

Birefringent Properties of 3D-Printed Polymers [†]

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Abstract

Birefringence describes the optical properties of optically anisotropic or chiral materials. As linear birefringence is a measure of anisotropy in the structure of a glass or a polymer, stress induced in glasses or polymers can be measured by stress birefringence. Interestingly, this effect can also be found in 3D-printed materials, such as fused filament fabrication (FFF)- or stereolithography (SLA)-printed objects, for some materials or material blends. Here, we report birefringence measurements on 3D-printed translucent objects, performed using a microscope with crossed polarizers as well as large-scale crossed polarizers, and show visual differences between macroscopic and microscopic effects.

Keywords: linear birefringence; 3D printing; fused filament fabrication (FFF); stereolithography (SLA); crossed polarizers

1. Introduction

Birefringent materials are optically anisotropic or chiral. Linear birefringence is defined as the difference between the refractive indices of the extraordinary and the ordinary ray direction, i.e., $\Delta n = n_e - n_o$, and is proportional to the optical path difference. Circular birefringence, on the other hand, occurs in chiral materials and describes the rotation of the plane of polarization.

Technically, linear birefringence can be used to measure stress induced in glasses or transparent polymers. Measurements of this so-called stress birefringence are very important in optical materials for lenses, micro-lens arrays, etc., especially when they are used in polarization-dependent and fine optical applications, where usually no birefringence should occur [1–3]. Besides stresses frozen in the material during manufacturing, stresses can also occur due to mechanical deformation of glasses or polymers [4,5]. In addition, aging after different treatments or on long-time scales can be investigated by birefringence measurements [6,7].

Birefringence due to frozen stress may also occur in 3D-printed polymer materials, such as fused filament fabrication (FFF)-printed models of different layer orientation and thickness [8], direct ink writing [9], or stereolithography (SLA)-printed objects containing liquid crystals or nanofibers [10]. The few papers showing these effects usually visualize either macroscopic or microscopic images of colored birefringence in parts of the 3D-printed objects [8–10]. A more sophisticated setup, based on a polarization camera, was reported by Kotula et al. [11], who investigated FFF-printed translucent polylactide acid.



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Similarly, polarized light microscopy on single layers or microtome-cut slices of FFF-printed samples from PETG (glycol-modified polyethylene terephthalate), showing colored or white birefringence depending on printing parameters and heat treatment [12].

Since white birefringence was observed in many cases, let us briefly recall its definition. White birefringence is usually a high-order optical phenomenon in which a material observed under crossed polarizers appears white due to the overlapping and blending of interference colors. This phenomenon occurs exclusively under polychromatic (white) light illumination.

Besides stress birefringence, there are other effects related to linear or circular birefringence. Birefringence can occur, e.g., in meta-surfaces with spatially inhomogeneous refractive index profile used for quantum differential holography [13]. Its exact measurement can be performed by anisotropic laser feedback polarization [14]. Among the diverse applications, there are dual-comb generation in a birefringent crystal [15], phase modulation, light splitting, phase matching of nonlinear optical crystals [16], or a vertical-external-cavity surface-emitting laser [17].

Here, we show combinations of macroscopic and microscopic images, taken on different 3D-printed materials as-printed and after thermal post-treatment steps, between crossed polarizers, to compare the birefringence effects that can be recognized on different scales.

2. Materials and Methods

The samples under investigation were prepared by fused filament fabrication (FFF) or direct light processing (DLP), a technique comparable to stereolithography (SLA). For FFF samples, a printer I3 MK3 (Prusa Research AS, Prague, Czech Republic) with nozzle diameter 0.4 mm was applied, using a layer height of 0.15 mm, three perimeters, linear filling pattern and an infill of 100%. DLP samples were printed on an Anycubic Photon Mono X SLA printer (Anycubic, Shenzhen, China) with a layer height of 50 μm , 8 bottom layers, an exposure time of 8 s, a bottom exposure time of 60 s, a light-off delay of 1 s, lift after print of 8 mm with lifting speed 1 mm/s and lowering speed 3 mm/s, and an infill of 100%.

