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Application News

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Thermal Analysis

Analysis of Resin Using FTIR and Thermal Analysis - "Silent Change" -

The act of part and material suppliers changing the composition of parts and materials without gaining the approval of their clients is known as "silent change." Product accidents resulting from such silent changes are increasing in recent years. This article introduces an example analysis of a case in which the material of a gear was changed.

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Measurement of a Resin Motor Gear

The shape of the gears that were analyzed are shown in Fig. 1. These two gears are made from the same type of resin, but after a certain period of use, the tooth profiles differed between the proper gear and the failed gear. The width of the top land of the failed gear has completely worn down. This analysis investigates the cause of this wear using Fourier transform infrared spectroscopy (FTIR) and simultaneous thermogravimetric analyzer (TG/DTA).

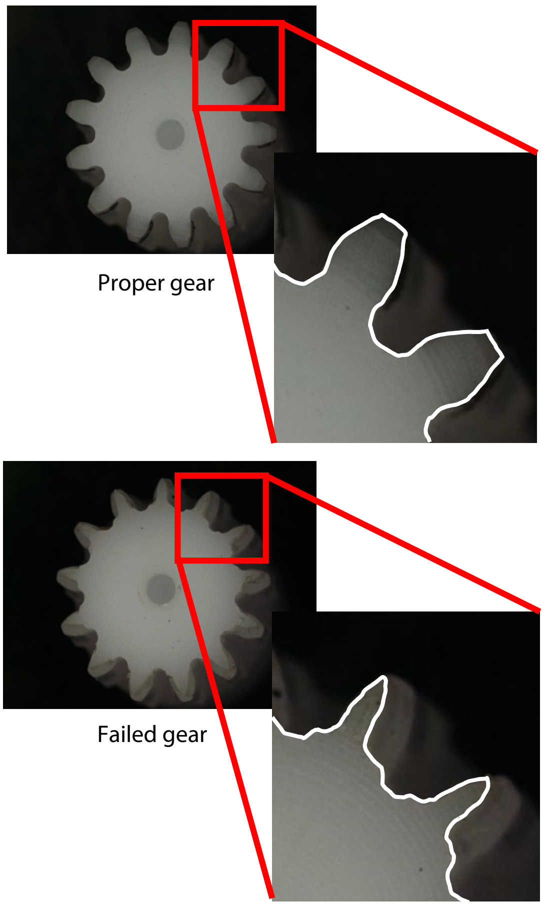


Fig. 1 Photographs of the Proper and Failed Gears

Analysis Results Using FTIR

Fourier transform infrared spectrophotometers (FTIR) are widely used for the determination of resins. We first measured the infrared spectra of the gears with an FTIR and compared the obtained spectra. Fig. 2 shows the FTIR measurement results. The infrared spectra of both the proper gear and the failed gear show a strong absorbance at about 1100 to 800 cm^{-1} caused by C-O-C stretching vibrations indicating that the resin is likely to be a polyacetal (POM). However, there is no significant difference between the two spectra.

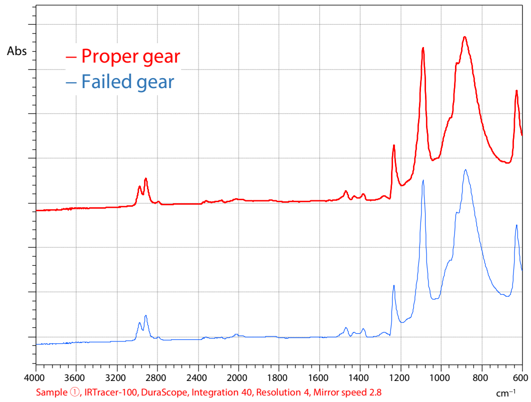


Fig. 2 FTIR Measurement Results

Analysis Results Using TG/DTA Measurement

The gears were next measured using a simultaneous thermogravimetric analyzer to compare their TG curves and DTA curves respectively. Fig. 3 shows the TG/DTA measurement results. The endothermic peaks at about 200 °C on the DTA curves indicate melting. The peaks show that the proper gear melted at 172.3 °C while the failed gear melted at 166.9 °C. The reason for the higher melting point of the proper gear is expected to be due to a higher crystallinity. The TG curves show that the failed gear starts decomposition at a higher temperature than the proper gear, indicating that the failed gear has a higher thermal resistance.

