TECHNIQUES AND RESULTS FOR FIBER LENGTH DISTRIBUTION DETERMINATION AS A FUNCTION OF THICKNESS IN LONG FIBER REINFORCED INJECTION MOLDED THERMOPLASTICS

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Abstract:

A novel measurement technique was developed to obtain unbiased fiber length distribution (FLD) measurements at specified locations in the thickness of the sample. This technique relies on elastic energy stored in long fiber thermoplastics (LFT), which is released during partially constrained burn-off. This release results in an increase of thickness dimension of the sample and partial disentanglement, allowing sample selection and subsequent filament separation. Quantitative FLD results and the measurement technique are discussed in detail. The FLD in long fiber reinforced injection molded thermoplastics is shown to vary as a function of thickness.

Introduction

In an effort to develop a suite of predictive tools for long fiber injection molded thermoplastics (LFT) [1,2], new experimental techniques are being developed to provide input data as well as validation of predicted results. A technique for measurement of fiber length distribution (FLD) has been described previously [3]. While the technique provides unbiased quantitative data, FLD measured via this technique provides only average FLD through the thickness of a sample. Based on qualitative observations made during these measurements, it was determined average FLD measurement may not provide suitable description of LFT microstructure. It is well established practice to describe fiber orientation results as a function of thickness in injection molded plastics [4,5] and it will be seen from the following discussion that skin, shell and core region (Figure 1.) of LFT materials contain not only varying fiber length orientation, but also varying FLD.

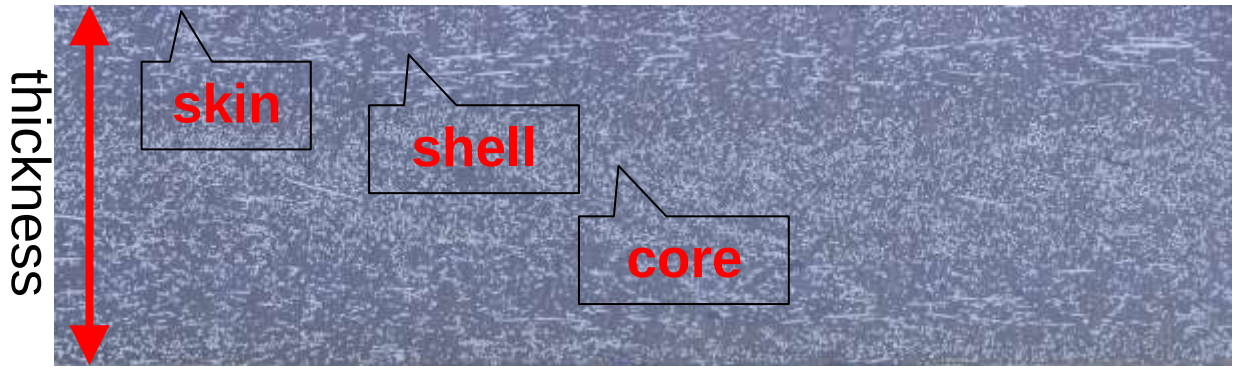

Figure 1. Cross-section of 3mm glass/polypropylene injection molded plaque with distinct skin, shell and core regions.

Initially uniform fiber length of LFT pellets is degraded to FLD containing a large amount of short fragments. This fiber length attrition occurs in the entire injection molding process and models for this phenomenon are not fully developed at this time. Reliable FLD measurement technique can therefore provide valuable input data to predictive tools as well as validation data for fiber length attrition models being developed.

A new procedure has been developed for fiber sample isolation during LFT measurement, that allows selecting fibers from a specific location in the plane of the part as well as specific location through the thickness of the part.

Procedure

General steps of the FLD measurement procedure are identical to those presented in [3]:

1. composite coupon isolation
2. constrained removal of matrix material
3. fiber sample isolation
4. filament dispersion
5. imaging and individual filament length measurement

The coupons are cut from locations specified as being at a certain position relative to the gate and beyond into the mold. Plaque thicknesses evaluated to date with this procedure are 3-mm and 5-mm. A brief description of the procedure follows.

