

RESEARCH PAPER

UTILIZATION OF ANALYTICAL METHODS FOR THE FAILURE ANALYSIS OF INJECTION MOLDED PART

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ABSTRACT

As the use of the thermoplastic materials becomes conventional in numerous parts and various applications, incidence of various types of failure becomes inevitable. Failure of injection molded parts can be a cause of economic and legal problems, as well as causing health damage or death. Public perception of plastics is adversely affected by their failures, such as the bad reputation that early plastics earned with toys that broke too easily. As the injection molding process is very complex, finding the cause of failure is very complicated. In industrial mass scale production, determining the cause of failure is key point in customer-supplier relationship. In the presented study, a type of injection molded product from HDPE affected by occurrence of cracks was investigated by mechanical, thermal testing and spectral analysis. Mechanical properties were evaluated by the uniaxial tensile test and the SHORE hardness test. Thermal properties of the samples were evaluated by DSC/TGA analysis and for the spectral analysis a Raman spectroscopy device was used. Obtained results provided information about fluctuating quality of used material, coupled with non/stable molding conditions and small molding window. All those aspects caused molded in stress, which was released in the form of crack during the installation.

Keywords: molding failure; DSC; Raman Spectroscopy; tensile test

INTRODUCTION

High-Density Polyethylene (HDPE) is one of the most widely used thermoplastic material and probably the most valuable polymer in our daily life. It is used for production of various types of products, ranging from make grocery bags, shampoo bottles, children's toys, up to bullet proof vests [1-2]. Despite its versatility, PE polymer has the simplest structure of all commercial polymers, and the performance of the PE can be further improved by creating various composites with PE matrix [1-4].

As the utilization of the HDPE and HDPE composites becomes conventional in numerous parts and various applications, incidence of various types of failure becomes inevitable, ranging from cosmetics defects [5]. Failure of injection molded parts can be a cause of economic and legal problems, as well as causing health damage or death. Public perception of plastics is adversely affected by their failures, such as the bad reputation that early plastics earned with toys that broke too easily [6]. As the injection molding process is very complex, finding the cause of failure is very complicated. In industrial mass scale production, determining the cause of failure is key point in customer-supplier relationship. It is necessary to determine, whether the problem was caused by the selection of inappropriate material, design error, variance in material quality or the actual processing. That is why sophisticated testing methods are applied in such cases [6-8]. Methods determining the properties and cause of failure of HDPE part are mostly focused on laboratory testing of mechanical properties, thermal properties and the structure of the polymer with different level of crystallinity [9, 10].

In the presented paper, a type of injection molded product from HDPE (Fig.1) affected by occurrence of cracks was investigated by mechanical, thermal testing and spectral analysis. Cover (outside diameter = 218 mm) made of commercial HDPE grade DOW HDPE KS10100 UE exhibits crack occurrence and propagation through central part of molding (Fig.2), before, during or short after installation and therefore could not meet the desired service lifetime. Suspecting not stable molding condition at the producer molding shop and the material quality issues a set of analytical tests was necessary to find out the cause of crack occurrence, as some batches of cover did not have any problem.



Fig. 1 Molded cover (inside)

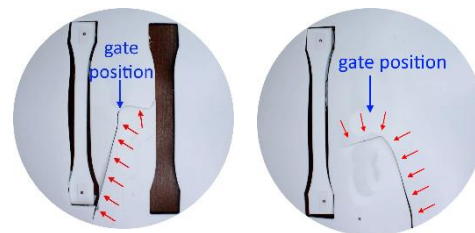


Fig. 2 Crack propagation/ cut location of tensile test samples

Numerous studies were focused on investigation of properties and crack causes in products made of HDPE, especially piping made of HDPE due to its wide use and application. To determine cause of failure of predict service lifetime standardized tests [11, 12], custom experiments [13] or FEA numerical modeling [14-16] were applied.

