

Creep and Stress Relaxation Evaluation of Virgin and Thermally Aged Glass-Filled Poly(butylene terephthalate) used in Automotive Electrical Connector Applications for Electrically-Powered Vehicles by Dynamic Mechanical Analysis

Robert A. Smith and Mikel Petty
Delphi Corp.

ABSTRACT

Poly(butylene terephthalate) stock with 0, 15, and 30% glass fiber filler, used in the housings of electrical connectors on hybrid and electric vehicles, was examined by creep and stress relaxation for viscous deformation and modulus loss at 25, 75, 125, and 150°C. Sets of virgin and aged samples were examined within each set then compared to each other.

Both the virgin and aged samples showed little viscous deformation or modulus reduction at 25°C, which was below the T_g of 40°C. Thermoplastic creep and stress relaxation was observed at the temperatures above T_g with, expectedly, more sample distortion and stiffness loss observed at higher test temperatures and lower filler contents both in virgin and unaged samples.

Aged samples became stiffer, and more resistant to irrecoverable strain and stress relaxation than virgin samples indicating connectors to have a “self-healing” aspect through in-service use at elevated temperatures. This phenomenon was noted in literature and verified as arising from thermally-induced secondary crystallization in the aged samples by observation of increased crystalline content using DSC; and analysis of the variation of storage modulus vs. temperature in the aged vs. virgin samples.

CITATION: Smith, R. and Petty, M., "Creep and Stress Relaxation Evaluation of Virgin and Thermally Aged Glass-Filled Poly(butylene terephthalate) used in Automotive Electrical Connector Applications for Electrically-Powered Vehicles by Dynamic Mechanical Analysis," *SAE Int. J. Mater. Manf.* 8(3):2015, doi:10.4271/2015-01-0603.

INTRODUCTION

Sealed connections are critical for the safe operation of all vehicles; particularly with the high voltages (350V) of HEV and EVs [1]; where they must withstand long term exposure to temperatures up to 150°C [2]. Leakage of water or petroleum-based contaminants into connections could cause immediate cessation of operation risking a catastrophic vehicle malfunction.

Sealed connectors consist of various designs which require a sufficient hold force between the male and female components to enable gasket sealing as shown in Figure 1:

The components of the connector are held together by compression of tabs usually made of the same polymeric material as the connector body. Loss of hold force or distortion from viscoelastic flow of polymer chains may lead to leakage through the gasket resulting in electrical failure.

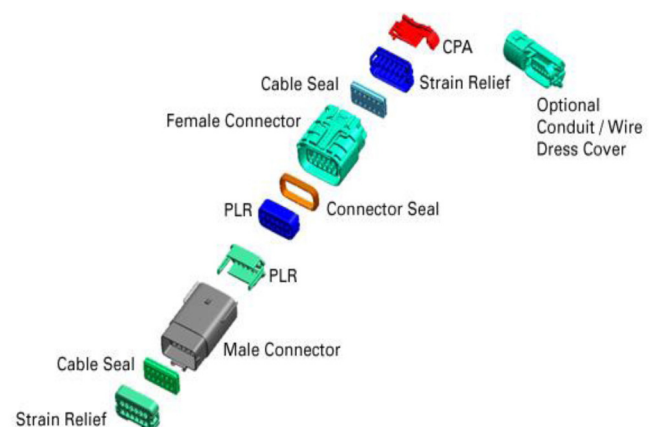


Figure 1. Schematic of a Sealed Connector

Poly(butylene terephthalate), PBT, has been a choice material in construction of electrical connectors due to the combination of good mechanical, electrical and chemical resistance properties and ease of molding [3]. It is often filled with glass fiber to improve stiffness and impact properties [4] and is normally used below the recommended

service range limits of 150 to 200°C. Since it is thermoplastic (not crosslinked), the molded items are subject to viscoelastic flow, particularly at high temperatures.

