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FAILURE ANALYSIS OF A GLASS FIBER REINFORCED THERMOPLASTIC AUTOMOTIVE PART

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Abstract: The present paper shows the failure analysis of an automotive heater control valve. It has been applied the Castro and Fernandez methodology. The strategy used has been the problem definition, material studies, like calorimetric trials, through microscopy and macroscopic examination. It has been studied sequence of failure, possible causes and mechanism of brittle breakage. Also we has simulated residual stress and fiber orientation in order to analyze effects of design and manufacturing on breakage. All this leads us to evaluate the mechanism of the part failure.. **Keywords:** failure, morphological fracture, thermal analysis, pyrolysis

1. INTRODUCTION

Twenty years ago the automobile industry suffered a considerable change in terms of the construction of its vehicles. The objective was to create something faster which also more ecologic. This objective can be achieved in two, distinct ways: Designing the engine better, by optimizing the combustion of fuel and programming the regularization of all of the systems which obstruct the movement of the vehicle. Another way is to achieve the highlighted objective through the reduction of the weight of the vehicle by substituting metal parts for parts made of polymeric material.

The plastic components used in engine fabrication present many problems, for this reason all parts should comply with a series of minimum requirements. They are already subjected to extreme working conditions:fuel pressure, high temperatures, and contact with oils. Under these conditions, the parts are designed and made with technical polymers or engineered, which present better properties than the commodities. Furthermore, these requirements should be assured. At present, we made an analysis failure of a refrigeration system component who has suffered a partial break in of one of its parts. This break, in turn, causes the loss of refrigerating liquid and consequently an important increase in engine temperature and its subsequent breakdown. This type of break is usual in vehicles between 5 to 10 years old and it is interesting to know the causes in order to prevent any future problems. The methodology employed [1] begins with problem definition, obtain background information, investigation of evidence (macroscopic and microscopic examination, including sem), evaluation of failure mechanism, sequence and causes and finally perform polymer testing [2,3]. Finally a compilation of this information will be used in the analysis.

2. EXPERIMENTAL

Morphological of surface were analyzed by Scanning Electron Microscopy with a HITACHI S¬3000N Scanning Electronic Microscope (Hitachi Ltd. Japan).

Calorimetric analysis was carried out using DSC Mettler-Toledo 821 equipment (Mettler-Toledo, Schwerzenbach, Switzerland). Weight samples between 6 and 7 mg were used. A heating (30 - 280 nitrogen environment) environment (flow rate 50 mL min-1).

TGA was carried out using a Mettler-Toledo TGA/SDTA 851 (Mettler-Toledo Inc, Schwerzenbach, Switzerland) with initial temperature 50° C and final temperature 800° C using a 20 °C min-1 heating rate, in a nitrogen atmosphere (20 ml min-1). The samples used weighed 5 mg approximately.

All samples were pyrolyzed with the use of a pyrolysator (Pyroprobe 1000, CDS Analytical, Inc., Oxford, Pennsylvania), which was interconnected to a gas chromatography/mass spectrometry apparatus (6890N, Agilent Technologies, España S.L., Madrid, Spain) equipped with a 5973N mass selective detector (Agilent Technologies). The sequential was programmed at 40 °C for 5 min, followed by a stepped increase of 5 °C min-1 to 300 °C, where it was held for 15 min. The gas used was helium with a 50:1 split ratio. The mass selective detector was programmed to detect masses between 40 and 650 amu. Samples (correct and failure zone) were pyrolyzed at 800 °C for 1 s.

3. RESULTS AND DISCUSSION

The part suffers temperature increases due to the natural functioning of the motor engine of a vehicle which are regulated by a refrigeration system. In this system, there is a fundamental part that regulates the flow of the refrigerating liquid from the engine chassis to the radiator and, logically, its return. This part is made with a mixture of PPE/PA, reinforced by ether polythene polymer technology (PPE). This mixture allows us to achieve the PPE properties together with the chemical resistance, stability, dimensions, low absorption of water and heat resistance and fluidity of nylon polymer. The result is a material of excellent thermal performance, extraordinary chemical and impact resistance [4, 5]. In order to increase mechanic properties in a major way an additional material containing 30% glass fiber is required [6].