Sample dimensions were 4 mm $\times$ 10 mm $\times$ 100 mm (according to EN ISO 178 “Plastics—Determination of flexural properties” [18]). The materials used for FFF printing were HD-glass (Filamentworld, Neu-Ulm, Germany) and HT-PLA (High-Temperature Poly(lactic acid) (Filamentworld), while DLP printing was performed with Clear Basic 3D printing UV-sensitive resin (Anycubic).

On some of the samples, a thermal treatment was performed in a climate chamber CTC256 (Mettler, Schwabach, Germany) as follows: thermal cycling between $-15\text{ }^{\circ}\text{C}$ and $+85\text{ }^{\circ}\text{C}$ (2 cycles, $\sim 21\text{ h/cycle}$) or between $-15\text{ }^{\circ}\text{C}$ and $+185\text{ }^{\circ}\text{C}$ (4 cycles, $\sim 38\text{ h/cycle}$). The sample identifications are given in Table 1 [19], while Table 2 shows glass transition temperature, melting temperature, and degradation temperature, if available (from [19,20]).

Table 1. Sample identification.

Thermal Treatment	HD-Glass (FFF)	HT-PLA (FFF)	Clear Basic Resin (DLP)
None	As-printed HD-glass	As-printed HTPLA	As-printed Clear Basic Resin
$2 \times (-15\text{ }^{\circ}\text{C} \dots +85\text{ }^{\circ}\text{C})$	HD-glass after heat treatment at $85\text{ }^{\circ}\text{C}$	HT-PLA after heat treatment at $85\text{ }^{\circ}\text{C}$	Clear Basic Resin after heat treatment at $85\text{ }^{\circ}\text{C}$
$4 \times (-15\text{ }^{\circ}\text{C} \dots +185\text{ }^{\circ}\text{C})$	HD-glass after heat treatment at $185\text{ }^{\circ}\text{C}$	HT-PLA after heat treatment at $185\text{ }^{\circ}\text{C}$	Clear Basic Resin after heat treatment at $185\text{ }^{\circ}\text{C}$

Table 2. Glass transition, melting and thermal degradation temperatures of the materials under investigation (from [19,20]). DLP resins are thermosets and thus do not have melting temperatures. (na): not announced.

Material	Glass Transition (°C)	Melting Temperature (°C)	Degradation Temperature (°C)
HD-glass	76	220	(na)
HT-PLA	60	155	250
Clear Basic Resin	85	no melting temperature	(na)

Macroscopic birefringence was tested by placing the samples between crossed polarizer sheets (20 cm × 30 cm) (Astromedia, Waltrop, Germany). For illumination, a flat light FL-4 × 8 with 32 W diffused along an area of 200 mm × 308 mm (Georg Ronge Lichtquellen, Hamm, Germany) was used. Photographs were taken by a camera Basler ace—acA2500-14uc with an objective Basler Lens C125-1218-5M f12mm (Basler AG, Ahrensburg, Germany).

Microscopic birefringence was investigated by an inverse microscope Axio Observer 7 (Carl Zeiss, Göttingen, Germany) using transmitted light with crossed polarizers and an objective 1.25×.

3. Results and Discussion

The analysis of the results is subdivided into sections based on macroscopic and microscopic images and discusses findings for the three materials under investigation.

3.1. Macroscopic Birefringence Tests

Generally, all FFF-printed samples show approximate white birefringence when investigated as-printed or after heat treatment at 85 °C, while the DLP-printed samples (as printed or after heat treatment at 85 °C) mainly show white birefringence along the edges (Figure 1a). A clear difference is visible for HD-glass after heat treatment at 185 °C (Figure 1b), showing a slightly orange color in the middle and slightly greenish edges.

