C	4100 ± 1860	15.4 ± 7.12	50.9 ± 24.9	4470 ± 120	18.9 ± 0.18	56.7 ± 1.6

When tested dry, samples produced at a high melt temperature (Mod 4 - 320°C) showed reduced impact properties due to two brittle failures, which is in accordance with the tensile toughness properties presented in Table 5. All other modifications, dry and conditioned, showed purely ductile behaviour.

4. Conclusion

By varying the applied melt temperature the toughness of injection moulded PA materials can differ significantly. Despite differences in loading rate and direction a good correlation was observed between obtained toughness data from tensile tests and instrumented impact test results. Samples which failed to neck during the uniaxial quasi-static test, showed a higher probability to fail by brittle mode when impact tested. It was demonstrated that adjusting the melt temperature can shift the ductile/ brittle transitions of a polyamide materials, resulting in failure by ductile yielding without the addition of any additives. While the PA-12 samples showed superior toughness properties across all modifications, the PA-6 and PA-610 were found to be more limited regarding their process window. Higher melt temperatures (290°C and 320°C) improved the impact properties of impact-modified PA-6 tremendously. A higher melt temperature was also found to be beneficial for PA-610, but when injected at 320°C the samples were extremely moisture sensitive, failing prematurely when tested dry. The positive impact of higher melt temperatures can be explained by structural modification, possibly an increase in molecular weight due to cross linking following thermal degradation which has been reported for homo- and co-polyamides [13]. As expected, PA-6 was most influenced by moisture content since it has the lowest CH_2 to NHCO ratio in its structural repeat units. The obtained differences in properties on the macro level will be investigated further by an extensive material characterization, using optical microscopy and Differential Scanning Calorimetry (DSC), to understand the effects on part microstructure and the resulting mechanisms of mechanical deformation and failure.

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