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Damage mechanisms in a short glass fiber reinforced polyamide under fatigue loading

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ABSTRACT

This paper presents a damage investigation on a short glass fiber reinforced polyamide (PA66-GF35) under fatigue loading. Plain and notched specimens were tested at room temperature and humidity with load ratio R = 0. Electron microscopy was used to analyze the fracture surface of failed specimens and the crack path of specimens subjected to interrupted fatigue tests. Damage mechanisms were studied investigating the following fractographic features: ductile/brittle matrix fracture behavior, fiber failure/pull-out, degree of fibermatrix interfacial adhesion. The aim of the present paper is to understand the nature of damage initiation and propagation in order to lay the foundations for the development of a multi-scale, mechanism-based lifetime prediction model for short fiber reinforced plastics.

1. Introduction

In the car engine compartment, Short Fiber Reinforced Plastics (SFRPs) are increasingly replacing metals in structural applications. High strength to weight ratio, high temperature and chemical resistance make these materials excellent candidates for under-the-hood applications. The competitiveness of SFRPs is related to the advantages offered by the injection molding process. This technology enables complex geometries to be manufactured at high production rates without the need for additional post-molding machining operations. The durability assessment of SFRPs plays a key role in the design of injection molded structural parts. Typical under-the-hood parts such as fuel rails, pump housings and sealing elements are exposed to cyclic loading due to vibrations, pulsating forces, temperature variations. Lifetime prediction models aim to reduce the number of recursions before the series production by virtually optimizing the product in the early stages of the project. The lifetime estimation of SFRPs is accurate when the damage mechanisms are understood and properly integrated in the model. In SFRPs, damage mechanisms occur at a length scale below the macroscopic scale due to the interactions between the material constituents (fiber, matrix, fiber-matrix interface). Multi-scale models aim to bridge the scale at which the damage mechanisms occur and the macro-scale of structural applications. The first step in developing such a model is an extensive damage investigation.

The aim of this work is to investigate the fatigue damage mechanisms in plain and notched short glass fiber reinforced polyamide specimens in order to lay the basis for the development of a multi-scale, lifetime prediction model. In the last 30 years, many papers were dedicated to the investigation of damage and failure mechanisms of SFRPs under fatigue loading. Despite this, it is common to use phenomenological approaches for the lifetime prediction of SFRPs [1–3]. However, such approaches do not take into account the damage mechanisms occurring in the material due to cyclic loading. With this paper, the authors want to identify the relevant damage mechanisms, relate them to the length scale at which they occur and order them chronologically. The development of a model can be described in six steps: (i) Determination of the condition for the initiation of the first event of damage; (ii) description of the damage evolution; (iii) damage quantification; (iv) analysis of the material response due to the effect of damage (stiffness-damage relationship); (v) definition of a criterion that identifies the criticality condition of a damage state with regard to the design function; (vi) integration of the model in the development strategy of a structural part. In this paper, the first two points are addressed. The focus is on damage initiation. The location of damage initiation and the mechanisms responsible for the first event of damage are deeply investigated by means of electron microscopy. Damage mechanisms during crack propagation are also described. Two specimen geometries are used with the aim to investigate the

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effect of the structural discontinuities on the damage mechanisms. In particular, the use of notched specimens for investigating the fatigue damage mechanisms of SFRPs is a novelty in the literature. Considering the first works in this field in the 1970s [4,5], the investigation techniques have taken huge steps in terms of accuracy of observation, image quality and development of new methodologies. Fractographic examinations by means of Field Emission Scanning Electron Microscope (FESEM) on a short fiber reinforced PBT were shown recently by Hoffmann [6]. Comparing the micrographs in [4,5,7,8] with those in [6], the improvement of the microscopy technique in terms of resolution, magnification, image quality becomes immediately evident. In Section 2, the authors reviewed the damage mechanisms from literature and sorted them based on specific fractographic features: ductile/brittle matrix fracture behavior, fiber failure, fiber pull-out and degree of fiber-matrix interfacial adhesion. The results from the literature were compared with the results of an extensive damage investigation carried out at Robert Bosch GmbH on plain and notched short fiber reinforced polyamide specimens (Section 5).