The composite coupon is placed in a form whose inside dimensions correspond to the shape-and-size dimensions of the coupon (a little larger than 25.4-mm x 25.4 mm). The form is made from a perforated aluminum or steel sheet metal and provides restraint at the edges of the coupon during the first burn-off step.

The fiber form and its contents are sealed at the bottom with aluminum foil. A perforated sheet metal lid is placed over the top to allow gases of combustion to escape during the burn-off process. The assembled form, coupon and lid are placed in a muffle furnace for approximately 2 hours at 500°C. These conditions are satisfactory to consume the nylon (or polypropylene) matrix and leave behind bare glass fibers.

The form’s height dimension is greater than the coupon thickness to allow for expansion of the glass fiber reinforcement during matrix burn-off. This expansion is believed to facilitate the separation and spreading of the fiber filaments during the dispersal step that follows later in this procedure (Figure 2).

Figure 2. Aluminum form containing expanded mass of filaments after the first burn-off.

Form heights of 12.7-mm have been used with the 3-mm thick coupons with good results while form heights of 19.0-mm work well with the 6-mm thick coupons. The lofting (expansion) of the fiber mass depends on the quality and composition of the injection molded plastic. Expansions typically encountered have been increases of 250-400 percent greater than the as-molded plaque thickness.

The expansion can be characterized by x-ray imaging of tungsten fibers inserted into the sample before and after the burn-off. Figure 3 shows 0.01 mm thick tungsten wires inserted into a 5 mm thick sample. Due to the small dimensions involved as well as varying fiber content in the sample, it is difficult to drill straight holes for wire insertion.

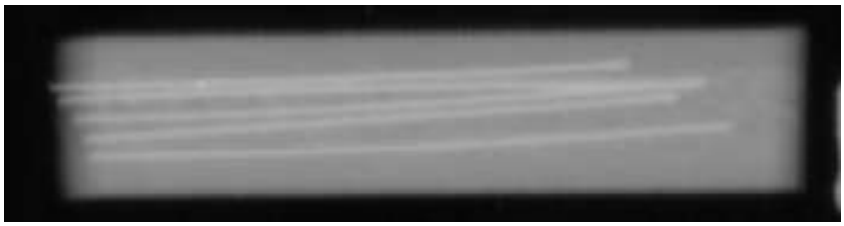

Figure 3. X-ray of 5 mm thick LFT sample with inserted tungsten wires.

Figure 4 shows expanded fiber mass with tungsten wires. It is clearly visible that the expanding filament mass carried the tungsten wires with it inside the aluminum form. Although wires that were added at the top and bottom of the mass rotated and moved, they stayed at the top and bottom of the mass. Wires in the thickness of the sample retained their relative position, albeit the distances between the wires changed.

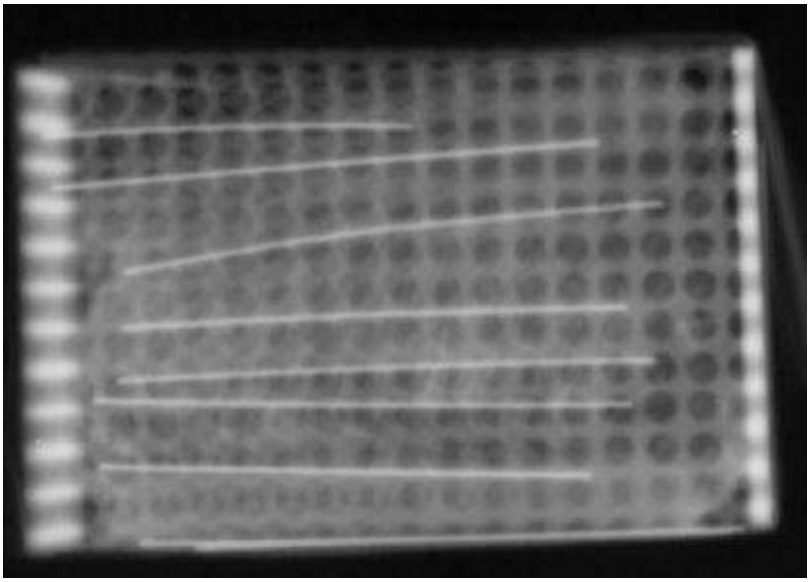

Figure 4. Expanded fiber mass containing tungsten fibers. Aluminum fiber form is clearly visible.