As the degree of crystallinity has significant influence on properties and performance of HDPE parts [2, 9], determining the degree of crystallinity in the part can provide important information about properties and structure of the material. One of the most applied method to determine the degree of crystallinity in the HDPE is the DSC. Although the sample size is very small, the sample destruction is inevitable. In cases where the sample destruction is not allowed, more detailed information about material structure is needed or to compare the DSC obtained crystallinity results a Raman spectroscopy is applicable [17-19].

MATERIAL AND METHODS

In this study totally six samples of injection molded part made of DOW HDPE KS10100 UE (supplier information) compound were investigated. Two of the samples with no quality issue and crack occurrence (sample no. 33 and 34) and four samples with crack (sample no. 35,36,37,38).

To evaluate mechanical properties of the samples material standardized uniaxial tensile tests according ISO 527-1 were performed and determination of samples surface Shore D hardness was determined according ASTM D2240. For structure and crystallinity observation the DSC testing method (ASTM D3418) on TA-

Q600 device and Raman spectroscopy with DeltaNu Rapid ID spectrometer were applied.

As presented in fig.2 a test sample for tensile test was cut out from the central part of the molding, that was not affected by crack. Obtained samples were analyzed before the tensile test with Raman spectrometry and the Shore D hardness. Samples for the DSC analysis were cut out from the molding, at the center of hole edge that remained after tensile test sample cut.

Three measurements were performed on each test sample. Average values of the hardness of individual samples were recorded. During the Shore D hardness testing, both sides of samples were measured. This approach was chosen, to capture eventually not uniform mold temperature resulting in different degree of crystallinity leading to differences in surface hardness.

RESULTS AND DISCUSSION

Tensile test

Table 1 shows the evaluated data from tensile test. The samples no. 33,34 without crack, meet the supplier tensile yield strength of 25 MPa and so the sample no.37 with crack do. Other samples with crack had the yield strength slightly below the supplier declared value. Standard deviation of tensile strength yield is 0.715.

Table 1 Results of tensile test

| Sample | Evaluated values | |
|---------------|------------------------------|------------------------|
| | Tensile strength yield [MPa] | Tensile elongation [%] |
| 33 | 25.50 | 614 |
| 34 | 26.15 | 652 |
| 35 | 24.80 | 344 |
| 36 | 24.29 | 389 |
| 37 | 25.35 | 400 |
| 38 | 24.04 | 351 |
| Mean | 25.08 | |
| St. Deviation | 0.715 | |

Markedly differences are visible when comparing the value of tensile elongation. None of the samples reached the supplier declared value (1600%) , but the samples without crack have approximately 2 times higher tensile elongation. This could explain their resistance to crack building. All values of tensile elongation below material datasheet value can lead us to a conclusion that either extensive application of recycled material was used, or the molding condition (shear rate) were so high, that they lead to material degradation.

Shore D hardness

Shore D hardness measurement was performed on samples No. 33, 34, 35, 36, 37, 38 and 38 (Table 2). Measurements of samples showed differences in hardness values depending on which side of the sample was measured. The top side of the samples (as received and marked) on which the sample number was placed is "blue" and the reverse side "green". The measured values listed in Table 2 do not show significant differences in hardness between the samples but the mean value on the outside 59.72 is lower than the Shore D hardness 61.08 observed on the inside of the molding. The calculated mean values of Shore D hardness (tab.2), and the graph (Fig.3) with standard deviation show difference trend between the "blue" and "green" side.

