Experimental validation on the effect of material geometries and processing methodology of Polyoxymethylene (POM)

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Abstract. POM-copolymer bond breaking leads to change depending with respect to processing methodology and material geometries. This paper present the oversights effect on the material integrity due to different geometries and processing methodology. Thermo-analytical methods with reference were used to examine the degradation of thermomechanical while Thermogravimetric Analysis (TGA) was used to judge the thermal stability of sample from its major decomposition temperature. Differential Scanning Calorimetry (DSC) investigation performed to identify the thermal behaviour and thermal properties of materials. The result shown that plastic gear geometries with injection molding at higher tonnage machine more stable thermally rather than resin geometries. Injection plastic gear geometries at low tonnage machine faced major decomposition temperatures at 313.61°C, 305.76 °C and 307.91 °C while higher tonnage processing method are fully decomposed at 890°C, significantly higher compared to low tonnage condition and resin geometries specimen at 398°C. Chemical composition of plastic gear geometries with injection molding at higher and lower tonnage are compare based on their moisture and Volatile Organic Compound (VOC) content, polymeric material content and the absence of filler. Results of higher moisture and Volatile Organic Compound (VOC) content are report in resin geometries (0.120%) compared to higher tonnage of injection plastic gear geometries which is 1.264%. The higher tonnage of injection plastic gear geometry are less sensitive to thermo-mechanical degradation due to polymer chain length and molecular weight of material properties such as tensile strength, flexural strength, fatigue strength and creep resistance.

1. Introduction

The use of POM is growing steadily in the automotive and electronic industries. Ever since the unique physical and chemical properties and the ease of production allow the special of materials to start thriving in the global market from household items to biomedical, civil, electronics, and automobile sectors. However, a large number of reactions take place during processing and small changes in the molecular structure result in considerable modification of properties of POM material.
In real application of development stage, prototyping is important to investigate and validate a new design to enhance confident level before shifting to mass production. These include plastic gear that are now frequently utilize at present industry. However, the potential deficiencies across dissimilar process and its effect on the integrity of the plastic material may rise due to this different.

These profounder issues are not often given proper consideration until a related gear failure demands study and evaluation. With molded plastic gears, this practice of relying on physical measurements as proof of quality overlooks potential deficiencies in the injection molding process and the effect of material geometries on the integrity of the plastic material. Thus the aim of the present study is to present changes the molding deficiencies and to discuss the hidden effects produced at the end properties of plastic gear. There is a lot of published data available on the physical properties only. Unfortunately, not much information about on how POM material’s properties are affected material geometries and processing conditions during injection molding. Nevertheless, it is understood that the thermodegradation conditions in both scenarios are equal in the sense of pursuing the effect of temperature and time on the polymer degradation [5].

For POM material, a good approach for the quantification of degradation induced under service lifetime is to determine stabiliser consumption and beginning degradation by means of TGA. Basically the decrease of the onset temperature in the thermogravimetric Analysis (TGA) is the first indication for thermo-oxidative damage, whereas a decrease of the onset temperature without a change of the first derivative of the TG signal Derivative thermo-gravimetric (DTG) refers explicitly to stabiliser consumption. Degradation can be determined by a change of the DTG signal [1].

2. Experimental

Three samples of POM (polyoxymethylene) material are prepared for resin, POM gear with higher tonnage and lower tonnage under injection moulding processing method. The resin in Figure 1 was Lupital F20-03 natural colour supplied from IDES with a volume flow rate MVR 7.70 cm³ /10 min, according to the standard ISO 1133. While Figure 2 for Gear 1 and Gear 2 conditions were standard for the type of polymer (Table 1) using a temperature profile ranging from 100 °C to 230 °C from hopper to nozzle with a moulding machine tonnage lower at 100 ton and 150 ton at higher tonnage.

| Condition                     | Value                           |
|-------------------------------|---------------------------------|
| Pellets pre-drying            | 130°C for 3 hours               |
| Melt temperature at Nozzle    | 195°C                           |
| Hot Runner Temperature        | 205°C ~ 190°C                   |
| Cavity Temperature            | 70°C                            |
| Part Weight (g)               | 16.28                           |

| Condition                   | Low  | High |
|-----------------------------|------|------|
| Injection Velocity (mm/s)   | 100  | 150  |
| Melting Temperature (°C)    | 190  | 230  |
| Moulding Temperature (°C)   | 60   | 100  |
| Holding time (s)            | 20s  | 28s  |
| Holding Pressure (bar)      | 500  | 1500 |
| Machine Tonnage (ton)       | 100  | 150  |

Thermogravimetric (TG) studies were carried out with a TGA 2050. The sample for higher and lower tonnage of plastic gear injection condition were cut into small pieces, conditioned at room temperature with 50% Humidity for 3h before it were heated from 30°C to 1000°C at 20°C/min with
N2 gas purging. For the condition of Differential Scanning Calorimetry (DSC), the investigation was carried out by cut off the samples of higher and lower tonnage condition into small pieces before it was heated with N2 gas purging based on the heat cycle which are; heat from 30°C to 250°C at 10°C/min, isothermal at 250°C for 1 minute, cool from 250°C to 30°C at 10°C/min, isothermal at 30°C for 5 minute and heat from 30°C to 250°C at 10°C/min:

![Figure 1: POM Resin](image1.jpg)