Dynamic Mechanical Analysis (DMA) can assess the tendency of materials to undergo viscoelastic flow through creep and stress relaxation analysis; where step stress and strain are applied to the samples, respectively.

A creep experiment measures the sample strain vs time after application of a fixed stress (creep deformation); then monitors the sample strain recovery after removal of the stress (creep recovery). Creep deformation would simulate the stress placed on both halves of a sealed connector by wire and cable tension. A generalized schematic is shown in Figure 2:

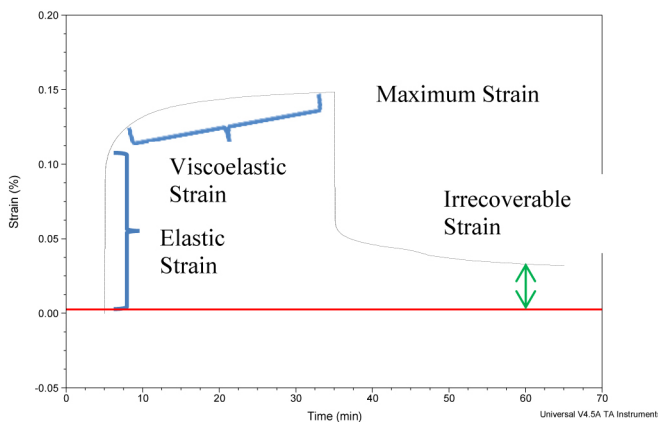


Figure 2. Schematic of a Creep Response

The maximum strain reflects the combined elastic and viscoelastic response (creep deformation) that occurred during application of the step stress, which was removed at 35 minutes. The strain descending portion of the curve at >35 minutes represents the creep recovery provided by polymer chains which have not flowed but merely extended their conformations as a response to the step stress. The irrecoverable strain represents permanent change in the sample caused by chain flow [5].

PBT with 0, 15, and 30% glass fiber was analyzed for maximum and irrecoverable strain during DMA creep experiments conducted at various temperatures on virgin and thermally aged samples (480 hrs at 150°C). The tests simulated tensile forces the connectors would experience from wire and cable tensions.

Stress relaxation imparts a step strain to the sample; then monitors the reduction in modulus as the chains entropically relax and reduce the stress. The reduction of stress is expressed by the general plot of relaxation modulus [$\sigma(t)/\epsilon_{\text{step}}$] vs time as shown in Figure 3:

The reduction in modulus would be reflected in hold force reductions in sealed terminals with strained tabs and could lead to loss of water-tightness. Ideal results would result in no reduction in relaxation modulus; which would only be obtained with materials of perfect elasticity such as a metallic spring.

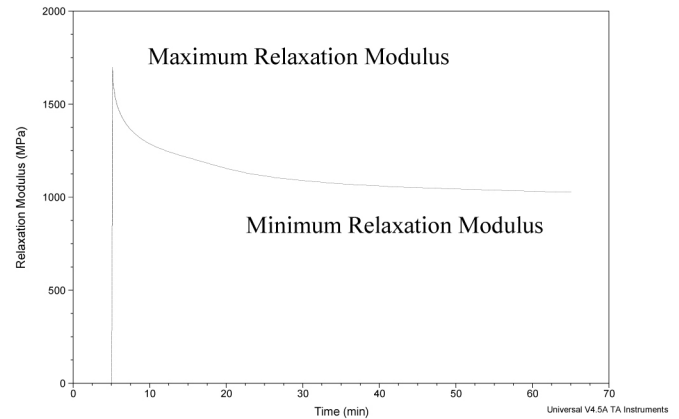


Figure 3. Schematic of a Stress Relaxation Response

Stress relaxation studies were conducted on the same set of PBT samples described previously utilizing maximum and minimum relaxation moduli.