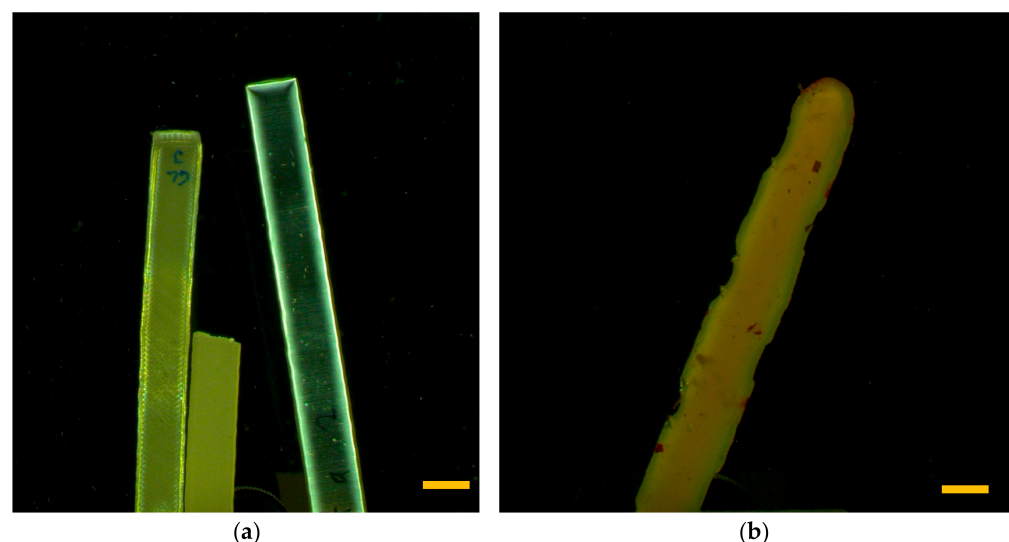


Figure 1. 3D-printed polymers between crossed polarizers. (a) As-printed HD-glass (left), HT-PLA (middle), and Clear Basic Resin (right); (b) HD-glass after heat treatment at 185 °C. Scale bars are 10 mm.

Generally, white appearance can occur if the residual intensity is approximately wavelength-independent along the visible range. However, even materials in which disper-

sion occurs can show white birefringence due to imperfect extinction, multiple reflections, or similar effects [21,22].

The macroscopic birefringence of the as-printed samples thus mainly shows that there are frozen strains throughout the FFF-printed samples, as could be expected due to the production process, while this effect only occurs along the edges of the DLP-printed samples, which are apparently relaxed inside the bulk material.

The HD-glass sample after heat treatment at 185 °C looks slightly yellow under standard illumination and is no longer translucent [19], so the visible birefringence colors are a superposition between intrinsic color and birefringence. This will be discussed in more detail in the next sub-section.

Besides frozen strains, birefringence also enables seeing the effect of external tension. Figure 2 exemplarily depicts colors in an as-printed HD-glass sample under tension. This investigation is especially interesting for 3D-printed samples, in which an applied tension may lead to a more complicated stress distribution than in homogeneous materials. Stress-strain examinations using a three-point bending test setup are planned for the near future.