Table 2 Results of Shore D hardness test

| Sample | Shore D hardness | |
|---------------|------------------|---------------|
| | blue /outside | green /inside |
| 33 | 59,9 | 60,3 |
| 34 | 59,8 | 60,5 |
| 35 | 59,5 | 62,7 |
| 36 | 59,8 | 61,3 |
| 37 | 59,8 | 60,8 |
| 38 | 59,5 | 60,9 |
| Mean | 59,72 | 61,08 |
| St. deviation | 0,172 | 0,864 |

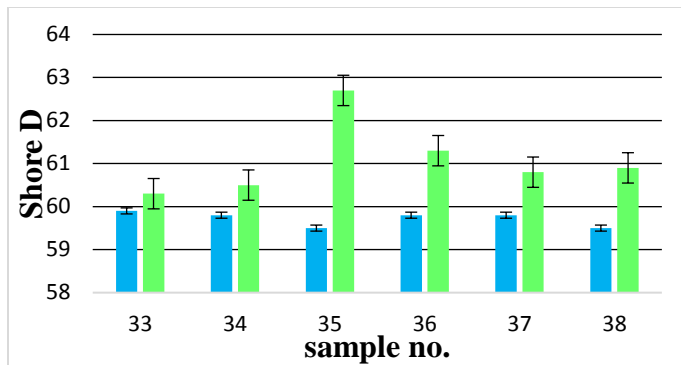


Fig. 3 Shore D results comparison for both sides of molding

DSC analysis

Data from DSC analysis are listed in Table 3. The results show that the melting temperature for individual samples is in the range from 133.93°C up to 135.37°C (Fig.4) and that corresponds to the HDPE polymer and no further peaks up to 200°C were observed. For samples no.33 and no.34 a higher degree of crystallinity was measured at level 71.92%, resp. 72.73%. Other samples taken from the moldings with crack achieve the measured crystallinity in the range from 67.19% up to 70.02%.

Table 3 Results of DSC analysis

| Sample | Evaluated values | |
|--------|--------------------------|-----------------------------|
| | Melting temperature [°C] | Degree of crystallinity [%] |
| 33 | 135.37 | 71.92 |
| 34 | 134.70 | 72.73 |
| 35 | 133.93 | 67.19 |
| 36 | 134.98 | 70.02 |
| 37 | 134.92 | 68.13 |
| 38 | 135.09 | 68.90 |

Obtained crystallinity results from DSC analysis shows strong correlation between degree of crystallinity and tensile elongation.

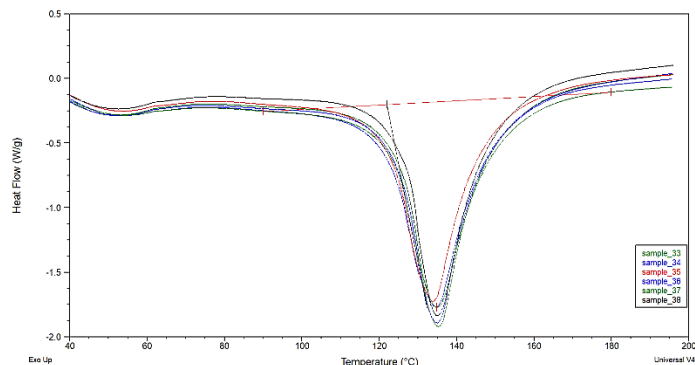


Fig. 4 DSC curves of measured samples

Raman spectrometry

Raman spectra of tested samples is presented in Fig. 5. Raman spectrum shift for samples in the range 1020-1550 cm⁻¹ was measured. The peaks of spectral shifts at 1065 cm⁻¹, 1130 cm⁻¹ and 1280 cm⁻¹ distinguish clearly the samples 33 and 34 with higher crystallinity level. Spectral peaks of samples 33 and 34 at 1065 cm⁻¹ resp. 1280 cm⁻¹ are highest among tested samples, whereas their peak at 1130 cm⁻¹ is the lowest. In Fig.5 are the spectral peaks of sample 34 with crystallinity of 72.73% marked with arrow.

These result of Raman spectral analysis show that this method is suitable for quick comparison of HDPE crystallinity degree between samples although not

exact value is obtained. The results from Raman spectroscopy match well to the results of the DSC analysis.