Position of wires in Figure 3 and Figure 4 can be measured and the nature of expansion can be characterized. Figure 5 shows a plot of wire positions through the thickness of the sample before and after burn-off. The expansion is approximately linear with the thickness of the sample increasing by 300%. This means that a location at a certain percentage of thickness in the expanded mass of fibers corresponds to a location at the same percentage of the initial thickness before burn-off.

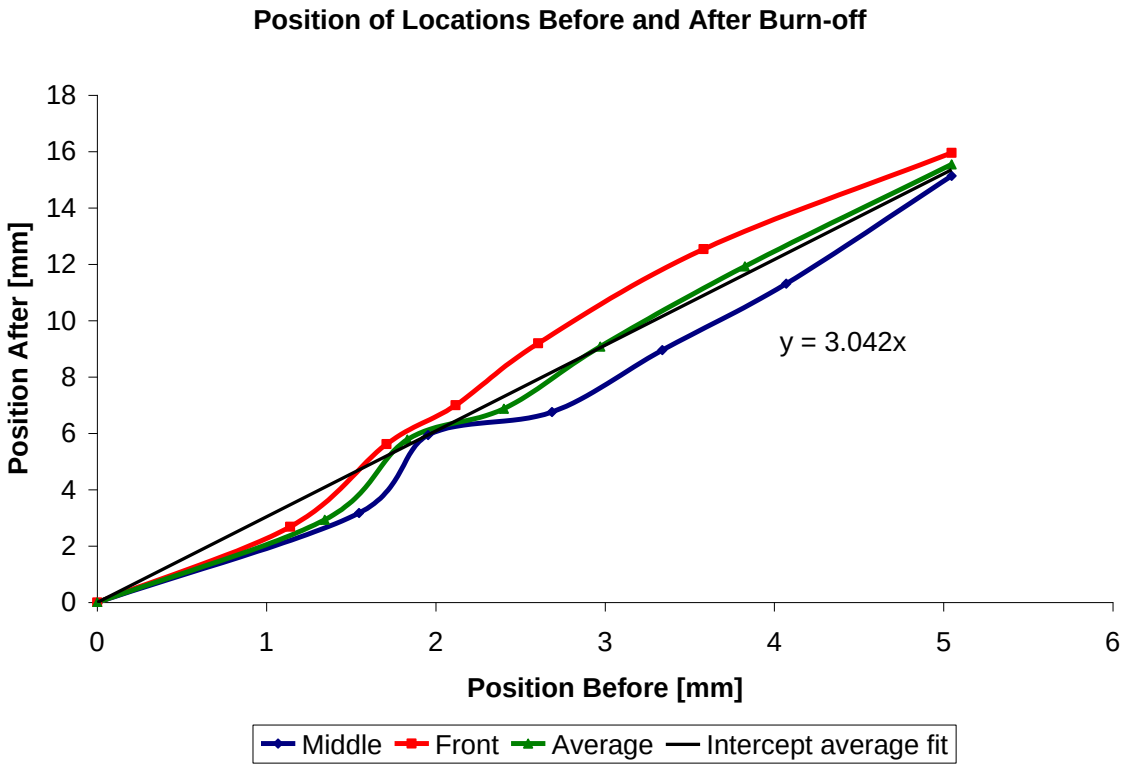

Figure 5. Plot of locations before and after burn-off showing linear nature of expansion.