The damage investigation was conducted by means of three methods: (1) Infrared (IR) Thermography (Section 5.1); (2) microscopic investigation of the fracture surface (Section 5.2); (3) microscopic investigation of the crack path (Section 5.3). Damage mechanisms were investigated at multiple scales. Macro- meso- micro- nano-scale are frequently referred in the literature. Macro-scale is the scale of the specimen. At this scale, SFRPs are treated as homogeneous materials. The objectives of the macroscopic damage analysis are: (1) Localization of the crack initiation; (2) investigation of the failure mode of plain and notched specimens. The meso-scale is the scale of the Representative Volume Element (RVE) [9]. At this scale, fibers and matrix are treated discretely, whereas the microstructural variables (fiber orientation, fiber volume fraction, fiber aspect ratio) are used to globally describe the RVE [10]. The analysis at the meso-scale aims to study the influence of the fiber orientation on the crack initiation/propagation. The micro-scale is determined by the fiber diameter. At this scale, SFRPs are described as three-phase materials (fiber, matrix, fiber-matrix interface). Particular attention is given to the analysis of the fiber-matrix interface after the application of cyclic loading. Fracture in composite materials can be either cohesive or adhesive depending upon whether a layer of matrix adheres to the fibers or not. Modern electron microscopes allow the investigation of the fiber-matrix interface at such a level of detail that was impossible just a few years ago. The issue whether damage occurs at the fiber-matrix interface or in the resin, at a certain distance from the interface, is crucial for the development of a mechanism-based model. Clean fibers on the fracture surface indicate fiber-matrix debonding. Otherwise, the mechanism is matrix cracking and the analysis should be focused on the resin layer covering the fibers. During the fiber forming process, fibers are coated by a mixture of chemicals and water called sizing in order to improve the fiber-matrix adhesion. Fiber sizing can chemically and mechanically affect the resin layer at the fibermatrix interface leading to the formation of an interphase layer with different mechanical properties from the bulk matrix. Fiber-matrix bonding represents a distinguishing factor between materials of different suppliers. The understanding of the role of the fiber-matrix interface on the damage process and the integration of the related mechanisms in a lifetime prediction model would lead engineers to compare different materials but also the same material from different suppliers. For this reason, the authors believe that the analysis of the fiber-matrix interface should be included in the damage investigation particularly in view of the technical improvements in electron microscopy.

Finally, a preliminary analysis of the damage mechanisms preceding the initiation of micro-cracks is presented. This damage investigation aims to detect the first event of damage causing the material degradation under fatigue loading.

2. Literature review on the damage mechanisms investigated by means of electron microscopy

The first works on the damage investigation in SFRPs under fatigue loading date back to the 1970s and were motivated by the following reasons: (1) Comparing the fatigue behavior of different material systems; (2) improving the mechanical properties varying the material microstructure; (3) finding quantitative relationships between material failure modes and macroscopic material response. Historically, the damage investigation by means of electron microscopy has been based on two techniques: analysis of the fracture surface or of the crack path [4,5,7,11–13]. In the former case, specimens are tested until failure. In the latter case, fatigue tests are interrupted before failure and the side surface of the specimen is polished for microscopic investigations. These two analyses offer complementary and mutually enriching points of view. The degree of fiber-matrix adhesion, the pull-out length, the amount of broken fibers can be examined through both analyses. Instead, the observation of the fracture surface is better for investigating the matrix fracture behavior (ductile/brittle) while the analysis of the side surface of the specimen enables the study of the influence of the fiber distribution on the crack path. Both methodologies present some disadvantages. The analysis of the fracture surface requires the specimen separation into two parts limiting the access to the mechanisms responsible for the first event of damage. The observation of the side surface of the specimen limits the damage analysis to one planar section of the crack without any information on the damage mechanisms along the specimen thickness. A comprehensive analysis should consider multiple sections along the specimen thickness increasing the effort in the samples preparation.

Through the traces left by the crack on the fracture surface, it is possible to study the damage mechanisms occurring due to cyclic loading in a fatigue test. Hence in the following, we review the damage mechanisms reported in literature by reference to specific fractographic features: ductile/brittle matrix fracture behavior, fiber failure/pull-out, degree of fiber-matrix interfacial adhesion. Finally, the effect of the fiber distribution on the damage mechanisms is also reviewed.

2.1. Matrix fracture behavior

Evidence of ductility/brittleness on the fracture surface reflects the local mode of crack advance. Ductile matrix behavior was observed on the fracture surface caused by initiation and stable Fatigue Crack Propagation (FCP) [7,14–18]. Matrix yielding leads to the formation of polymer filaments on the fracture surface. The length of the polymer filaments depends on the material system, testing conditions (temperature, humidity), load level. Evidence of brittle matrix behavior on the fracture surface indicates that unstable crack propagation occurred. Horst and Spoormaker [14] compared the fracture surface of conditioned (1.5 wt.%) PA66-GF35 plain specimens, failed under static and fatigue loading. They observed a much larger ductile area in the latter case. Horst and Spoormaker [14] also compared the fracture surface of conditioned and dry-as-molded fatigued specimens, reporting a higher degree of the matrix ductility in the former case. Karger-Kocsis and Friedrich [20] observed that the degree of matrix ductility on the fracture surface of PA66GF30 specimens failed under fatigue loading increases with testing temperature. The same result was reported by Noda et al. [21] for a short glass fiber reinforced polyamide (PA66-GF33) tested under fatigue