Firstly we started with a visual study of the part in order to present a hypothesis of the break causes. Figure 1 shows the break of one of the connections between the valve and the refrigeration system. The microscopic study of the break in this connection indicates that a fragile break has occurred where no type of deformation could be found in the plastic originally.



Figure 1: Break brittle part

In the image, small glittering specks can be seen in the fracture which are present due to the use of the glass fiber to improve the mechanical properties of the polymer The observation of the part poses possible causes of the fracture [6], the break could be caused by a fault in the material such as degradation phenomenon in its environmental surroundings or the effect of refrigerating liquid that passes through the part.

A correct and failure zoneof fractured surfaces provides information about interaction of fiber and matrix. Failure and correct zones show significant differences. The failure surface zone shows low adhesion between glass fibers and matrix. We can see the fibers separated from the matrix (Figure 2).



Figure 2: Fiber adhesion in failure zone

The next step has been the 3D simulation of residual stress and fiber orientation. The different simulation images don't' show weld lines near of the failure zone, this is due to various slide existence. The piece design reduces the potential effect of welding line and air trapping (figure 3).



Figure 3: Weld lines and volumetric shrinkage

Additionally, fiber orientation study show normal parameters in orientation tensor values (Figure 4). To corroborate the results of the simulation we have prepared a sample of the failure zone using the method of Bay, RS [7]. The image analysis show typical fiber distribution in the frozen and core layer, where we can see fiber orientation in the flow direction for frozen layer and transverse orientation in the core layer. These results allow us discard the orientation fiber as a trigger for the rupture of the piece.



Figure 4: Fiber orientation

Initially, a fault in the material involved a study by means of diverse thermal analysis techniques which allowed us to locate both the changes in different thermal transitions and the location of the degradation phenomenon. The thermal analysis technique, such as the different scanning calorimetry (DSC) and the thermogravimetry (TGA), allowed us to observe the variations in the thermal transitions and the way in which they determine the cause of the break in the analyzed part.

In order to observe differences in the area of the break, we analyzed the internal part (the part which makes contact with the refrigerating liquid) as well as the external part by using the above-mentioned techniques. Figure 5 shows the calorimetric curves in the analyzed zones (failure zone and correct zone), in both cases an endothermic peak corresponding with the fusion of the corresponding spherulites can be found along with the presence of polyamide in the polymer.



Figure 5: Comparative DSC of failure zone

On the other hand, the calorimetric study shows no significant differences in terms of the failure zone and correct zone, including the enthalpy fusion which is very similar in both zones (enthalpy energy: failure zone 20'1 J g-1 and correct zone 22'1 J g-1). The EPP has a vitreous transition temperature (Tg) of about 217 degrees celsius. If variations in Tg exist they are unobservable due to the presence of the PA fusion. The thermogravimetric curves show significant differences between failure zone and correct zone. The degradation process starts earlier in the fractured zone, where the values of the temperature of the beginning and end of the degradation are lower (Tzi = 350 °C -Tzf = 490 °C) that correct zone (Tzi = 390 °C -Tzf = 512 °C). By the other hand, failure zone show two jumps in the thermogravimetric curve which correspond with the thermal degradation of the materials which form the blend (PPE/PA¬GF30). The first jump, which occurs at approximately 350° C and finishes at 440° C, suggest a loss in weight of around 35% in total. This first jump is associated with the PA thermal degradation [8](Figure 6).



Figure 6: Thermal degradation

After this jump a second process of degradation is initiated associated with the PPE [9] which finishes at around 480 °C with a loss of 25%. At the end of the degradation, 40% of the weight is left which corresponds with the presence of glass fibre in the material, first derivate TGA curve show these phenomenon more easily.