Fiber expansion after the first burn-off is therefore uniform throughout the wall thickness of the coupon provided that there are no edge effects. For example, the fiber composition at the molded edges of the plaques tends to constrain the glass fiber mass from expanding relative to material further inward and the fibers from these coupons do not loft uniformly. Coupons cut from the edge of the plaques are therefore not suited to this procedure. This means that the fibers extracted from a discrete location through the fiber stack thickness can be correlated back to the fibers located at a proportional distance through the wall thickness in the molded plaque.

The fiber selection step involves isolating the central fibers of the residual fiber mass ("stack") for subsequent collection and characterization. Locations designated for harvesting fibers in this study are from the plaque surfaces, at a position corresponding to one fourth of the distance through the wall thickness from the plaque surface, and from a position at mid-wall thickness. Fiber "plug" samples for average FLD measurements are also extracted that contain a cross section of fibers from the entire through-thickness of the molded plaque (Figure 6). Notice that longer filaments appear to be trapped in the middle of the plug.

Figure 6. Extracted glass fiber-and-epoxy "plug" specimen

The fiber collection process is conducted using a hypodermic needle with a syringe loaded with a liquid epoxy: Epon 828 resin mixed with 55 phr Versamid 125 curing agent. The number of fibers collected from the specimens using these procedures is ultimately proportional to the epoxy volume dispensed by the hypodermic needle. Factors controlling the volume include the resin's viscosity, the needle gage size, duration of injection, etc. The mixed viscosity of Epon 828/Versamid 125 is sufficiently high to prevent it from spreading through (wetting) the fibers very far from the needle injection site, but still low enough to be dispensable through a hypodermic needle. In these experiments, a 21 gage-size needle has been found to work well with this resin system.

Collection of the plaque's surface fibers is conducted by dispensing a small bead of epoxy at a location corresponding to the center of the 25.4-mm x 25.4-mm coupon area and onto the

visible (exposed) fibers of the expanded fiber stack. The resin viscosity is sufficiently high that the glass fibers adhere to the resin bead. The uncured bead-and-adhered fibers are extracted by withdrawing the needle and are dispensed into an aluminum pan. Pumping a little excess resin from the syringe is sufficient to flush the sample from the needle tip and into the pan. A perforated sheet metal lid is placed over the top of the pan to allow gases of combustion to escape during a second burn-off process. The extracted sample, pan and lid are placed in a muffle furnace for approximately 2 hours at 500°C to consume the epoxy resin and leave behind bare glass fibers.

The perforated sheet metal lid is replaced with aluminum foil pressed lightly over the top of the form and fiber stack to prevent the fiber stack from shifting during handling and the form is turned over on its side during ollection of the fibers from discrete locations through the wall thickness. The needle is inserted at position that corresponds to one half of the stack height for the fibers to be extracted from the mid-wall position; and at a position that corresponds to one fourth of the stack height for the fibers to be extracted from the location at one fourth of the molded plaque wall thickness.

After insertion, a bead of resin is dispensed from the hypodermic needle into the fiber stack. Resin drop size (and number of fibers collected) is controlled by injection time duration. Between 2-3 seconds has worked well with this procedure. The hypodermic needle is then cut free from the syringe with wire clippers. Care is taken following this step not to disturb the needle remnant and the resin bead remaining embedded within the fiber stack (Figure 7).

Figure 7. Hypodermic needles inserted in the fiber mass at pre-determined locations.

The form containing the fiber stack designated to provide the through-thickness plug specimen is first readied by replacing the perforated sheet metal lid with aluminum foil pressed lightly over the top of the form and fiber stack to prevent the fiber stack from shifting during handling. The hypodermic needle is inserted through the top foil layer at a location corresponding to the center of the 25.4-mm x 25.4-mm coupon area and is pushed through the entire fiber stack thickness until the needle tip visibly protrudes through the foil layer at the opposite side.