EXPERIMENTAL

Polybutylene terephthalate of various fiberglass filler contents was purchased from Sabic Innovative Plastics, Wixom, Michigan and are described below:

Table 1. Characteristic Physical Properties of PBT Samples

Sample Name	Glass Fiber Content %	Density (g/cc)	Melt Flow Rate (g/10 mins.)
PBTGF0	0	1.29	52.5
PBTGF15	15	1.41	37.5
PBTGF30	30	1.50	35

The samples were injection molded into tensile specimens. A portion of the samples were aged at 150°C in a Thermo Scientific Heratherm forced-air oven for 480 hrs. DMA test specimens were cut from the neck portion of the tensile dogbone.

Samples were tested in either creep or stress relaxation analysis on a TA Instruments Q800 Dynamic Mechanical Analyzer (DMA) at 25, 75, 125, and 150°C. Creep samples were tested at 0.1N preload force and 2.0 MPa step stress with a 5 minute temperature soak. The samples were exposed to the step stress for 30 mins, followed by a 40 minute creep recovery after removal of the stress.

Stress Relaxation tests were conducted at 0.1N preload force and 2% step strain, also with a 5 minute temperature soak. The samples were strained for 65 minutes with continuous monitoring of the relaxation modulus.

Storage modulus vs. temperature plots were obtained using DMA analysis at 1 Hz and 20μ strain amplitude. The samples were cooled to -60°C, then heated at 5°C/min. to 250°C.

Samples were analyzed by Differential Scanning Calorimetry (DSC) on a TA Instruments Q2000 Modulated DSC by heating from 30 to 300°C at 10°C/min. under nitrogen atmosphere. Melting points were

obtained as the minima in the downward melt endotherms. Melt enthalpies, obtained by integration of the melt endotherms, were used to calculate crystalline contents by dividing by the theoretical melt enthalpy of 100% crystalline PBT of 96 J/g [6].

RESULTS AND DISCUSSION

Sabic reported tensile creep data on PBT with various glass fiber reinforcement [7], however the samples had applied step stresses of 3-12 MPa which were likely much higher than connectors would encounter in automotive applications; and they did not include the creep recovery. Step stresses were limited to 2.0 MPa in this study.

Reproducibility was checked by creep analysis of the maximum and residual strains of three samples of unfilled, virgin PBT at 75°C, as shown in Table 2:

Table 2. Creep Analysis of Three Samples of Unfilled Virgin PBT at 75°C

Sample #	Maximum Strain %	Irrecoverable Strain %
1	.972	.248
2	.910	.213
3	.980	.227

Creep results of PBT with 0, 15, and 30% glass fiber content at 25, 75, 125, and 150°C are shown in Figures 4, 5, 6, respectively.

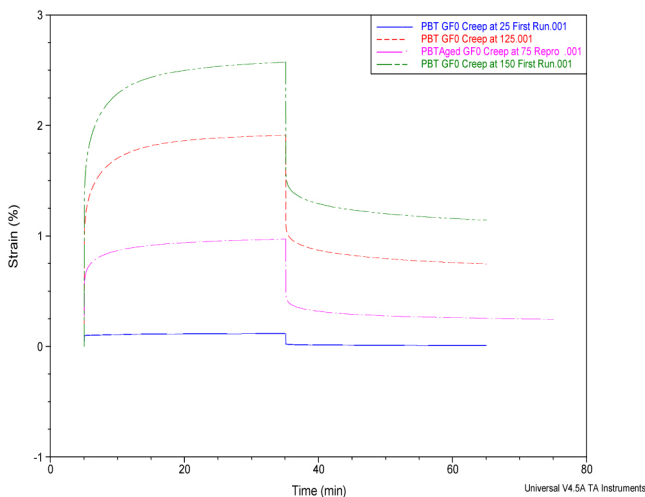


Figure 4. Creep Analysis of Unfilled, Virgin PBT

Expectedly, the amount of viscous flow and irrecoverable strain increased with reduced filler content and increased temperature (note scaling). Samples examined at 25°C were below T_g and little viscoelastic response was found due to low chain mobility [8].