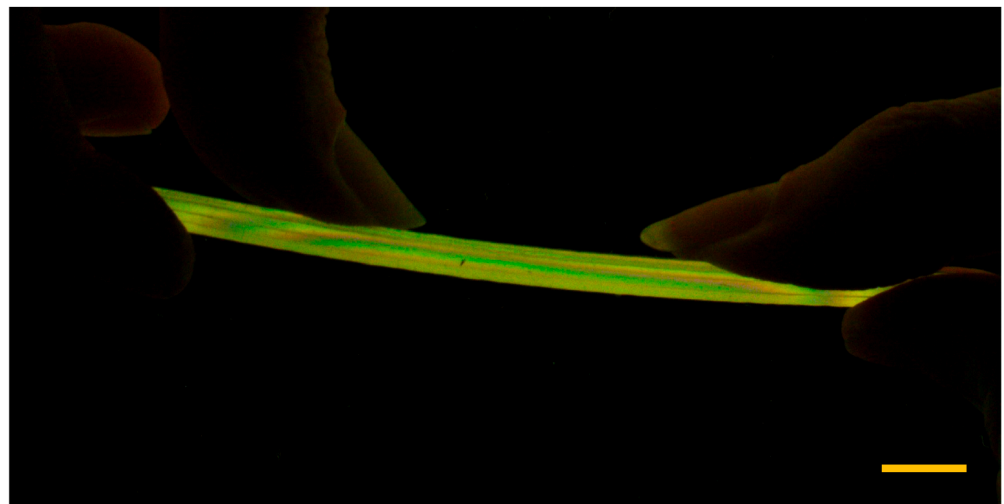


Figure 2. As-printed HD-glass sample under tension between crossed polarizers. Scale bar is 10 mm.

3.2. Microscopic Birefringence Tests

Firstly, Figure 3 shows DLP printed samples under microscopic crossed polarizers. Firstly, small matrix-like structures are visible in all images, stemming from the DLP matrix that UV-polymerizes the resin. Irregularities in the images show the positions where support material was attached.

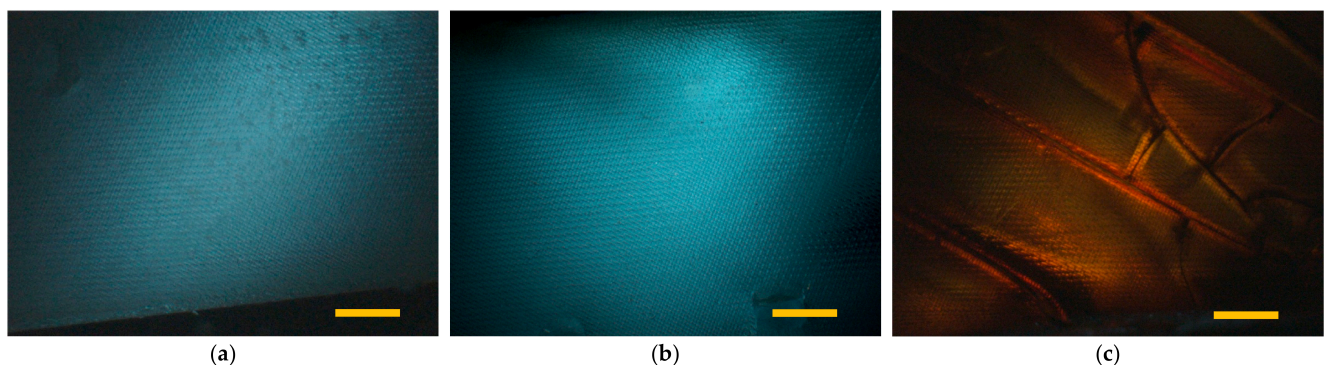


Figure 3. Microscopic images of DLP printed samples between crossed polarizers, nominal magnification 1.25 $\times$. (a) Sample as-printed; (b) after heat treatment at 85 °C; (c) after heat treatment at 185 °C. Scale bars are 1 mm.

Next, the as-printed samples (Figure 3a) and the samples heat-treated up to 85 °C show very similar microscopic birefringence. Opposite to the macroscopic images (Figure 1a), not only do the edges show white birefringence, but the whole samples do. This indicates that the illumination direction has a significant influence on these effects, potentially showing the edges brighter in the macroscopic image due to internal reflections. However, there are again no color differences visible in these samples under microscopic investigation.

After heat treatment at 185 °C, the sample has turned brown under standard illumination [19], which is also reflected by the brownish appearance of the birefringence image (Figure 3c). Here, color differences are visible along the cracks inside the sample that appeared during heat treatment, indicating differences in internal strain.