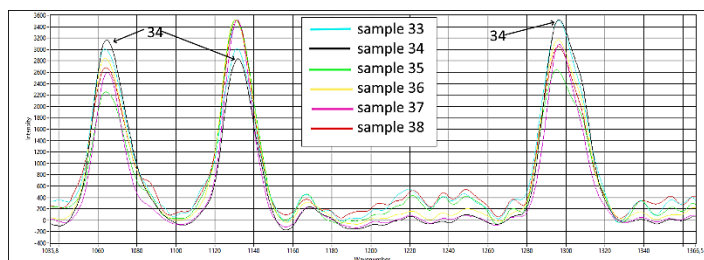


Fig. 5 Raman spectrum shifts of measured samples

CONCLUSION

Utilization of the HDPE as versatile material is nowadays present in wide variety of products. The wide variety of applications leads to occurrence of defects and failures of injection molded parts. Complexity of the injection molding process causes difficulties on the understanding of the failure cause. In mass scale production, determining the cause of failure is crucial factor in customer-supplier relationship. In practice there is high demand for methods that can quickly determine whether the problem was caused by the selection of inappropriate material, design error, variance in material quality or the actual processing. In presented study four types of methods for analyzing HDPE material properties were applied to compare six molding. Two of them that meet the customer quality criteria (33,34) and four of them with failure (crack) (35,36,37,38).

- From the tensile test it was determined that yield strength of all samples fit in range 24.04-26.15MPa. The significant difference appears in the values of elongation. Samples 33,34 from good moldings exhibit approximately 2 times higher elongation at break (614%,652%) that samples from failure moldings (344-400%)
- Results from hardness measurement shows slightly difference and trend on all samples when comparing the inside and outside of the moldings. Difference between molding inside and outside is probably attributable to different mold temperatures during injection molding. However, this assumption could not be verified by processing data from supplier and further tests were considered unnecessary by the customer.
- DSC analysis provided values of melting temperature and degree of crystallinity. While the melting temperature remain in narrow range 133.93 - 135.37°C and no correlation between failure and melting temperature was observed, the influence of degree of crystallinity is obvious. Samples 33 and 34 have the crystallinity above 71.92% and samples from failure moldings have crystallinity lower than 70.02%.
- Data from Raman spectroscopy and analysis of Raman spectra shifts show that a correlation between degree of crystallinity determined by DSC analysis and spectrum peaks at 1065 cm⁻¹, 1130 cm⁻¹ and 1280 cm⁻¹ is observable.

Based on obtained results it can be stated that material used to produce the moldings was the same, although the amount of virgin material is questionable due to low elongation at break. The degree of crystallinity was key factor that distinguishes the moldings with good quality and the molding with crack, and this factor was affected by not stable molding conditions. Taking into account the easy and non-destructive way to obtain Raman spectra, the method seems as ideal way for customer to quickly compare injection molded part and predict their quality.

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REFERENCES

1. A. Ghanbari-Siahkali, P. Kingshott, D. Werner Breiby, L. Arleth, C. Koch Kjellander, K. Almdal: Investigating the role of anionic surfactant and polymer morphology on the environmental stress cracking (ESC) of high-density polyethylene. *Polymer Degradation and Stability*, 89, 2005, 442-453. <https://doi.org/10.1016/j.polymdegradstab.2005.01.023>