Creep Analysis of the thermally aged samples is shown in Figures 7, 8, 9 for 0, 15, and 30% glass fiber filled samples, respectively. The scaling was made identical to the virgin sample plots to better visualize changes which occurred as a result of thermal aging:

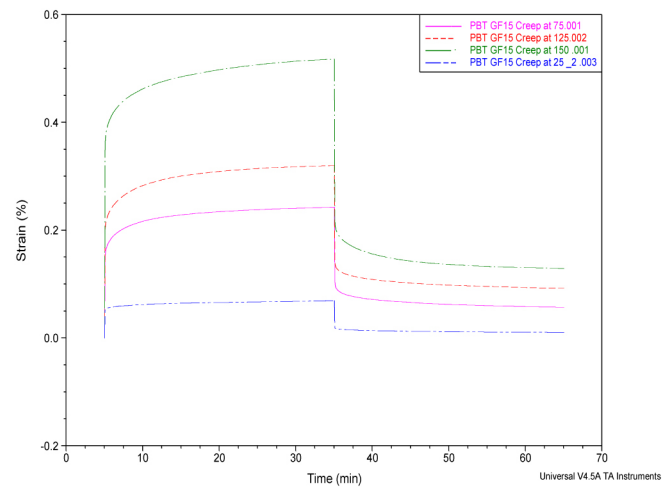


Figure 5. Creep Analysis of Virgin PBT with 15% Glass Fiber

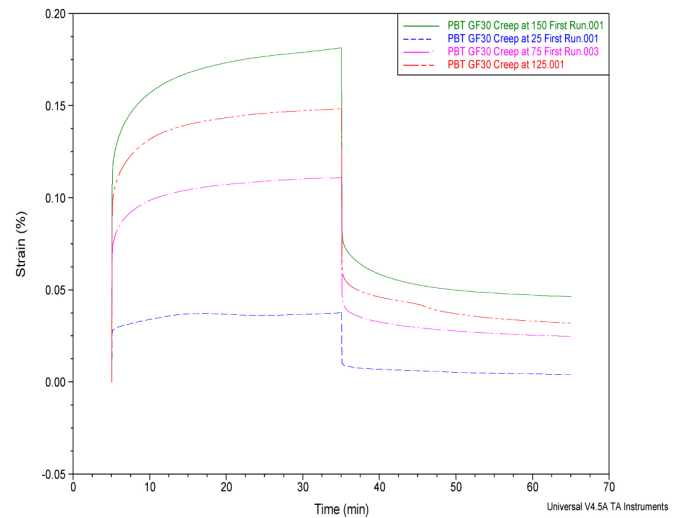


Figure 6. Creep Analysis of Virgin PBT with 30% Glass Fiber

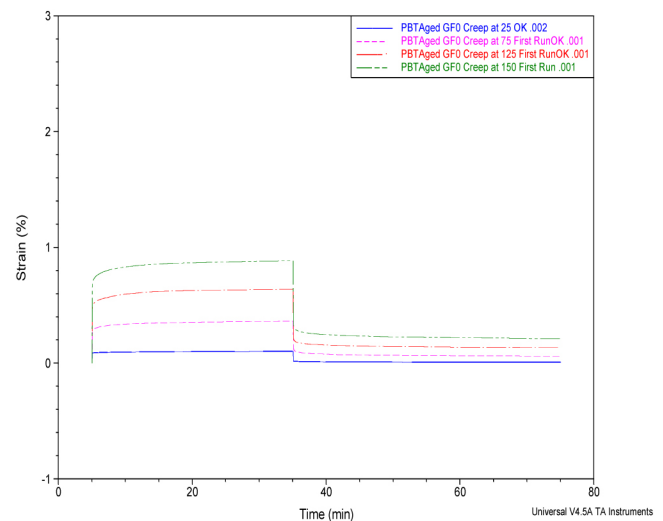


Figure 7. Creep Analysis of Aged, Unfilled PBT