As mentioned before, white birefringence is usually a high-order optical phenomenon in which a material observed under crossed polarizers appears white due to the overlapping and blending of interference colors. This effect cannot be distinguished, however, from white birefringence gained by non-wavelength-dependent retardance.

As an example for FFF-printed samples, Figure 4 shows microscopic images of HT-PLA samples between crossed polarizers. After heat treatment at 85 °C (Figure 4a), the sample looks identical to the as-printed sample, showing approximate white birefringence.

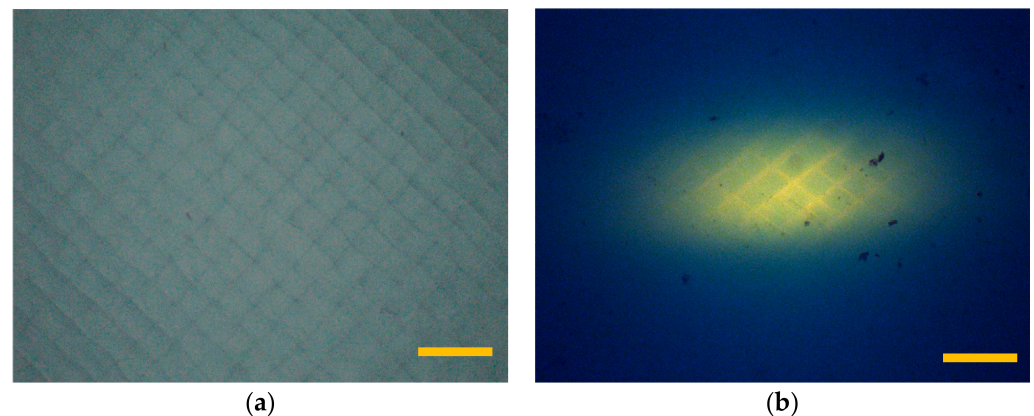


Figure 4. Microscopic images of HT-PLA samples between crossed polarizers, nominal magnification 1.25 $\times$. (a) After heat treatment at 85 °C; (b) after heat treatment at 185 °C. Scale bars are 1 mm.

Heat treatment at 185 °C, however, results in partly melting and re-solidifying the sample, which leads to colored birefringence showing yellow colors in the middle of the sample and bluish colors outside. It should be mentioned that the sample looks slightly yellow under standard illumination [19], which will overlap with the birefringence effect.

While heat treatment at relatively low temperatures does not change the appearance of the HT-PLA samples, HD-glass samples show a slight modification upon increasing temperatures. To investigate this effect, Figure 5 shows a series of images taken after heating HT-PLA samples up to different temperatures.

As these microscopic images show, the structural modifications and the related birefringence effects of the samples increase steadily with increasing heat treatment temperature, until the material is fully molten and re-solidified after heat treatment at 185 °C (Figure 5f). This is unexpected at first glance, since thermal treatment should release the frozen strains and thus reduce the birefringence. On the other hand, the specific structure of FFF-printed materials has to be taken into account—due to the air voids between adjacent filament strands, even samples printed from fully transparent materials are not transparent. It can thus be imagined that heat treatment leads to re-organization inside the sample and thus reduction in air voids, which will make the sample more transparent and thus enable the

detection of more polarized light transmitted through the sample. To further investigate this idea, single-layer samples will be investigated in the near future.