![Figure 2: Sample Gear 1 and Gear 2 (Area Analyse)](image2.jpg)

3. Results.

3.1. Thermogravimetric Analysis (TGA).

The thermal stability of sample was judged from its major decomposition temperature. By referring to the Table 2, testing samples of gear with higher tonnage processing method is slightly more stable thermally compare than pure resin and gear with lower tonnage samples where their respective major decomposition temperatures are 313.61°C, 305.76°C, 307.91°C and respectively. Results of TGA in Table 2 shown that sample of Gear 1 are not fully decomposed (0.337%) even though the temperature testing reached 890°C proved that Gear 1 have higher life time of polymer behaviour after proceed in higher tonnage injection condition as cited by Sichina (2015) which report that the data of TGA decomposition information can be used to predict the useful product lifetimes of some polymeric materials [8].

![Table 2: The composition and major decomposition temperature of resin, sample of gear for higher tonnage and gear with lower tonnage processing method](table2.csv)
The chemical composition of gear with both higher and lower tonnage injection process are comparable based on their moisture and VOC content, polymeric material content and the absence of filler. The result showing that the chemical composition of pure resin and gear (higher tonnage injection) are comparable except for significantly higher moisture and VOC content in pure resin (0.120%) as compared to 1.264% for sample of gear that undergoes the process of higher tonnage injection molding condition.

Different machine setting were examined in order to confirm the effect of the processing conditions and to discover to what extent degradation takes place during processing. Additionally, different injection moulding geometries and their influence on the degradation behaviour were compared. Fig. 3, 4 and 5 compare the results of the Resin (mixing), sample Gear 1 and Gear 2 each after variation of the processing conditions. It can be shown that POM copolymer in Fig. 3 until Fig. 5 shows onset line indicating some chains that are affected due to the higher temperature [2]. Application of the different processing methodology, for example increasing the injection moulding tonnage shows a decreased onset temperature corresponding to a degradation of POM. It is also reveal from the TGA investigations in nitrogen atmosphere with 50% humidity for 3 hours at room temperature, reveal that there is an influence of the used mould geometry on the degradation behaviour of POM copolymer (Fig. 4 and Fig.5). On the other hand, the thermal stability reduce for all investigated materials extracted from structural components either pellets or specimens due to the processing methodology.

Although resin, Gear 1 and Gear 2 are made of comparatively same material base, their thermal stability which is reflected by their onset decomposition temperature is different. The onset temperature of the resin is 305.76 °C, Gear 1 is 313.61°C while Gear 2 is at 307.91°C. For such parts requiring optimum mechanical properties such as gear, it should be mold with high specific hold pressures. For this case, Polyoxymethylene (POM) is equally stiff and a lower flow material, so the injection mold clamp factor used must be towards the high side [3, 4, 5]. The holding pressure and holding time for POM have the highest influence on achieving high part mass. [7].

![TGA thermogram for pure Resin](image)

**Figure 3:** TGA thermogram for pure Resin POM
3.2. Differential Scanning Calorimetry (DSC)
Below table (Table 3) shows the data collection on the composition and major decomposition temperature of resin, sample of gear for higher tonnage and gear with lower tonnage processing method. The results are obviously shown that there are no significant difference was observed in these samples for peak melting temperature, onset crystallization temperature and peak crystallization
temperature. Other than that, there are no significant difference are recorded in peak melting
temperature, onset crystallization temperature and peak crystallization temperature was observed
between first heat cycle and second heat cycle for the same sample. The material is likely to be the
copolymer of POM based on its melting temperature of 168°C ~172 °C.

Table 3: The composition and major decomposition temperature of resin, sample of gear for higher tonnage and gear with lower tonnage processing method

| Heat Cycle | Samples ID | Melting Temp (°C) | Crystallization Temperature (°C) |
|------------|------------|-------------------|----------------------------------|
|            |            | Peak              | Onset                           |
| First      | Resin      | 168.03            | 147.11                          |
|            | Gear 1 (HT)| 169.03            | 147.81                          |
|            | Gear 2 (LT)| 169.82            | 148.51                          |
| Second     | Resin      | 171.63            | 147.29                          |
|            | Gear 1 (HT)| 171.45            | 147.44                          |
|            | Gear 2 (LT)| 171.68            | 148.35                          |

Figure 6: Comparison DSC Thermogram for melting temperature between resin, sample Gear 1 (higher tonnage injection mould) and Gear 2 (lower tonnage injection mould) - First Scan
Figure 7: Comparison DSC Thermogram for melting temperature between resin, sample Gear 1 (higher tonnage injection mould) and Gear 2 (lower tonnage injection mould) - Second Scan

Figure 8: Comparison DSC Thermogram for Crystallization between resin, sample Gear 1 (higher tonnage injection mould) and Gear 2 (lower tonnage injection mould)-First Scan
Figure 9: Comparison DSC Thermogram for Crystallization between resin, sample Gear 1 (higher tonnage injection mould) and Gear 2 (lower tonnage injection mould) - Second Scan

4. Conclusion.
Gear 1 (higher tonnage injection) and Gear 2 (lower tonnage injection) were prepared using POM copolymer material. They have comparable melting temperatures, crystallization temperatures and chemical compositions. However Gear 1 has slightly higher thermal stability than Gear 2 although the difference may not be significant due to the processing injection method condition and its geometries.

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