The resin injection is conducted with the aid of a laboratory jack. The needle is clamped vertically in position to control the needle (and epoxy plug) orientation through the center of the specimen. The form containing the fiber stack is mounted to the base plate of the jack which is lowered at a constant rate to provide for a controlled withdrawal of the needle through the fiber stack thickness. Dispensation is controlled by pressing down on the plunger of the hypodermic

syringe manually with a constant force. The continuous stream of epoxy results in a column or “plug” of resin that extends through the entire thickness of the fiber stack.

Following injection, the forms containing the plug and through-wall thickness specimens are processed to cure the epoxy, thereby locking the collected fibers in place. The epoxy is first gelled overnight (~16-17 hours) at room temperature. The forms are then placed in an oven and the epoxy is cured for 2 hours at 85°C. Filaments in the vicinity of the epoxy are subsequently bonded in-situ to the resin beads or plugs for later separation and collection.

The bonded glass fibers are separated from the remainder of the fiber stack by loosening the foil layers and manually shaking the form to remove most of the un-bonded (“loose”) fibers from the sample. A final clean-up of the extracted fibers with short blasts of low pressure air removes any remainder, un-bonded fibers still clinging to the specimens.

The end of the needle remnant bonded to fibers collected at locations corresponding to one fourth and one half of the wall thickness can be grasped with tweezers for further manipulation of these samples. Figure 8 is a typical glass fiber specimen extracted from a discrete position through the wall thickness of a molded plaque.

Figure 8. Extracted glass fiber-and-epoxy specimen

The extracted fiber specimens are placed in aluminum pans with a perforated sheet metal lid placed over the top. These are placed in a muffle furnace for approximately 2 hours at 500°C for a second burn-off process to consume the epoxy resin, leaving behind the bare glass fibers collected from the specimen.

The dispersal process involves transferring the extracted glass fibers from the aluminum pan and into a glass petri dish. Remnants of the needles and aluminum foil layers are carefully removed using tweezers. The glass fibers can generally be separated and spread by gentle tapping of the petri dish. Manipulation to divide any fiber clumps is done manually. Wooden

sticks from cotton swabs work well for this purpose. The dispersal process is facilitated by using a microscope to visually ensure fibers are well separated and dispersed.

The petri dishes containing the dispersed fibers are placed on the bed of a scanner. Digital images of the glass filaments are scanned through the glass petri dish and into a computer file for later analysis. Details of obtaining unbiased FLD and representation of thereof can be found in [3]. Improvements to digital image analysis [6] to speed up measurement are being explored and will be discussed in the future.

Results

Results of first set of measurements using this technique are presented below. Figure 9 and 10 show FLD for 3 mm and 5 mm thick samples respectively measured at the skin, 25% of thickness and core of the sample. Measurements from the 25% thickness location should correspond to the shell region, although no attempt was made to determine the boundaries of the skin, shell and core region. Results presented in Figure 9 and Figure 10 contain no correction for short fibers or for bias towards long fibers. 2000 filaments were counted to produce each curve, except for the skin of 5 mm sample, where 657 filaments were counted. It can be observed from the plots that the average fiber length generally increases as the middle of the thickness is reached. While this is somewhat obscured on the 5 mm thick sample results, where only a few short fragments were counted for the skin, the pattern is visible regardless.