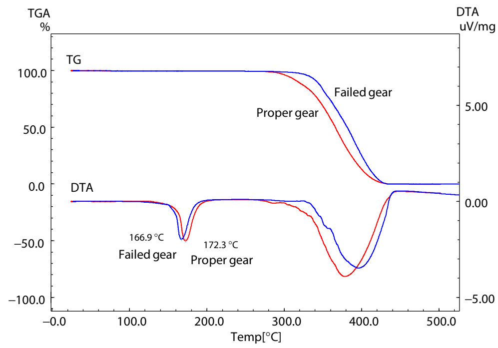
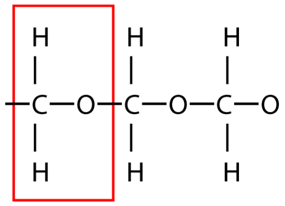


Fig. 3 TG/DTA Measurement Results

Types of Polyacetal

Polyacetals (POM) are a type of engineering plastic and there are homopolymer and copolymer versions. Fig. 4 shows the molecular structure of homopolymers and copolymers. The main chain of a homopolymer is comprised only of repeated oxymethylene units (-CH2O-). Since it is formed by the same molecules, the molecular chain becomes more dense and the crystallinity is high. The main chain of a copolymer includes C-C bonds and therefore crystallinity is lower compared to a homopolymer. However, the long-term thermal stability of copolymers is superior to homopolymers.

Homopolymer



Copolymer

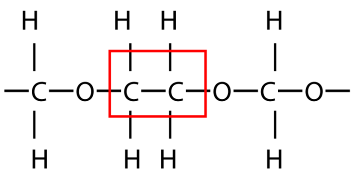


Fig. 4 Molecular Structure of Homopolymers and Copolymers

Conclusion

The most easy and reliable method for the determination of resins is to measure their infrared spectra. POMs can be easily identified by the strong absorbance at about 1100 to 800 cm^{-1} caused by C-O-C stretching vibrations. However, the differentiation between homopolymers and copolymers is difficult.[1] An effective method for differentiating these two is to check the melting point. The difference between melting points can be measured clearly by either differential scanning calorimetry (DSC) or DTA. In this research, we used FTIR to determine the resin type and then TG/DTA to identify the POM version. Analyses revealed that the proper gear is made of a homopolymer POM material and that the failed gear is made of a copolymer POM material. The conclusion of this research is that the wear of the resin motor gear occurred because the gear material was changed to a copolymer material which is inferior in mechanical strength.

Crystalline PET and Amorphous PET

We next measured two polyethylene terephthalate (PET) samples with differing properties of crystallinity using an FTIR. The infrared spectra of PET have crystalline and amorphous bands which are utilized in measuring their crystallinity. These bands are considered to be caused by the trans and gauche conformations of "-OCH2CH2O-".[2]

Fig. 5 compares the infrared spectra of each of the PET samples, showing that although there are differences at 1400 to 1300 cm^{-1} and 1100 to 800 cm^{-1}, the difference is minute. The samples were then measured using a differential scanning calorimeter to obtain their DSC curves (Fig. 6). For the crystalline PET, melting was observed at 250.3 °C whereas for the amorphous PET, glass transition occurred at 79.1 °C, crystallization at 159.6 °C and melting at 249.5 °C. These results show that DSC is an effective method for identifying differences in properties of crystallinity which are largely dependant on forming conditions and thermal history.

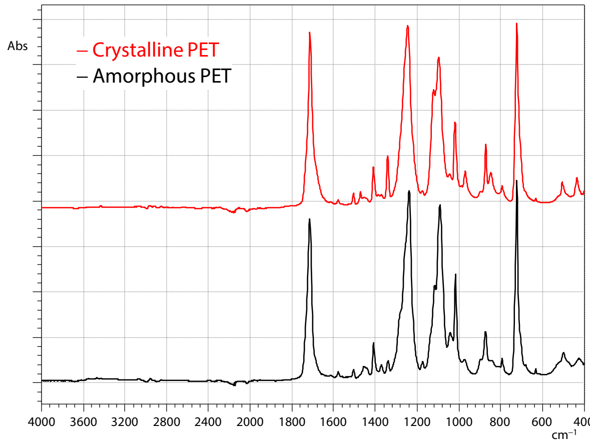


Fig. 5 FTIR Measurement Results

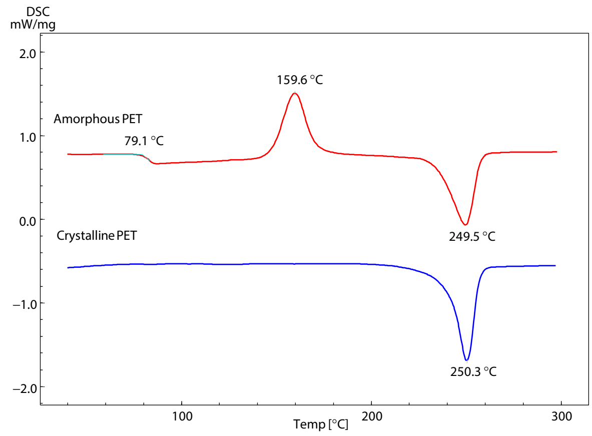


Fig. 6 DSC Measurement Results

References
1) Handbook of Polymer Analysis, p. 481-482
2) Handbook of Polymer Analysis, p. 903

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