3. CONCLUSION

A lot of parts has been designed to perform their function perfectly, but sometimes do not consider the effect of degradation phenomena under certain conditions. Fracture morphology analysis indicates a cohesion loss between the fibers and the polymer matrix, which indicates some degradation in the matrix. Injection simulation process and fiber orientation study allow us discard the piece design and injection process as failure origin. However, analysis techniques show significant differences between correct zone and failure zone. Enthalpy of fusion is used to observed degradation phenomena, although in this case are significant differences between the correct and failure zone. Moreover, the results obtained using the thermogravimetric analysis indicate that the degradation occurs in two phases, corresponding to Polyamide phase and another, later, Poly ethylene phenyl phase. These results corroborated that an increase of cyclopentanone in the failure zone. There is every indication that hydrolysis ageing of polyamide phase produce failure process.

REFERENCES

[1] de Castro P, Fernandes AA. Methodologies for failure analysis: a critical survey. 2004;25:117-123

[2] Yousefi AM, Collins P, Chang S, DiRaddo RW. A comprehensive experimental study and numerical modeling of parison formation in extrusion blow molding. 2007;47:1-13

[3] Wintle JB, Pargeter RJ. Technical failure investigation of welded structures (or how to get the most out of failures). 2005;12:1027-1037

[4] Dunn RF, McSwain RH, Mills T, Malone B. Failure of plastic press release buttons in automobile seat belts. 2005;12:81-98

[5] Loyd E, White JR. Photo-sensitivity of recycled photo-degraded polypropylene. 2002;21:627-629

[6] Moura GCR, Aguilar MTP, Pertence AEM, Cetlin PR. The failure analysis of a deep drawing die in the manufacturing of an automotive shock absorber cap. 2004;11:943-950

[7] Bay RS, Tucker CL. Fiber Orientation In Simple Injection Moldings.2. Experimental Results. Polym Composite 1992;13:332-341

[8] Nielsen M, Jurasek P, Hayashi J, Furimsky E. Formation Of Toxic Gases During Pyrolysis Of Polyacrylonitrile And Nylons. J. Anal. Appl. Pyrolysis 1995;35:43-51

[9] Stack S, O'Donoghue O, Birkinshaw C. The thermal stability and thermal degradation of blends of syndiotactic polystyrene and polyphenylene ether. 2003;79:29-36

[10] Parres F, Sanchez L, Balart R, Lopez J. Determination of the photo-degradation level of high impact polystyrene (HIPS) using pyrolysis-gas chromatography-mass spectrometry. J. Anal. Appl. Pyrolysis 2007;78:250-256

[11] Faravelli T, Pinciroli M, Pisano F, Bozzano G, Dente M, Ranzi E. Thermal degradation of polystyrene. J. Anal. Appl. Pyrolysis 2001;60:103-121

[12] Parres F, Balart R, Crespo JE, Lopez J. Effects of the injection-molding temperatures and pyrolysis cycles on the butadiene phase of high-impact polystyrene. 2007;106:1903-1908

[13] DiPasquale G, LaRosa AD, Recca A, DiCarlo S, Bassani MR, Facchetti S. Pyrolysis of glass-hardened polyamides used in the automotive industry. 1997;32:3021-3024

[14] Takayama S, Mathubara N, Arai T, Takeda K. Rearrangement of the main-chain and subsequent thermal degradation of polyphenylene-ether. 1995;50:277-284

[15] Kinoshita M, Nemoto T, Souda T, Takeda K. Semi-quantitative analysis of thermal degradation in polyphenylene-ether. 2000;68:437-443

[16] Thomason JL. Structure-property relationships in glass-reinforced polyamide, Part

3: Effects of hydrolysis ageing on the dimensional stability and performance of short glass-fiber-reinforced polyamide 66. Polym Composite 2007;28:344-354

[17] Choi SS. Correlation of crosslink density with pyrolysis pattern of natural rubber vulcanizates with efficient vulcanizing cure system. 1999;52:105-112