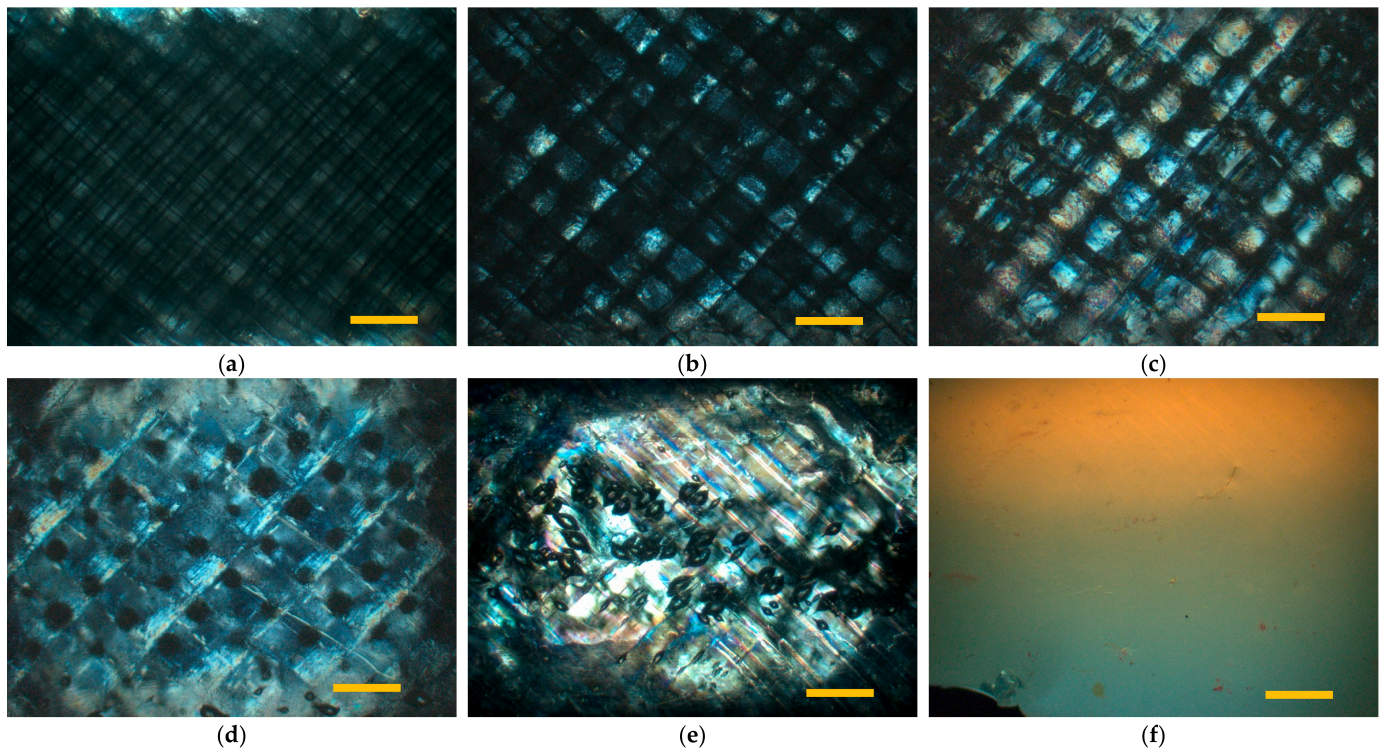


Figure 5. Microscopic images of FFF-printed HT-PLA samples between crossed polarizers, nominal magnification $1.25\times$. (a) Sample as-printed; after heat treatment at (b) 100 °C; (c) 120 °C; (d) 140 °C; (e) 160 °C; (f) 185 °C. Scale bars are 1 mm.

4. Conclusions

FFF- and DLP-printed samples have been investigated macroscopically and microscopically between crossed polarizers, revealing internal strains and external tensions by birefringence measurements. As-printed samples show white birefringence on both scales, while heat treatment, especially until the samples are molten and re-solidified, leads to colored effects. All these findings indicate structural transformations in which high-order interference effects, responsible for the occurrence of white or in some cases colored birefringence, lead to a structural arrangement exhibiting a higher degree of order than the as-printed structure that has not been subjected to thermal treatment.

Future investigations are especially planned to investigate stress birefringence on macroscopic and microscopic scales by applying three-point bending tests during imaging, in order to examine the space-resolved stresses inside layered FFF-printed samples.

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Abbreviations

The following abbreviations are used in this manuscript:

FFF	Fused filament fabrication
SLA	Stereolithography
DLP	Direct light processing

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