2. N. Pundhir, G. Arora, H. Pathak, S. Zafar: *Ballistic Impact Response of HDPE/UHMWPE Polymer Composite*. In.: *Advances in Mechanical Engineering Select Proceedings of ICRIDME 2018*. <https://doi.org/10.1007/978-981-15-0124-1>
3. K. Głogowska et al.: Assessment of the resistance to external factors of low-density polyethylene modified with natural fillers. *Advances in Science and Technology Research Journal*, 11(4), 2017, 35-40. <https://doi.org/10.12913/22998624/75984>
4. J. Sikora et al.: Polyethylene-Matrix Composites with Halloysite Nanotubes with Enhanced Physical Thermal Properties. *Polymers*, 11(5), 2019, 787-787. <https://doi.org/10.3390/polym11050787>
5. L. Dulebová, T.Garbacz, V. Krasinskyi, B. Duleba: The influence of modifying HDPE on properties of the surface, *Materials Science Forum*, 818, 2015, 101-104. <https://doi.org/10.4028/www.scientific.net/MSF.818.101>
6. M. Ezrin: *Plastic Failure Guide. Causes and Prevention*. Hanser Publishing, 1996, ISBN 3-446-15715-8
7. T. Jachowicz: Investigation of Selected Properties of Injection-Molded Parts Subjected to Natural Aging. *International Journal of Polymer Analysis and Characterization*, 20, 2015, 307-315. <https://doi.org/10.1080/1023666X.2015.1016788>
8. T. Jachowicz, V. Krasinskyi, I. Gajdoš: The investigation of the influence of pro-degradant content on chosen properties of polymer composite. In: *Technological and design aspects of extrusion and injection moulding of thermoplastic polymer composites and nanocomposites*, volume 2. Košice, TU, 2014, 53-77. ISBN 978-80-553-1677-2
9. E. Nezbedova, A. Zahradnickova, Z. Salajka: Brittle failure versus structure of HDPE pipe resins. *Journal of Macromolecular Science, Part B*, 40(3-4), 2001, 507-515. <https://doi.org/10.1081/mb-100106173>
10. J.L. Jordan et al.: Low pressure shock response and dynamic failure of high-density polyethylene (HDPE). *AIP Conference Proceedings*, 1979(1), 2018, 090006. <https://doi.org/10.1063/1.5044863>
11. D.E. Duvall, D. Edwards: Field Failure Mechanisms in HDPE Potable Water Pipe, *Plastics Engineering*, 2012, <https://doi.org/10.1002/j.1941-9635.2012.tb00808.x>
12. S. Na, L. Nguyen, S. Spatari, Y. G. Hsuan: Effects of recycled HDPE and nanoclay on stress cracking of HDPE by correlating Jc with slow crack growth. *Polymer Engineering & Science*, 58, 2018, 1471-1478. <https://doi.org/10.1002/pen.24691>
13. F. Majid, M. Elghorba: HDPE pipes failure analysis and damage modeling. *Engineering Failure Analysis*, 71, 2017, 157-165. <https://doi.org/10.1016/j.engfailanal.2016.10.002>
14. S. Kalyanam et al.: Constraint Effects in Slow Crack Growth Test Methods for Service Life Prediction of High Density Polyethylene Piping. *Journal of Pressure Vessel Technology*, 2020, <http://dx.doi.org/10.1115/1.4046884>
15. M. A. Bouaziz et al.: Failure Analysis of HDPE Pipe for Drinking Water Distribution and Transmission. In: *Design and Modeling of Mechanical Systems - II. Lecture Notes in Mechanical Engineering*. Chouchane M., Fakhfakh T., Daly H., Aifaoui N., Chaari F. (eds). Springer, Cham, 2015, 407-414. https://doi.org/10.1007/978-3-319-17527-0_41
16. Z. Hao, J. Liu, J. Chang: Study on the Effect of HDPE Stress Absorbing Layer in Preventing Reflective Cracks, *Transportation Soil Engineering in Cold Regions, Lecture Notes in Civil Engineering*. Proceedings of TRANSOILCOLD 2019, p. 331-340, https://doi.org/10.1007/978-981-15-0450-1_34
17. I. Gajdoš, E. Spišák, V. Moravskyi: *Raman spectroscopy in polymer composites identifying and processing technologies*. In: *Technological and design aspects of modern methods of composite and nanocomposite processing: Book of abstracts: 18-19 February 2015, Lviv, Ukraine*, ISBN 978-617-607-711-4

18. I. Gajdoš et al.: Raman spectroscopy in polymer processing technologies. *Acta Mechanica Slovaca*, 15(4), 2011, 68-73
19. R. P. Paradkar, et al.: Raman Spectroscopy of Polymers. *Encyclopedia of Analytical Chemistry*, (2009), <https://doi.org/10.1002/9780470027318.a9032>