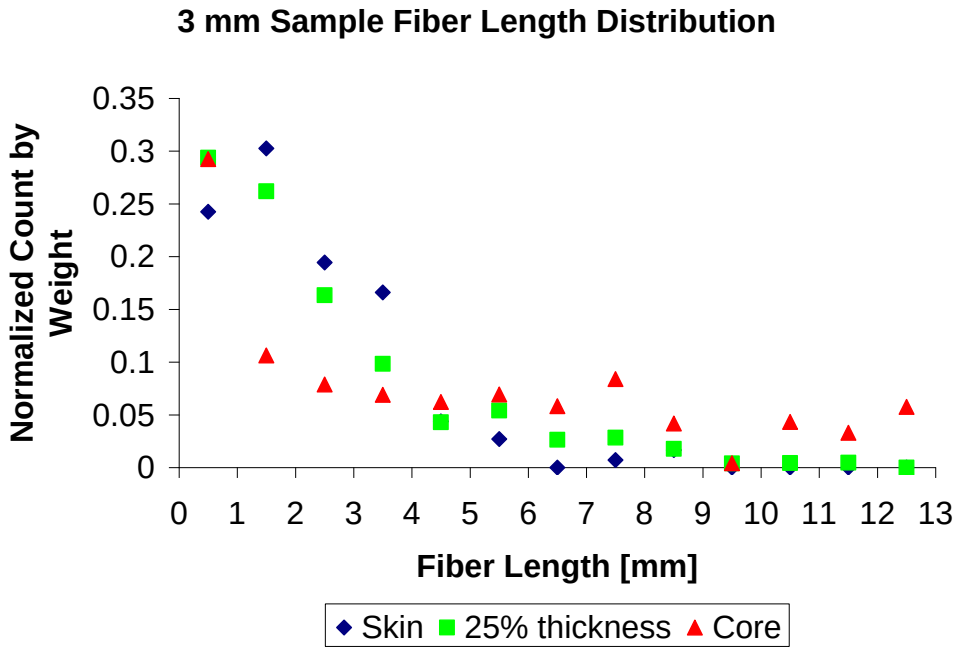

Figure 9. FLD for 3 mm thick glass/polypropylene LFT sample

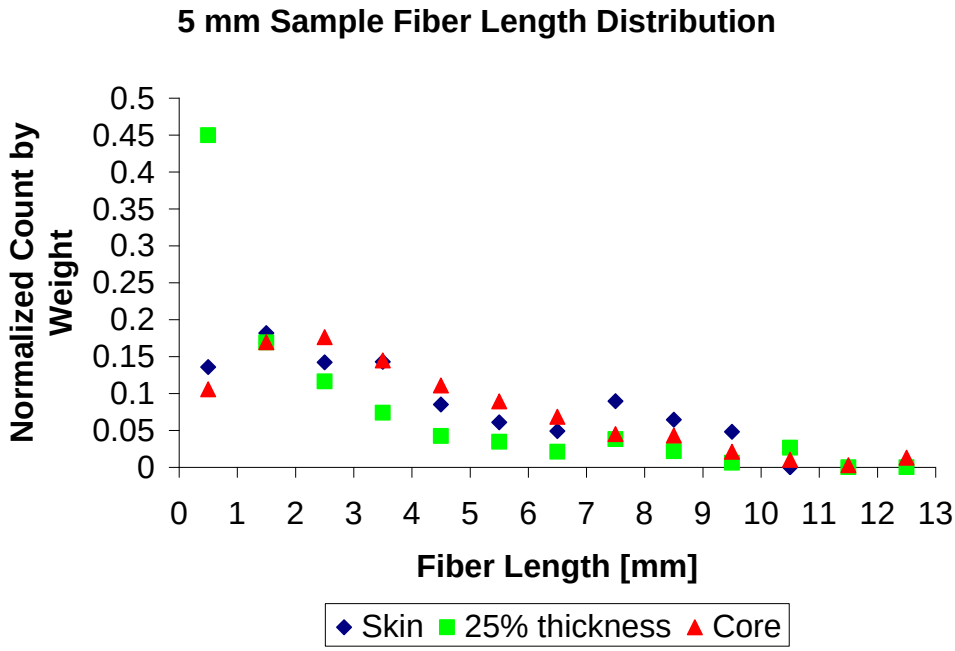

Figure 10. FLD for 3 mm thick glass/polypropylene LFT sample

Conclusions

Novel measurement technique allowing characterization of FLD as a function of thickness was developed and tested. This method relies on expansion of fiber mass during burn-off. Preliminary results show varying FLD through the thickness of glass/polypropylene injection molded LFT samples. While the variation may not be an important factor for determination of in-plane response of LFT, it may become significant for calculation of out-of-plane response such as bending. Further research is being conducted to provide a set of quantitative results with improved digital image processing technique.

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